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REGION I, EPA-NEW ENGLAND DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ENVIRONMENTAL ANALYSES

U.S. EPA-NEW ENGLAND
Region I
Quality Assurance Unit Staff
Office of Environmental Measurement and Evaluation

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Preface

The Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses, consists of Part I, the "Data Validation Manual: The Data Quality System", December 1996 Revision, Part II, "Volatile/Semivolatile Data Validation Functional Guidelines", December 1996, and Parts III, "Pesticide/PCB Data Validation Functional Guidelines" and IV, "Inorganic Data Validation Functional Guidelines", which are not yet released. This Preface will be updated with the finalization of Part II, the release of Parts III and IV and any subsequent revisions or additions, and will accompany those revised documents.

This document was written by the QA Unit Staff of Region I, EPA New-England to formalize technical direction given since the original Region I Functional Guidelines were implemented in 1988. Data validation is necessary to ensure that only data of known and documented quality are used in making environmental decisions. As such, this guidance serves as a standard operating procedure that documents Region I's commitment to using only scientifically defensible data in environmental decision-making, it documents compliance with Headquarters' directives and guidance, and it ensures that data generated by or for the region are evaluated consistently. Part I, the "Data Validation Manual: The Data Quality System" includes by attachment other Regional and National Quality Assurance guidance documents utilized in conjunction with this new guidance to support Region I's data quality system.

REGION I, EPA-NEW ENGLAND DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ENVIRONMENTAL ANALYSES

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PESTICIDE/PCB DATA VALIDATION
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REGION I, EPA-NEW ENGLAND DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ENVIRONMENTAL ANALYSES

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This document is under development. In the interim, validators must use the information in Part I, the "Data Validation Manual: The Data Quality System" for the data validation process and deliverables but use procedures contained in "Region I Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", 6/13/88, modified 2/89, to validate inorganic data generated by or submitted to EPA-NE.

PART I - DATA VALIDATION MANUAL: THE DATA QUALITY SYSTEM

U.S. EPA-NEW ENGLAND
Region I
Quality Assurance Unit Staff
Office of Environmental Measurement and Evaluation

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PART I - DATA VALIDATION MANUAL: THE DATA QUALITY SYSTEM

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DATA VALIDATION MANUAL: THE DATA QUALITY SYSTEM

1.0 INTRODUCTION

The Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses, consists of Part I, the "Data Validation Manual: The Data Quality System", and Parts II, III and IV, the specific Functional Guideline procedures for validating multi-media organic and inorganic data. Additional Functional Guideline procedures will be prepared as needed.
The data validation guidance presented in this document is intended to ensure that data of known quality are provided to both Superfund and non-Superfund EPA-NE program personnel. It is applicable to data generated for all Superfund work performed by EPA, Potentially Responsible Parties (PRPs), other Federal Agencies, States, and for oversight activities performed by EPA-NE. In addition, it is applicable to data generated for all non-Superfund work performed by EPA, other Federal Agencies, and State, Tribal and industrial partners and voluntary monitors.

These data validation procedures are not limited to Contract Laboratory Program (CLP) data. They can be employed regardless of the mechanism used to generate the data and the program for which they were generated. They may be modified to suit any organic or inorganic sample separation procedure, including chromatographic techniques such as gas chromatography or ion chromatography, and any analytical method including performance-based methods utilizing a variety of detectors. The data validation guidelines in Part II -IV of this document are not limited to aqueous and soil/sediment matrices but may be modified to evaluate other environmental matrices including, but not limited to, oil, fly ash, biological tissue and air.

2.0 DEFINITION OF DATA VALIDATION

Data validation, the first step in assessing data quality, is a standardized review process for judging the analytical quality and usefulness of a discrete set of chemical data. Thus, data validation identifies the "analytical error" associated with a data set. Data validation can also identify some (e.g., incorrect preservation techniques), but not all of the "sampling error" associated with a data set. The sum of the "analytical error" and the "sampling error" is known as the "measurement error", as per Equation 1.

Equation 1: Measurement Error = Sampling Error + Analytical Error

The "measurement error" is used in conjunction with "sampling variability" (spatial variability of pollutant concentrations) to determine "total error" or "total uncertainty" associated with a data set, as per Equation 2. It should be noted that "sampling error" and "sampling variability" usually contribute a greater percentage of the "total error" associated with a sampling event than the "analytical error".

Equation 2: Total Error (uncertainty) = Measurement Error + Sampling Variability

Once the "total uncertainty" has been estimated, the end user can assess the usability of a data set in the context of previously developed project Data Quality Objectives (DQOs). For additional QA Guidance, refer to EPA Order 5360.1 and Publication 9200.2-16FS contained in Attachment A.

Data validation can be viewed as a decision making process during which established quality control criteria are applied to the data. During this process, individual sample results are either accepted, rejected or qualified. Data which meet all the validation criteria are accepted as unqualified and can

be used as needed, assuming that no problems occurred during the sampling events. Data which are rejected (R) for not meeting one or more of the validation criteria cannot be used at all. Some data fall into the grey area between accepted and rejected. These data are qualified as "estimated" (J) to indicate that one or more of the validation criteria were not met. Estimated data may or may not be usable depending on the intended use of the data. In general, estimated (J) data can be used after examining the reasons for data qualification and its impact on the achievement of the project DQOs. Estimated data, however, should not be used indiscriminately.

> **The end product of data validation is data of known and defensible analytical quality and, therefore, data should not be assessed for usability and used in environmental decision making until after completion of the data validation process.**

3.0 PURPOSE OF DATA VALIDATION

Data validation serves many purposes. As previously discussed, the primary purpose of data validation is to assess and summarize the quality and defensibility of the laboratory's analytical data for the end users: site managers, risk assessors, hydrogeologists, and lawyers. The data validation process focuses on evaluating the analytical laboratory's performance so that the "analytical error" associated with a data set can be determined. It provides a technical judgment on the validity of the laboratory results as a first step in determining their overall usability and legal defensibility. To this end, the data validator may be required to consult with the sampler in an effort to identify field problems. For example, incorrect preservation procedures result in "sampling error" and contribute to the overall "measurement error" associated with a data set. The data validation process does not include consideration of "sampling variability"; **this is left to the end user in the final assessment of data usability.**

Second, for data generated under the Superfund Contract Laboratory Program, data validation assists the Region I Technical Project Officer (TPO) in monitoring Regional CLP laboratory performance. If a laboratory fails to produce contractually-compliant data, then payment to the laboratory may be reduced or denied by procedures initiated by the EPA Field Sampling Contractor and recommended to the National Program Office (NPO) by the CLP-TPO. The TPO can also recommend that the CLP Contracting Officer take contract action against a contractually non-compliant laboratory.

Similarly, for data generated by non-CLP laboratories, data validation assists those organizations procuring analytical services in monitoring laboratory performance. If a non-CLP laboratory fails to produce contractually-compliant data, then payment to the laboratory may be reduced or denied.

It is important to emphasize that the purpose of data validation is to identify "analytical error" and not to make final determinations about the <u>overall</u> usability of the data for a project. The end user of the data must specify the overall Data Quality Objectives (DQOs) for the project during the up-front scoping process. Then, during data validation, the effect of individual analytical problems on the accuracy and precision of the data is detailed for specific analytes and proper qualifiers are applied to the data. Validation is just the first step in deciding whether or not data for a particular sample can be used for a specific purpose. Ultimately, only the end user can assess usability based on the "measurement error" and "sampling variability" associated with the data package. The project chemist and/or validator, however, are generally consulted by the end user to interpret decisions made with regard to measurement error during the usability determination.

4.0 <u>REGION I, EPA-NEW ENGLAND DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ENVIRONMENTAL ANALYSES</u>

All Superfund data generated for and/or used by EPA-NE must be validated in accordance with the

most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, and this requirement should be clearly documented in the project Quality Assurance Project Plan (QAPjP) or Sampling and Analysis Plan (SAP). Any deviation from this stated data validation policy must be documented and justified in the site QAPjP or SAP and approved by the Agency.

> **If CLP methods are used to generate site data, then the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses must be used without deviation for the data validation process.**
>
> **If non-CLP methods are used to generate site data and modified validation criteria are necessary to validate those data, then all deviations to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses must be documented in an approved QAPjP or SAP specific to that site.**

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses is based on the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, February 1994 and the U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, February 1994, but has been modified to provide generic guidance for reviewing any organic data generated by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS) and any inorganic data generated by Atomic Absorption (AA) or Inductively Coupled Plasma (ICP) spectrometry.

In some aspects, this document is equivalent to a standard operating procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. Those areas where specific validation procedures are appropriate have definitive performance requirements established in the contract or the method. These requirements are not sample dependent; they specify performance on parameters that should be fully under a laboratory' s control, such as laboratory blanks, calibration standards, performance evaluation standard materials, GC/MS mass calibration, peak shape and resolution.

Other performance requirements, such as the frequency of Quality Control (QC) actions, are dependent on the contract or the method, the number of samples, sample preparation technique, time of analysis, etc., and are not identical for every case or batch of samples. Individual case requirements and the impact of non-conformance must be addressed on a case-by-case basis; therefore, no specific guidance is provided. For example, the CLP organic contract requirement that a laboratory blank analysis be performed a minimum of once every twelve hours of analysis time must be translated into the number of blanks required for a specific set of samples. The data validator may have to consider the impact on data quality for a sample analyzed thirteen hours after a blank, in terms of the quality of that particular sample data.

For some CLP data, a Contract Compliance Screening (CCS) automated review is performed by the CLP NPO Sample Scheduling and Coordination Contract (currently Contract Laboratory Analytical Services Support [CLASS], formerly Sample Management Office) to assess both technical and contractual deficiencies as presented by the laboratory in an electronic format. CCS is available to the validator and can be utilized to assist in data validation and in determining reduced value/data rejection recommendations (See Section 8.4 for additional information). However, for some CLP data (i.e., dioxin) and for all EPA-generated non-CLP data, a contractual screen is not performed by the CLP National Program Office. In the future, those organizations procuring analytical services may choose to implement their own contractual screening procedures. Until that time, the validator must assess both technical and contractual deficiencies in order to determine analytical quality as well as contractual non-compliance. Contractually non-compliant data, which are unusable for making site decisions and are considered "unacceptable" to the Region, should be considered for reduced payment or data

rejection/non-payment to ensure that EPA does not pay for "unacceptable" data.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data that are contractually non-compliant is strictly to facilitate the progress of projects requiring the availability of the data. A laboratory submitting non-compliant data may be required to re-extract and/or reanalyze samples and/or resubmit data even if the previously submitted data have been utilized due to urgent program needs. Data that are not fully usable may be recommended for reduced payment if those data are contractually non-compliant. Data that are rejected due to contractual non-compliance should be returned to the laboratory and payment denied. Data that have been rejected and returned to the laboratory cannot be used by the Region in site decisions.

If the nature of the sample itself limits the attainment of contract or method quality control and/or validation specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid, legally defensible, of known quality, and ultimately usable in making site decisions.

5.0 GENERAL OVERVIEW OF THE DATA VALIDATION PROCESS

In order to perform data validation, certain quality control (QC) checks and analytical procedures must be performed in association with the analysis of the environmental samples. Examination of the results of these checks and procedures allows the trained validator to determine the analytical quality of the data in question.

To provide data of known quality, the data validator should: 1) review the data package to ensure that it contains all the required documents and forms, 2) assess the results of all QC checks and procedures, and 3) examine the raw data in detail to verify the accuracy of all information presented by the laboratory. These three levels of review constitute the Region I Tiered Validation approach. Refer to Attachment B, Region I Tiered Organic and Inorganic Data Validation Guidelines, July 1, 1993, Draft or most recent revision. Note that the tiered validation procedures specific to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses have been incorporated into the text in each section of Parts II-IV.

Data completeness is the first item checked during validation. The validator needs all the laboratory documents in order to verify the accuracy of sample analysis results reported by the laboratory and to ensure the legal defensibility of the data. Prior to submitting sample results, the laboratory must do a complete file purge. In the CLP, this is known as the Complete SDG File (CSF) purge. This purge assembles all the supporting documentation and deliverables needed to substantiate the reported results that are used in site decisions and/or litigation support. If any part of the complete file purge information is not present, then the validator or designated Regional representative contacts the laboratory to obtain the missing documentation. This process ensures that all the required deliverables are present in the package. If missing deliverables are not obtained at this time, in all likelihood they will never be recovered. Since any data package has the potential of being used in court for enforcement or to support a site decision, all CLP and non-CLP data packages must be routinely checked for completeness. Refer to Attachment C for Region I CSF Completeness Evidence Audit Program, July 3, 1991 or most recent revision. **The validator should evaluate any Performance Evaluation sample results to assess potential usability issues, as part of the first step in data validation.**

Second, the reported results of all QC checks and analytical procedures are evaluated. Items such as holding times, sample preservation techniques, QC sample results, etc., are assessed. QC samples are designed to identify problems in three specific areas: laboratory/instrument performance, sample preparation/matrix effects, and field performance. The validator checks laboratory and instrument performance by reviewing items such as laboratory blank contamination and instrument calibration. Unusual matrix effects can be detected by examining the results from matrix spike/matrix spike duplicates (MS/MSD), surrogate spike recoveries, and internal standard responses. These matrix

effects can be caused by high concentrations of non-target analytes which mask the analytes of interest. High levels of peat or clay can bind the target analytes to produce unwanted matrix effects. Potential problems originating from field sampling work are assessed by examining the field duplicate, equipment blank, and trip blank results. It should be noted that field QC checks cannot completely assess the "error" associated with field sampling procedures. If the evaluation of QC checks indicate laboratory or field problems, then the validator must discuss their impact on the data in the Data Validation Memorandum and qualify the sample results in accordance with the guidance in Parts II, III and IV of this document.

Last, the validator examines the raw data in detail to verify the accuracy of the results reported by the laboratory. Reported sample concentrations are checked by recalculating about 10% of the original calculations unless problems warrant further investigation. Proper identification of all the analytes is confirmed by examining the laboratory instrument print-outs. The validator is responsible for resolving discrepancies in the reported data with the laboratory and obtaining resubmittals from the laboratory whenever necessary. Occasionally, the identification and concentration of target analytes reported in the samples may need to be changed upon validation.

In summary, the data validation process involves the following three steps:

Tier I: The data package is checked for completeness. The DC-2 Form (Inventory Sheet) is completed and signed. This ensures that the data set is complete for potential use in court. The PE sample results are evaluated to assess potential usability issues. For Tier I validations, a Tier I Validation Cover Letter is produced by the validator.

Tier II: The results of the QC checks, analytical procedures and PE sample results are assessed and applied to the data set. This will result in the proper qualifiers being applied to the data. For Tier II validations, a Data Validation Report is produced by the validator.

Tier III: The raw data are examined in detail to check for calculation, compound identification, and/or transcription errors. For Tier III validations, a Data Validation Report is produced by the validator.

The validation tier used to validate each data package must be documented in the first paragraph of either the Tier I Validation Cover Letter for Tier I validations, or the Data Validation Memorandum from the Data Validation Report for Tier II and Tier III validations. For Tier I validations, the Tier I Validation Cover Letter must document the site-specific justification for limiting the validation to Tier I and the validator's evaluation of the PE sample results.

In general, validation should be completed within 21 days of receipt of the data package from the laboratory. This enables the user and/or site manager to assess contractual compliance and data usability in order to make timely site decisions. Accelerated site schedules may necessitate shorter turnaround times for validation. In general, the completion of a Data Validation Report should not be delayed because the laboratory failed to forward a resubmittal. In most cases, the Data Validation Report should be completed, the laboratory omission noted, and the data qualified using professional judgment. When/If the resubmittal is received, an amendment to the original Data Validation Report should be forwarded.

In some cases, the validator must wait for critical information before the validation can be completed. In these cases, the user and/or site manager must be notified of the delay. If validation reports are time critical, the site manager may request that a partially completed Data Validation Report be generated. Subsequently, an amendment should be written to incorporate all late resubmittals.

6.0 SUMMARY OF DATA VALIDATION OBJECTIVES

Data validation must concurrently accomplish the following:

- Assess and summarize the analytical quality and defensibility of data for the end user.
- Document for the historical record all factors contributing to "analytical error" that ultimately affect data usability, such as: data discrepancies, poor laboratory practices that impact data quality, site locations for which samples were difficult to analyze, i.e., matrix effects. Also, document any "sampling error" that may be identified by the data validation process, such as, contaminated trip or equipment blanks, incorrect storage or preservation techniques, improper sampling containers, and improper sampling techniques, i.e., headspace in VOA containers.
- Assist Regional TPOs in monitoring CLP laboratory performance for contract administration.
- Assist in monitoring any laboratory' s performance of CLP methods in generating data for submittal to EPA.
- Assist in monitoring any laboratory' s performance of non-CLP methods in generating data directly for EPA or for submittal to EPA.
- Identify contractually non-compliant data that are unusable by the Region. For CLP data, a letter documenting the contractual non-compliances and recommending reduced payment or data rejection must be written and addressed to the CLP-TPO, in accordance with EPA-NE Standard Operating Procedures for Submitting Data for Reduced Payment/Data Rejection, September 1991 (Attachment I). For non-CLP data generated directly for EPA, i.e., under the DAS program, contractually non-compliant data should also be identified and documented so that contractual action can be taken to ensure that the Region does not pay for unusable, contractually non-compliant data. In general, contractually non-compliant data should always be identified and documented to support any contractual action taken by the data requestor.
- Provide information concerning the effectiveness of analytical methods and SOWs, and identify problems requiring method revision and/or resolution.

7.0 ROLES AND RESPONSIBILITIES

The end users of the data are responsible at the time of project scoping for determining the validation criteria, including validation Tier, that are necessary to support the achievement of project DQOs.

The question then arises as to who is responsible for performing data validation. In general, whoever collects field samples at the site is also responsible for validating the analytical data. An exception exists when the organization collecting the samples uses their own internal laboratory to analyze the samples; in this situation an independent third party must validate the data. In general, EPA Field Sampling Contractors working on Fund-lead sites are responsible for validating the results for samples that they collect. States working on Fund-lead sites under Cooperative Agreements with EPA are responsible for validating their own samples. Likewise, other government agencies working on Fund-lead sites under Interagency Agreements are responsible for validating results for samples that they collect from their sites, i.e., the Army Corp of Engineers. For non Fund-lead sites, PRPs and Federal

Facilities traditionally have been required to use an independent third party for data validation.

When an EPA Field Sampling Contractor performs PRP or Federal Facility oversight, duplicates (splits) for approximately 10% of the PRP's or Federal Facility's samples are analyzed by EPA. The PRP or Federal Facility must validate the data for the samples which it collects. If after PRP or Federal Facility validation, the two sets of data agree within the predetermined limits presented in the EPA-approved QAPjP and/or SAP, then the EPA oversight contractor data may not need to be validated. If they do not agree within the predetermined limits, then the EPA oversight contractor data must also be validated to investigate the cause of the discrepancy. Further corrective actions may be necessary to identify the source of the discrepancy.

7.1 EPA-NE Delivery of Analytical Services (DAS) Team (Quality Assurance Unit-Office of Environmental Measurement and Evaluation)

The EPA-NE DAS Team located within the Quality Assurance Unit of the Office of Environmental Measurement and Evaluation (OEME) is responsible for developing data validation guidance, training EPA Field Sampling Contractors in data validation, and operating an oversight program to ensure that EPA Field Sampling Contractors are performing data validation in accordance with EPA-NE policy.

The DAS Team also provides technical assistance concerning analytical methods, data validation and data usability to EPA Site Managers and EPA Field Sampling Contractor Lead Chemists. Technical assistance is also offered to the States, Tribal and industrial partners, other Federal Agencies, the public, and PRPs through the responsible EPA Site Manager.

In general, OEME does not perform site-specific data validation with the exception of OEME sampling events and all dioxin/furan samples collected by EPA personnel and EPA Field Sampling Contractors.

The EPA-NE DAS Team acts as the Regional contact point for all CLP matters and maintains the EPA-NE Performance Evaluation Sample Program.

7.1.1. EPA-NE CLP-Technical Project Officer

The CLP Technical Project Officer (TPO) is responsible for monitoring the CLP contract laboratories within EPA-NE. This includes responding to the laboratory's technical questions; reviewing laboratory performance trend information and data reviews provided by the National Program Office (NPO) and other Regional TPOs; discussing and documenting CLP laboratory performance problems; tracking laboratory corrective action requests/responses; assessing the adequacy of a CLP laboratory's corrective action response; recommending contract action to the Administrative Project Officer (APO) and Contracting Officer (CO); conducting routine and problem resolution on-site audits; and monitoring the continued effectiveness of corrective actions implemented by the laboratory.

The CLP-TPO is also responsible for: reviewing and developing Superfund analytical methods and CLP SOWs; reviewing and developing CLP policies, guidance and procedures; disseminating information concerning CLP operation and available services; and participating in workgroups to revise and/or write analytical methods, National Functional Guidelines and other national QA guidance.

7.1.2. EPA-NE Data Validation Chemist

The Data Validation Chemist (DV Chemist) is responsible for all aspects of data validation within the Region, including: revising regional data validation Functional Guidelines;

providing guidance in using the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses; writing reduced payment and data rejection recommendation letters to the CLP-APO; and directing the Regional Data Validation Oversight/Methods Review Program. Through the Regional Data Validation Oversight/Methods Review Program, the DV Chemist identifies analytical issues/problems and needed corrective actions in order to reduce systematic "analytical error". Sampling issues and needed corrective actions are also identified in order to reduce systematic "sampling error". This program also helps to identify inherent problems in the analytical methods that require programmatic changes.

7.1.3. EPA-NE Performance Evaluation Chemist

The Performance Evaluation Chemist (PE Chemist) is responsible for all aspects of the Performance Evaluation Program within the Region, including: preparing, stocking, distributing, and tracking PE samples; scoring EPA-provided PE sample results and providing PES Score Reports to the data validators; and trending laboratory performance on PE samples.

7.1.4 EPA-NE Regional Sample Control Center

The EPA-NE Regional Sample Control Center (RSCC) serves as the central point of contact for questions concerning Superfund sampling efforts utilizing the CLP and any future EPA-NE analytical contracts. CLP and EPA-generated non-CLP (i.e., DAS) samples are collected, preserved, packaged, and shipped in accordance with EPA-NE, DOT, and NPO policy as described in EPA-NE Standard Operating Procedures (SOPs) and guidance documents pertaining to this subject and as documented in the EPA-approved QAPjP and/or SAP. Refer to Attachments D and E for selected guidance on the subject.

The responsibilities of the EPA-NE RSCC include: scheduling CLP sample analysis slots with the NPO Sample Scheduling and Coordination Contract to correspond with the projected demand for analytical services; providing CLP sample tags, sample labels, custody seals, and CLP COC/CLP Traffic Report Forms for EPA Field Sampling Contractors; coordinating with the NPO Sample Scheduling and Coordination Contract during sampling and sample shipment, and resolving any shipment problems concerning the CLP samples; receiving CLP data from laboratories and distributing Complete SDG Files (CSFs) to Region I Field Sampling Contractors for validation; and maintaining the New England Sample Tracking System (NESTS) database which tracks information pertaining to CLP and EPA-generated non-CLP samples delivered for EPA under the DAS mechanism.

7.2 EPA-NE Site Managers

EPA Site Managers include Site Assessment Managers (SAMs), Remedial Project Managers (RPMs), On-Scene Coordinators (OSCs) and RCRA Facility Managers (RFMs). They work in the EPA-NE Office of Site Remediation and Restoration (OSRR) and have primary responsibility for directing and/or overseeing response efforts and coordinating all actions at Superfund and RCRA corrective action sites. The SAMs, RPMs, OSCs, and RFMs establish the project Data Quality Objectives (DQOs) for their sites.

EPA Site Managers coordinate scoping meetings, assembling all technical personnel and data users to help identify the appropriate analytical methods, detection levels, level of quality assurance and, ultimately, the tier level of data validation required for specific sample results to achieve the project Data Quality Objectives. The EPA Site Managers receive copies of Data Validation Reports and Tier I Validation Cover Letters. The OEME QA Unit also receives copies of all Data Validation Reports and Tier I Validation Cover Letters for use in the Data Validation Oversight/Methods Review Program.

7.3 CLP National Program Office

The CLP is administered by the EPA National Program Office (NPO) under the Office of Emergency and Remedial Response (OERR), located in Washington, D.C. The NPO is primarily responsible for the overall management of the CLP in terms of program objectives. The NPO is also responsible for developing and administrating CLP contracts. CLP analytical contracts include Statements of Work for the organic and inorganic analyses of single-phase aqueous or soil/sediment samples. The NPO CLP short sheets and the Region I Statement of Work (SOW) short sheets for selected past and present CLP contracts are included in Attachment F.

The NPO is also responsible for formulating and implementing policy and budget; developing and administrating CLP analytical and support services contracts which include a contract responsible for sample scheduling and coordination; coordinating the production and dissemination of Superfund Performance Evaluation Samples; developing and reviewing analytical protocols; and directing CLP quality assurance in accordance with overall OERR quality assurance activities and directives.

7.3.1 NPO Sample Scheduling and Coordination Contract ([CLASS], formerly Sample Management Office)

The contractor-operated sample scheduling office provides management, operation and administrative support to the CLP under the direction of the NPO. The primary objective of this NPO contract is to maintain optimal use of program analytical resources. The contractor supports the NPO in sample scheduling and tracking and performs Contract Compliance Screening to help ensure proper and timely payment of CLP laboratories.

7.3.2 NPO Quality Assurance Technical Support Contract (QATS)

The QATS contract provides quality assurance (QA) support to the CLP under the direction of the NPO. QATS performs the following functions: preparing performance evaluation (PE) samples for CLP pre-award and post-award laboratory performance evaluations; evaluating pre-award and post-award PE sample data; performing QA audits on CLP-generated data including mass spectrometer data tapes; and assisting in the evaluation and development of CLP analytical protocols.

7.4 Potentially Responsible Parties (Non Fund-lead)

Potentially Responsible Parties (PRPs) as defined by CERCLA, Section 107, include 1) the current owners or operators of the facility; 2) any person who at the time of disposal of any hazardous substance owned or operated the facility at which the hazardous substances were disposed of; 3) any person who by contract, agreement or otherwise arranged for disposal or treatment, or otherwise arranged with a transporter for transport for disposal or treatment, of hazardous substances; or 4) any person who accepts or accepted any hazardous substances for transport to disposal or treatment facilities or sites from which there is a release or a threatened release. "Persons" are defined by the statute as individuals, commercial entities, corporations, partnerships, associations, joint ventures and governments.

PRPs that have entered into an agreement with EPA-NE to bear the cost of site investigations and cleanup or have been unilaterally ordered to implement site cleanups when there is an imminent and substantial endangerment presented by the site, must use an independent party to validate their data. All data must be validated in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. Any deviations and/or modifications to these Functional Guidelines must be documented in the QAPjP and/or SAP and must be approved by EPA prior to sampling.

7.5 Other Federal Agencies (Non Fund-lead)

When a Federal Agency other than EPA owns a Federal Facility designated as a Superfund site, then as mandated by Section 120 of CERCLA, that Federal Agency is designated the lead Agency for that Federal Facility Site. That Federal Agency is responsible for validating its own data in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. Any deviations and/or modifications to these Functional Guidelines must be documented in the QAPjP and/or SAP and must be approved by EPA prior to sampling.

7.6 Other Federal Agencies (Fund-lead)

Other Federal Agencies may enter into Interagency Agreements with EPA-NE to work on Fund-lead sites. Under an Interagency Agreement, the Federal Agency, i.e., Army Corp of Engineers, may use the CLP to analyze samples. Alternatively, it may choose to use a non-CLP laboratory to generate data for EPA. In either case, the Federal Agency should obtain a complete laboratory data package, in accordance with requirements and/or specifications described in Attachment G so that the data may be validated in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. Any deviations and/or modifications to these Functional Guidelines must be documented in the QAPjP and/or SAP and must be approved by EPA prior to sampling.

Federal Agencies that utilize CLP for sample analysis must submit quarterly CLP sample projections to the EPA RSCC. Completed DQO Summary Forms for each sampling event must accompany the quarterly projections. To reserve sample slots, other Federal Agencies must follow the procedures outlined in Section 9.1.3.1.

All CLP Data Validation Reports should be sent to the EPA-NE RSCC who then forwards them to the EPA-NE CLP-TPO for purposes of contract administration. Non-CLP Data Validation Reports should not be sent to the EPA-NE RSCC. Rather, non-CLP Data Validation Reports (including DQO Summary Forms) and/or Final Project Reports should be forwarded to the EPA Site Manager.

7.7 States (State-lead/Fund-lead)

New England States may enter into Cooperative Agreements with EPA-NE to work on Fund-lead sites within their State. Under a Cooperative Agreement, a State may use the CLP to analyze samples. Alternatively, it may choose to use a non-CLP laboratory such as their own State laboratory to generate data for EPA. In either case, the State should obtain a complete laboratory data package in accordance with requirements and/or specifications described in Attachment G so that the data may be validated in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. Any deviations and/or modifications to these Functional Guidelines must be documented in the QAPjP and/or SAP and must be approved by EPA prior to sampling.

States that utilize CLP for sample analysis must submit quarterly CLP sample projections to the EPA RSCC. Completed DQO Summary Forms for each sampling event must accompany the quarterly projections. To reserve sample slots, States must follow the procedures outlined in Section 9.1.3.1.

All CLP Data Validation Reports should be sent to the EPA-NE RSCC who then forwards them to the EPA-NE CLP-TPO for purposes of contract administration. Non-CLP Data Validation Reports should not be sent to the EPA-NE RSCC. Rather, non-CLP Data Validation Reports (including DQO Summary Forms) and/or Final Project Reports should be forwarded to the EPA Site Manager.

7.8 EPA Field Sampling Contractors (Fund-lead and PRP/Federal Facility Oversight)

EPA Field Sampling Contractors work under the direction of EPA Site Managers and are primarily involved in Fund-lead site work and PRP/Federal Facility oversight. Samples collected by EPA Field Sampling Contractors may be analyzed through the CLP, by the OEME laboratory or through an EPA-

NE analytical contract. Alternatively, the EPA Field Sampling Contractor may be directed by EPA to procure their own analytical services.

7.8.1 EPA Field Sampling Contractor Lead Chemist

This section details the responsibilities of the EPA Field Sampling Contractor's Lead Chemist working on Fund-lead or oversight activities for EPA-NE. However, many of the activities, roles, responsibilities and qualifications discussed below are applicable to non Fund-lead work performed by a PRP or Federal Facility as well as to Fund-lead work performed by another Federal Agency (i.e., ACOE) or a State.

7.8.1.1 Project Scoping

The Lead Chemist is a key participant in project scoping meetings where project Data Quality Objectives (DQOs), plans, schedules, sampling techniques, analytical methodologies and data validation criteria including validation tiers are discussed and agreed upon by the end users of the data. The Lead Chemist should ensure that all agreed upon DQOs, plans, schedules, sampling procedures, and analytical methodologies are incorporated into an EPA-approved QAPjP and/or SAP prior to field sampling.

During the scoping meeting, the Lead Chemist must identify the CLP and non-CLP analytical methods that are needed to generate data that achieve project DQOs. If CLP methods are used, then the QAPjP and/or SAP must specify the validation tier, document that the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses will be used without modification to validate the data, and must be approved by EPA-NE prior to sampling. If non-CLP methods are used and modified validation criteria are necessary to fully evaluate the data, then the QAPjP and/or SAP must document the modified validation criteria and provide justification for the modification. If modified validation criteria are not documented in the EPA-approved QAPjP and/or SAP prior to sampling, then an amendment to the QAPjP and/or SAP should be submitted and approved prior to the use of modified validation criteria.

The Lead Chemist should ensure that the appropriate data validators receive copies of the completed DQO Summary Forms. The DQO Summary Forms will identify the project DQOs, PE samples and validation tier. The Lead Chemist should also ensure that the field sampling notes for the sampling event are provided to the data validator for inclusion in the Tier I Validation Cover Letter or Data Validation Report for historical purposes.

7.8.1.2 Procuring Non-CLP Analytical Services

When required by EPA, the Lead Chemist is responsible for developing technical specifications for non-CLP analyses that may require modified validation criteria. They are also responsible for providing technical guidance to subcontracted laboratories to ensure that fully documented, technically valid, legally defensible and usable data are delivered to EPA. To this end, it is recommended that the Region I Laboratory Pre-Qualification Standard Operating Procedure from the Region I ARCS Delivery of Analytical Services Pilot Program, Final Report, Volume II. Appendices, 15 March 1994, (Attachment Q), be followed when procuring non-CLP analytical services (Attachment Q). It is also recommended that the Region I Laboratory Audit Standard Operating Procedure, from the Region I ARCS Delivery of Analytical Services Pilot Program, Final Report, Volume II. Appendices, 15 March 1994, (Attachment Q) be followed to audit laboratories performing non-CLP analyses, as well as to resolve technical problems and monitor corrective actions implemented by those laboratories.

7.8.1.3 Performance Evaluation Program

The Lead Chemist is responsible for requesting and maintaining an appropriate inventory of PE samples and for obtaining all pertinent information regarding their identification and content in accordance with the EPA Region I Performance Evaluation Program Guidance, July 1996, Revision (Attachment H) or most recent revision. The Lead Chemist must ensure that a single blind PE sample is included, whenever available, with every sample delivery group sent to a laboratory for each matrix, analytical parameter, and concentration level.

Upon receipt of the laboratory data package, the Lead Chemist is responsible for submitting to the EPA PE Chemist a copy of the tabulated PE sample results for scoring for those PE samples provided by EPA.

The PES Score Reports for EPA-provided PE samples and PE results for PE samples procured from commercial vendors must be evaluated with the laboratory data package during data validation. If only a Tier I validation is performed, the PES Score Reports for EPA-provided PE samples and PE results for PE samples procured from commercial vendors are evaluated in addition to performing the Completeness Evidence Audit.

7.8.1.4 Tracking Data Package Delivery

If a CLP data package is late, then the Lead Chemist is responsible for alerting the EPA RSCC. If the EPA RSCC is unable to resolve late data delivery within 2 weeks, the RSCC will contact the CLP-TPO to expedite problem resolution.

If data are late from a non-CLP laboratory subcontracted by an EPA Field Sampling Contractor, then the Lead Chemist should contact the laboratory to ascertain the problem and confirm a delivery date. Similarly, if data are late from the Contractor's own internal laboratory, then the Lead Chemist should contact the laboratory to ascertain the problem and confirm a delivery date. In both cases, the Lead Chemist is responsible for expediting late data.

7.8.1.5 Data Validation

The Lead Chemist is responsible for providing a copy of the project DQO Summary Form, the field sampling notes, the technical specifications for non-CLP analyses, the PES Score Reports and/or the QC acceptance ranges for commercial PE samples, and any Telephone Logs/Communication Forms generated prior to data validation to the data validator.

The Lead Chemist is responsible for reviewing and approving all Data Validation Memoranda written by corporate and subcontracted data validators. The Lead Chemist is responsible for all statements that their validators make in the Data Validation Memorandum concerning the final data assessments including the limitations and potential uses of validated data.

The Lead Chemist, as a designated Regional CLP representative, is responsible for contacting the laboratory to obtain necessary data resubmissions for non-compliant CLP data. They must adhere to the EPA-NE policy for contacting the laboratory, must document in Telephone Logs/Communication Forms all requests for data resubmissions and time frames, and must transmit copies of the Telephone Logs/Communication Forms to appropriate locations. Refer to the procedures in Section 9.1, The Regional/Laboratory Communication Network.

The Lead Chemist ensures that a Data Validation Report or a Tier I Validation Cover Letter is delivered to the EPA Site Manager within 21 days of the receipt of a data package from the laboratory or in accordance with the pre-approved site schedule. Expected delays in the delivery of the validation reports must be reported to the EPA Site Manager.

The Lead Chemist ensures that Fund-lead and PRP/Federal Facility oversight CLP and non-CLP Data Validation Reports (Tiers II and III) and Tier I Validation Cover Letters are correctly distributed within EPA-NE and to other Regions. See Section 13.0 for the list of recipients and correct distribution.

7.8.1.6 Reduced Payment/Data Rejection Recommendations

It is the responsibility of the EPA Field Sampling Contractor's Lead Chemist to ensure that all contractual defects and unresolved deliverable deficiencies are noted in the Data Validation Memorandum and on the ORDA/IRDA Form.

Any CLP or EPA-generated non-CLP data that are deemed to be contractually non-compliant (based on laboratory analytical technical specification) and unusable in making site decisions should be recommended for rejection, returned to the laboratory and payment denied. In this case, sample results are reported as "rejected" to the EPA Site Manager in the Data Validation Report. If only one analytical fraction is rejected, then only the data for that fraction should be returned to the laboratory and the remaining fractions should be validated in accordance with the guidance provided in Parts II - IV.

Any CLP or EPA-generated non-CLP data that are deemed to be contractually non-compliant and of reduced worth to the Region in terms of making site decisions should be recommended for reduced payment. In this case, sample results should be qualified in accordance with the guidance provided in Parts II-IV of this document.

7.8.1.6.1 CLP Data

The Lead Chemist is responsible for notifying the EPA Data Validation Chemist when CLP data are contractually non-compliant and unusable, in accordance with EPA-NE Standard Operating Procedures for Submitting Data for Reduced Payment/Data Rejection, September 1991 (Attachment I) or most recent revision and in accordance with the Federal Acquisition Regulation (FAR). The Lead Chemist is responsible for providing to the EPA Data Validation Chemist a letter describing the contractual non-compliances and providing supporting documentation within the time frame specified in the SOP mentioned above.

7.8.1.6.2 EPA-Generated Non-CLP Data (i.e., DAS Data)

The Lead Chemist is responsible for documenting contractual non-compliances, determining if they affect the potential usability of the data, and ensuring that EPA does not pay for unusable, non-compliant EPA-generated non-CLP data.

7.8.1.7 Data Validation Oversight and Implementation of Corrective Action

The Lead Chemist is responsible for responding to all requests for data validation oversight by the Region. The Lead Chemist reviews the "EPA-NE-generated Data Validation Oversight/Methods Review Memoranda" and directs the implementation of appropriate corrective actions. Subsequently, the Lead Chemist monitors the implementation and is responsible for the continued effectiveness of all corrective actions.

7.8.1.8 Qualifications of the Lead Chemist

The Lead Chemist should have a B.S. degree in chemistry or a related physical science and be a professionally trained analytical chemist with at least eight to ten years of combined inorganic and organic analytical experience which includes familiarity with GC/MS and ICP instrumentation. The Lead Chemist should have extensive knowledge of CLP methods, deliverables, and program operation as well as extensive knowledge of all other EPA program analytical methodologies, i.e., RCRA SW 846 methods, Drinking Water Program 500 series methods, ambient air and stack testing, etc., to enable them to recommend the appropriate methods and modifications for those methods for achieving project DQOs.

The Lead Chemist should have extensive knowledge of the most recent EPA-NE validation requirements, as specified in this document. The Lead Chemist must be technically able to identify the need to modify validation criteria when non-CLP analyses are performed and must be able to incorporate and document the modified validation criteria into an EPA-approved QAPjP and/or SAP.

7.8.2 Data Validator

This section specifically details the responsibilities of the EPA Field Sampling Contractor's data validator performing validation on data generated for Fund-lead sites or EPA oversight activities. However, many of the activities, roles, responsibilities and qualifications discussed below are applicable to non Fund-lead work performed by a PRP or Federal Facility as well as to Fund-lead work performed by another Federal Agency (i.e., ACOE) or a State.

7.8.2.1 Data Validation

Data validators must assess the analytical deficiencies and contractual non-compliances of a data package in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. They are responsible for using modified validation criteria when required by an EPA-approved QAPjP and/or SAP.

The data validator is responsible for obtaining resubmittals for non-compliant data from the laboratory. In the CLP system, only designated regional communication representatives may contact a CLP Laboratory (usually the Lead Chemist), therefore the data validator must contact the laboratory through their designated CLP communication representative or alternate.

The validator reviews the Data Quality Objectives for the project as documented in the QAPjP or SAP and DQO Summary Form and determines if the degree of "measurement error" associated with the data potentially compromises data usability. The driving force for data validation is that contractually compliant data are not always technically usable for making site decisions and that contractually non-compliant data are sometimes very usable. **Only the end user can determine actual usability of the data.**

The data validator must notify the Lead Chemist immediately if significant contractual deficiencies warrant recommendation for data rejection or reduced payment.

The data validator is responsible for using the appropriate DQO Summary Form and should contact the Lead Chemist if this document has not been provided.

The data validator must contact field samplers whenever necessary to obtain information to assess "sampling error". All communications must be documented in a Telephone Log/Communication Form and included in the Data Validation Report. If a copy of the field sampling notes was not provided, then the data validator should contact the Lead Chemist to obtain the notes.

The data validator must have the technical specifications for non-CLP analyses and any additional data quality criteria specified in the QAPjP or SAP in order to validate non-CLP data. If the technical specifications for non-CLP analyses and additional data quality criteria were not provided, then the data validator should contact the Lead Chemist to obtain the applicable technical specifications and data quality criteria.

The data validator must obtain the PES Score Reports for CLP and non-CLP analyses in order to validate the sample data. If PES Score Reports were not provided, then the data validator should contact the Lead Chemist to obtain the applicable PES Score Reports. If commercial PE samples were used, then the data validator should obtain the vendor's QC acceptance limits from the Lead Chemist in order to evaluate the PE sample results.

The data validator must obtain any Telephone Logs/Communication Forms generated prior to data validation for CLP and non-CLP analyses in order to validate sample data. If any Telephone Logs/Communication Forms generated prior to data validation for CLP or non-CLP analyses were not provided, then the data validator should contact the Lead Chemist to obtain any applicable Telephone Logs/Communication Forms generated prior to data validation.

The data validator generates a Tier I Validation Cover Letter with the following attachments in the order specified below: (Refer to Section 10 for complete description of Tier I Validation Cover Letter).

1. Cover Letter
2. Attachments
    a. CADRE-generated Data Summary Table of Unvalidated Data (not required if CADRE Review not performed)
    b. Data Validation Worksheet XI-Accuracy Check and EPA PE Score Reports and/or non-EPA PES results with Vendor PES QC Acceptance Limits
    c. Support Documentation
        i. Copy of non-CLP analytical method, e.g., DAS methods, modified EPA methods
        ii. Copies of Telephone Logs/Communication Forms for:
            - RSCC communications
            - Requests for laboratory data resubmissions/

clarifications
- Communications with samplers resolving sampling problems
- Communications with TPO/Lead Chemist to report contractually-deficient data for rejection/reduced payment
- Communications with EPA Site Manager concerning possible data rejection
- EPA Site Manager authorization for alternate DV tier

iii. Copies of data supporting recommendations for reduced payment resulting from CSF Audit and/or PE sample result evaluation

iv. Original data to support recommendations for data rejection/non-payment resulting from CSF Audit and/or PE sample result evaluation

v. Copies of field sampling notes and/or field report supplied by field sampler

vi. Copies of EPA-approved amendments to QAPjP and/or SAP describing modified criteria to be used for validating site data

d. CSF Completeness Evidence Audit

e. DQO Summary Form

The data validator generates a Data Validation Report, applicable to Data Validation Tiers II and III, that consists of the following components in the order specified below: (Refer to Section 11 for a description of each of the Data Validation Report components).

1. Organic Regional Data Assessment/Inorganic Regional Data Assessment (ORDA/IRDA) Form
2. Data Validation Memorandum
   a. Narrative
   b. Table I-Qualifier Recommendation Summary Table
   c. Table II-Overall Evaluation of Data
   d. Table III-Tentatively Identified Compounds
   e. Data Summary Tables
3. Standard Data Validation Worksheets
   a. Manual
   b. Automated Data Review Reports (i.e., CADRE)
4. Support Documentation
   a. Copy of non-CLP analytical method, e.g., DAS methods, modified EPA methods
   b. Copies of EPA PES Score Reports and/or non-EPA PES results with Vendor PES QC Acceptance Limits
   c. Copies of Telephone Logs/Communication Forms for:
      - RSCC communications
      - Requests for laboratory data resubmissions/clarifications
      - Communications with samplers resolving sampling problems
      - Communications with TPO/Lead Chemist to report contractually-deficient data for rejection/reduced payment
      - Communications with EPA Site Manager concerning possible

data rejection
- EPA Site Manager authorization for alternate DV tier

d. Copies of data supporting recommendations for reduced payment resulting from CSF Audit and/or PE sample result evaluation
e. Original data to support recommendations for data rejection/non-payment identified from Tier II or Tier III data validation
f. Copies of field sampling notes and/or field report supplied by field sampler
g. Copies of EPA-approved amendments to QAPjP and/or SAP describing modified criteria to be used for validating site data

5. CSF Completeness Evidence Audit
6. DQO Summary Form

The data validator is responsible for implementing all corrective actions required by the contractor Lead Chemist in response to EPA-NE data validation oversight findings.

7.8.2.2 Qualifications of the Data Validator

7.8.2.2.1 Senior Validator

The senior data validator should have a B.S. or B.A. degree in chemistry or a related physical science and be a trained analytical chemist specializing in a particular discipline such as GC pesticides, GC/MS organics, or ICP metals. The validator should have at least five years of related analytical/instrumentation experience working with laboratory instrumentation and analyzing multi-media environmental samples (soil, water, oil, waste, fly ash, biological tissue and air). Data validation experience cannot be substituted for any of the five years required laboratory experience.

The senior validator should have extensive knowledge of the most recent EPA-NE validation requirements as specified in this document. The validator must also be capable of applying modified validation criteria when required by the EPA-approved QAPjP and/or SAP.

All Data Validation Reports must undergo internal peer review by the organization performing the validation. A senior validator must perform secondary review of all Data Validation Reports prepared by junior validators. If a senior validator prepares a Data Validation Report, then a different senior validator or other qualified senior chemist must peer review that Report.

7.8.2.2.2 Junior Validator

The junior validator should have a B.S. or B.A. degree in chemistry or a related physical science and be a trained analytical chemist specializing in a particular discipline such as GC pesticides, GC/MS organics, or ICP metals. The validator should have at least two years of related analytical/instrumentation experience working with laboratory instrumentation and analyzing multi-media environmental samples (soil, water, oil, waste, fly ash, biological tissue and air). Data validation experience may be substituted for some of the two years of required laboratory experience. However, the junior validator must have at least six months of instrumentation experience in the areas described above.

The junior validator should have extensive knowledge of the most recent EPA-NE validation requirements as specified in this document. The validator must also be capable of applying modified validation criteria when required by the EPA-approved QAPjP and/or SAP.

8.0 INFORMATION REVIEWED DURING THE DATA VALIDATION PROCESS

Figures 1, 2 and 3 illustrate the normal flow of the data validation process. Sources of information are noted, as well as communication channels and key decision points in the validation process. To evaluate data quality and the extent of "measurement error", the following items must be incorporated into the review of sample data: project scoping information documented in the EPA-approved SAP and/or QAPjP; analytical results presented in the laboratory data package; field sampling information; Contract Compliance Screening results; and Performance Evaluation Sample results.

8.1 Project Scoping Information

8.1.1 Objective

The QAPjP and/or SAP is a planning document that provides project history and background data and documents the project DQOs and sample custody procedures, evidentiary

requirements, analytical methods, laboratory QA/QC, laboratory documentation and deliverables, and data validation criteria and validation tier to be used for the project. **These items should be agreed upon by all end users in the initial planning phase of the project.**

8.1.2 Requirements

The DQOs should be fully discussed and documented in the EPA-approved QAPjP and/or SAP and identified in abbreviated format in a DQO Summary Form. A copy of the SAP and/or QAPjP should be available to the validator and should include: the data validation criteria to be used (refer to Figure 4), reference to the Tier level of validation to be performed, modified validation criteria to be used (if any) or alternate validation criteria, i.e., USATHAMA and split sample comparability criteria and decision trees to be used in assessing split sample analyses. Project documents should detail the exact number of samples, types of samples (field and QC), PE samples, sample matrices, sample locations/descriptions and knowledge of any positive detects from prior site sampling efforts. Background information on the site is essential to identifying potential usability issues. The EPA Site Manager or Field Sampling Contractor Site Manager are the best sources for additional site information.

8.1.3 Evaluation

a. The validator should ascertain from the EPA-approved QAPjP, SAP and/or DQO Summary Form which validation criteria were selected by the end users. The validator should ascertain whether the validation criteria contained in the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses are to be used without modification to validate site data, or whether modified EPA-NE validation criteria are to be utilized. Also, the validator must ascertain from the project planning documents if alternate validation criteria, i.e., USATHAMA are to be used for data validation.

b. If the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses were selected as the validation criteria by the end user, then the data validator should ascertain from the EPA-approved QAPjP, SAP and/or DQO Summary Form, the validation tier that are to be used to evaluate the project data.

c. The validator should determine if the correct analytical method as cited in the EPA-approved SAP, QAPjP, and/or DQO Summary Form was used for analysis and if required detection/quantitation limits were achieved.

d. The validator should be familiar with the project DQOs, as summarized on the DQO Summary Form, in order to identify potential usability issues for the end users.

e. Comparability criteria for split sampling should be presented in the EPA-approved QAPjP and/or SAP. Split sampling analyses are performed for PRP/Federal Facility oversight using standardized EPA (full protocol) methods. Field screening confirmatory analyses are also performed using standardized EPA methods. The % Difference Criteria between data sets should be based on the following standard equations to ensure consistency in presenting and assessing split data. Note: Comparability criteria should be based on historical data generated for the site and should take into account associated field precision. Homogenous matrices may allow for lower % Difference Criteria while heterogeneous matrices may necessitate higher % Difference Criteria to be set. A discussion and justification for selection of

comparability criteria should be included in the EPA-approved QAPjP and/or SAP.

Split Sampling Analyses

$$\textit{Equation 3: \% Difference (Split Sampling)} = \frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

C_1 = Concentration Determined by EPA Oversight Analysis

C_2 = Concentration Determined by PRP, Federal Facility, or State Analysis

Note that this equation assumes that values generated by EPA and those values generated by equivalent methods used by the PRP (or other entities) are equally accurate. While this may not always be true, the equation serves to standardize reporting conventions and to promote data comparability. Note that this equation retains the sign of the difference, thus absolute numbers are not used.

Confirmatory Analyses

$$\textit{Equation 4: \% Difference (Confirmatory Analysis)} = \frac{C_1 - C_2}{C_1} \times 100$$

C_1 = Concentration Determined by Full Protocol Confirmatory Analysis

C_2 = Concentration Determined by Field Screening Analysis

Note that this equation assumes that values generated by the full protocol confirmatory method are more accurate than those generated by field screening methods. While this may not always be true, the equation serves to standardize reporting conventions and to promote data comparability. Note that this equation retains the sign of the difference, thus absolute numbers are not used.

8.1.4 Action

a. If no validation tier or an inappropriate validation tier has been referenced in the DQO Summary Form, then the validator should contact the Lead Chemist who will obtain clarification/direction from the EPA Site Manager. The validator and/or Lead Chemist should document this call in a Telephone Log. The validator should note in the first paragraph of the Data Validation Memorandum if, in the validator's opinion, the validation Tier selected during project scoping does not meet the project DQOs.

b. i. If the EPA-approved QAPjP and/or SAP does not cite specific validation criteria, then the validator must validate site data according to the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. The validator should note in the first paragraph of the Data Validation Memorandum that the data has been validated in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses.

ii. If modified or alternate validation criteria have been described in an EPA-

approved QAPjP and/or SAP, then the validator should note these modified or alternate validation criteria in the first paragraph of the Data Validation Memorandum and copies of the relevant QAPjP or SAP pages should be attached to the Memorandum as supporting documentation.

iii. Alternatively, if the validator determines that modified or alternate validation criteria are necessary to validate the site data in order to support project DQOs and/or the use of non-CLP methods and those criteria have not been included in the EPA-approved site QAPjP/SAP, then an amendment to the QAPjP or SAP must be submitted to EPA and approved prior to validation. The amendment should be noted in the first paragraph of the Data Validation Memorandum and a copy attached to the Memorandum as supporting documentation.

c. If the data are contractually compliant but unusable because the wrong analytical method was selected and/or utilized, then this should be noted in the Data Validation Memorandum and an alternative method should be identified for future site work.

d. If the DQO Summary Form was not provided, then the validator should contact the Lead Chemist to obtain the Form. If a DQO Summary Form was not completed prior to the sampling event, then this should be noted in the first paragraph of the Data Validation Memorandum.

e. If split sampling criteria for oversight analyses or field screening confirmatory criteria for confirmation analyses have not been established, then the validator should contact the Lead Chemist who will obtain clarification/direction from the EPA Site Manager. The validator and/or Lead Chemist should document this call in a Telephone Log.

8.2 The Data Package

8.2.1 Objective

The CLP Complete SDG File (CSF) data package is designed to provide all necessary documentation to verify compliance with the Statement of Work (SOW) and to permit verification of the accuracy and defensibility of the reported results. It contains all the original data generated for the data package.

A non-CLP data package should also provide all necessary documentation to verify compliance with the analytical method and/or contracts/subcontracts to permit verification of the accuracy and defensibility of the reported results. It should contain all the original data generated for the data package.

8.2.2 Requirements

A list of the required CLP deliverables may be found in the appropriate CLP SOWs.

Required non-CLP deliverables may be found in the appropriate methods and/or contracts/subcontracts developed for the analytical service. Most data collection activities will require all original data and a complete case file purge. See Attachment G for Training Manual for Reviewing Laboratory Data Package Completeness, June 1994.

8.2.3 Evaluation

Procedures for the evaluation of specific deliverables are detailed in Parts II, III, and IV of this document.

8.2.4 Action

When contract-required information necessary for data validation is missing from the data package, then the validator should arrange for the Lead Chemist to contact the laboratory to obtain the omitted data according to the procedure referenced in Section 9.2.

Only authorized personnel that are designated Regional CLP representatives may contact CLP laboratories. Only prime contractors may contact their subcontracted laboratories due to privity of contract.

8.3 Field Sampling Information

8.3.1 Field QA/QC Samples

8.3.1.1 Objective

Field QA/QC samples, such as trip blanks, equipment blanks, bottle blanks, and field duplicates enable data validators to identify some, but not all, of the "sampling error" associated with the project. Specifically, the field QA/QC assist the data validator in evaluating sampling conditions, techniques, field precision, and sample homogeneity.

8.3.1.2 Requirements

All field QA/QC sample requirements should support the project DQOs and should be documented in the EPA-approved QAPjP and/or SAP and DQO Summary Form.

At a minimum, equipment blanks and field duplicates must be included at a frequency of five percent per analytical parameter/matrix/sampling team.

At a minimum, volatile trip blanks are required at a frequency of one per shipment cooler.

At a minimum, temperature indicator blanks are required at a frequency of one per shipment cooler and should be clearly identified as temperature indicator blanks.

Bottle blanks are used to verify the cleanliness of a specific Lot Number of bottles and should be included at the discretion of the sampling team. At a minimum, bottle blank analyses should be performed on one bottle per container type per lot. The Lot Number for the bottle blank should be noted on the Traffic Report and/or Chain-of-Custody Form and in the field sampling notes.

8.3.1.3 Evaluation

Note that for large projects containing several sample delivery groups (SDGs) with many field samples and inter-related QC samples, the EPA Field Sampling Contractor may assign a Project Chemist to coordinate data collection and review. For large projects where the data validator alone may not be able to fully assess field QA/QC compliance with the EPA-approved QAPjP and/or SAP, the assigned Project Chemist should review all the individual project Data Validation Reports to assess project compliance for field QA/QC requirements.

The validator should confirm that the required field QA/QC samples were provided to the laboratory at the proper frequency.

It is recommended that the results for each bottle blank (used to verify the cleanliness of a specific Lot Number of bottles) be evaluated prior to use of bottles from that Lot Number for field sampling.

The validator should evaluate contamination found in the equipment, trip and bottle blanks as part of the laboratory method blank review. Similarly, field duplicate precision should be evaluated concurrently with laboratory duplicate (MS/MSD) precision data to determine whether precision problems were laboratory or field related.

8.3.1.4 Action

a. If the field QA/QC samples were not provided to the laboratory in accordance with the frequency specified in the EPA-approved QAPjP/SAP, then the validator should note this deviation in the Data Validation Memorandum and the EPA Field Sampling Contractor should initiate corrective action procedures.

b. If the laboratory has not provided results for one or more of the samples that were shipped, the validator should check the Data Package Narrative and Telephone Logs/Communication Forms for a possible explanation (broken sample, insufficient sample volume for reanalysis, etc.). If no explanation is found, then the validator should contact the Lead Chemist who in turn contacts the RSCC to further investigate and resolve CLP issues. For non-CLP samples, the validator should contact the appropriate personnel to resolve the problems.

c. The field sampler must be informed immediately by the validator, and the call documented in a Telephone Log/Communication Form, if any of the following problems are noted:

- trip blanks, equipment blanks, bottle blanks or field duplicates are not identified on the Traffic Report/Chain-of-Custody Form
- anomalies such as Traffic Report numbers being listed twice, etc.
- high contamination in equipment, trip, or bottle blanks that is not present in the laboratory blanks

8.3.2 Sample Descriptions

8.3.2.1 Objective

All sample locations should support the project DQOs and be documented in the EPA-approved QAPjP and/or SAP.

Sample descriptions/locations/sampling dates are necessary information for preparing the Data Summary Tables and for the evaluation of holding times. In addition, sample descriptions are useful as supplementary information for the consideration and discussion of matrix problems and chemical constituents identified in particular samples.

8.3.2.2 Requirements

All sample locations should be sampled and numbered in accordance with the EPA-approved

QAPjP and/or SAP.

For CLP data packages, copies of properly completed Traffic Reports (Attachment J, Form vi) are mandatory deliverables.

Copies of Chain-of-Custody Forms (Attachment J, Form v) must be included in all non-CLP data packages and must contain the date of sampling, sample numbers, as well as the sampling locations.

The sampler or Project Chemist should provide a copy of the field sampling notes to the Lead Chemist or data validator to be included in the Data Validation Report. In situations where sampling events extend over a period of weeks producing two or more SDGs and generate numerous pages of field log book notes, the field notes should be copied only once, included in one Data Validation Report and that Data Validation Report should be referenced by Case, SDG, and date of Data Validation Report.

8.3.2.3 Evaluation

Again, note that for large projects containing several sample delivery groups (SDGs) with many field samples and interrelated QC samples, the EPA Field Sampling Contractor may assign a Project Chemist to coordinate data collection and review. For large projects where the data validator alone may not be able to fully assess field QA/QC compliance with the EPA-approved QAPjP and/or SAP, the assigned Project Chemist should review all the individual project Data Validation Reports to assess project compliance for field QA/QC requirements.

The validator should confirm from the EPA-approved QAPjP and/or SAP and DQO Summary Form that all sample locations have been sampled and that there are sample results for all locations.

Traffic Reports and COC Forms must be compared for consistency with respect to the designation of quality control samples (blanks and duplicates) and the identification numbers for field samples.

The data validator is <u>not</u> responsible for evaluating field sampling notes. They are to be included in the Data Validation Report to be used by the end user to assess data usability and to support potential litigation.

8.3.2.4 Action

a. If sample locations are not sampled in accordance with the EPA-approved QAPjP and/or SAP, then the validator should note this deviation in the Data Validation Memorandum and the EPA Field Sampling Contractor should initiate corrective action procedures.

b. If discrepancies on the COC or Traffic Report Forms are identified, then the sampler must be contacted for resolution. The resolution must be documented in a Telephone Log (Attachment J, Form iii) and the Telephone Log must be included in the Data Validation Report.

c. If information is illegible (sample descriptions, locations, sampling date, etc.), then the sampler must be contacted to provide a legible copy of this information.

d. If Traffic Reports or COC Forms are missing, then the laboratory should be contacted to obtain this required deliverable. If the laboratory cannot provide this required

deliverable, then the sampler must be contacted to provide a copy of these documents. If the Traffic Reports or COC Forms were not properly completed and/or signed by the laboratory personnel, then the laboratory must be contacted to obtain a written letter detailing the deficiency. This letter should be included in the CSF/data package as part of the site record and a copy included in the Data Validation Report.

e. If the field sampling notes are not provided prior to validation, then the validator must obtain a copy from the Lead Chemist for inclusion in the Data Validation Report.

8.4 CLP Laboratory Contract Compliance Screening (CCS)

8.4.1 Objective

CCS provides a high volume assessment of CLP deliverables for compliance with some, but not all, contract requirements. Its primary application is to determine payment recommendation. Because of this direct link to payment, CCS fosters a somewhat timely resolution of contractual problems.

8.4.2 Requirements

The NPO Sample Scheduling and Coordination Contract (currently named CLASS) performs CCS on all low/medium organic and inorganic data packages submitted through the CLP. Laboratories are required by EPA to submit all identified missing data, and resubmit or explain all data identified as non-compliant during CCS. To date, CCS has not been performed on CLP dioxin data packages. Also, CCS is not performed on EPA-generated non-CLP data.

8.4.3 Evaluation

CCS may be used, when available, during data validation to evaluate those technical criteria that are also contractual criteria and to determine the completeness of the data package. If available, CCS results should be previewed to determine important compliance issues. The validator should compare the findings of CCS to the laboratory data package in the course of data validation. An example regional CCS Report is contained in Attachment K.

8.4.4 Action

a. If the CCS information is not provided with the data package, it can be requested through the RSCC. CCS information is not necessary in order to perform validation because the validator assesses contractual compliance during the validation process.

b. If the CCS information indicates significant contractual non-compliance which coincides with poor technical quality and potentially limits the usability of the data, then the validator should recommend reduced payment or rejection of data (See Attachment I).

c. When a contract-required reanalysis or deliverable was noted as missing by CCS, the validator should contact the laboratory to ascertain the expected delivery date.

8.5 Performance Evaluation Samples

8.5.1 Objective

The EPA-NE Performance Evaluation (PE) Program essentially serves three functions: (1) PE

samples may be used in laboratory pre-award evaluations to identify a community of technically capable laboratories, (2) PE samples are used to evaluate laboratory performance over a period of time, (3) PE samples are included in a sample group to provide information on a laboratory's ability to accurately identify and quantitate analytes of interest during the period of sample analysis. In the third function, the PE program works in conjunction with the Region I Tiered Validation approach.

8.5.2 Requirements

EPA-NE established a Performance Evaluation Program on July 1, 1993. A copy of the most recent revision of the EPA Region I Performance Evaluation Program Guidance, July 1996, Revision, may be found in Attachment H. The document describes the purpose, use, quality assurance documentation requirements, responsibilities, and general procedures for utilization of the EPA-NE PE Program and includes a list of EPA-PE samples that are currently available through the EPA-NE QA Unit and a list of commercially available PE samples.

It is recommended that blind PE samples be included in each sample set sent to a laboratory, whenever appropriate, to assist in evaluating analytical data quality. One PE sample should be included for each sample matrix, parameter, and concentration level for each Sample Delivery Group (SDG) sent to a laboratory. The PE samples should be counted as field samples in the 20 sample SDG. The use of PE samples should be specified as a quality control measure at the planning stage of each project and documented in the EPA-approved QAPjP and/or SAP.

8.5.3 Evaluation

Upon receipt of the laboratory data package, the Lead Chemist or validator should determine if a PE sample was included for each sample matrix, parameter, and concentration level for each SDG sent to the laboratory. Next, the laboratory's EPA PE sample results must be submitted by the EPA Field Sampling Contractor performing data validation to the EPA-NE PE Chemist for scoring. In the situation where data validation is performed by a subcontractor, only the prime contractor may submit PE results to EPA.

For Tier II and Tier III validations, the data validator must incorporate the EPA-PE sample score results into the evaluation of data in accordance with Section XI in Parts II, III and IV of this document.

For Tier I validations, EPA-PE sample results must also be scored and evaluated in accordance with the guidance noted above to determine whether laboratory problems exist and whether a higher validation tier is warranted based upon analytical problems identified by the PES.

If non-EPA (commercial) PE samples are reported in the data package, then the validator should assess the results of the PE samples based upon the vendor's QC acceptance limits in accordance with Section XI in Parts II, III, and IV of this document.

8.5.4 Action

a. If PE samples were not submitted by an EPA Field Sampling Contractor in accordance with the frequency requirements stated in the Region I policy, then the validator should note this deficiency in the Data Validation Memorandum. If an EPA Field Sampling Contractor consistently fails to comply with Region I policy, corrective action will be required.

b. If PE sample results are acceptable or do not indicate major laboratory performance problems, then the validator should complete the Tier I, II or Tier III validation.

c. If PE sample results indicate major laboratory performance problems and are unacceptable and a Tier II or Tier III validation was required, then the validation should be completed to ascertain the source of the analytical error. If the data quality is suspect, then the data should be recommended for reduced payment or, alternatively, rejected as unusable, returned to the laboratory and payment denied.

d. If PE sample results are unacceptable and a Tier I validation was required, then the validator should document this in the Tier I Data Validation Cover Letter and consider the need to upgrade the tier level to determine if the data is unusable and should be rejected. The validator must receive authorization from the EPA Site Manager to upgrade the data validation tier prior to doing so. Authorization must be documented in a Telephone Log and included in the Tier I Validation Cover Letter, or (if the validation tier was upgraded) in the Data Validation Report.

8.6 Computer-Aided Data Review and Evaluation (CADRE) Reports

8.6.1 Objective

CADRE is a computer program that was developed to perform automated validation of organic and inorganic Low/Medium CLP data that have been entered into the national CLP Analytical Results Database (CARD). The automated review criteria are based on the USEPA CLP National Functional Guidelines for Organic Data Review, February 1994, and the USEPA CLP National Functional Guidelines for Inorganic Data Review, February 1994. In most cases, CADRE criteria are similar to Region I Tier II validation criteria. Where the criteria are different, the CADRE program has been customized for EPA-NE to incorporate EPA-NE Validation criteria. For those additional validation criteria, e.g. field duplicates, that are not assessed by CADRE, a Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, February 1995 (Attachment L) is available to assist data validation completion.

Currently, this automated validation program is available only for EPA CLP Fund-lead and oversight use. However, in the future, computer-assisted data validation for EPA-generated non-CLP data may be available.

8.6.2 Requirements

Eventually, all EPA-NE CLP Organic and Inorganic Low/Medium SOW laboratory data packages will be validated using CADRE. Currently, CADRE reports for CLP Organic Low/Medium Volatile and Semivolatile analyses are provided to the EPA Field Sampling Contractor along with the CSF/CLP laboratory data package to assist in data validation.

8.6.3 Evaluation

Tier I validation does not include the review of CADRE Reports. The validator should include the CADRE-generated Data Summary Table of **NOT VALIDATED DATA** as an attachment to the Tier I Validation Cover Letter.

Tier II and III validations include the use of CADRE reports. Refer to the Guidance Document For Completing Region I Data Validation Utilizing CADRE Data Review, February

1995, or most recent revision for guidance on data validation completion in conjunction with CADRE review.

8.6.4 Action

Occasionally laboratory electronic deliverables are unavailable, incomplete or of such poor quality that they cannot be used by the CADRE program. If a Low/Medium Organic or Inorganic CLP CSF is received by the EPA Field Sampling Contractor from EPA without a CADRE report but with a notification that manual validation is required, then the EPA Field Sampling Contractor must perform manual validation for that CLP CSF.

If the CADRE report is incomplete, then the validator should contact the EPA DV Chemist to obtain the complete report.

9.0 COMMUNICATION NETWORKS

9.1 The CLP-Regional Sample Control Center (RSCC) Communication Network

9.1.1 Objective

9.1.1.1 CLP

The Regional Sample Control Center (RSCC) is synonymous with the Regional Sample Control Coordinator (RSCC) for EPA-New England. The RSCC places all regional requests for CLP sample analyses through the NPO Sample Scheduling and Coordination Contract (currently named CLASS). Requests for CLP analyses may be initiated by EPA Site Managers or Field Sampling Contractors doing Fund-lead or PRP/Federal Facility oversight, or States (or their contractors) performing Fund-lead work under Cooperative Agreements with EPA, or other Federal Agencies (or their contractors), i.e., the Army Corp of Engineers, performing Fund-lead work under Interagency Agreements.

The RSCC tracks CLP samples originating from Region I, regardless of the organization that collects them, in the New England Sample Tracking System (NESTS) database.

9.1.1.2 Non-CLP

The RSCC does not schedule non-CLP analytical services for EPA Field Sampling Contractors, States or other Federal Agencies. However, the RSCC schedules non-CLP analytical services that are obtained directly through any of EPA-New England's regional environmental analytical procurements.

The RSCC tracks all non-CLP samples collected by EPA Field Sampling Contractors doing Fund-lead or PRP/Federal Facility oversight work, i.e., through the DAS mechanism.

The RSCC does not track non-CLP samples collected by the States or other Federal Agencies doing Fund-lead work under Cooperative Agreements and Interagency Agreements, respectively.

9.1.2 Requirements

9.1.2.1 CLP

EPA Field Sampling Contractors must submit quarterly CLP sample projections to the RSCC. Completed DQO Summary Forms for each sampling event should accompany the quarterly sample projections and must be submitted prior to sampling. To reserve sample slots the EPA Field Sampling Contractor must follow the procedures outlined in 9.1.3.1.

States and Federal Agencies that utilize CLP for sample analysis must also submit quarterly CLP sample projections to the RSCC. Completed DQO Summary Forms for each sampling event must accompany the quarterly projections. To reserve sample slots States and other Federal Agencies must follow the procedures outlined in 9.1.3.1.

If EPA personnel will be collecting samples at a site for CLP analyses, then the Site Manager must notify the RSCC and submit a completed DQO Summary Form by 5:00 p.m. on the Tuesday before the scheduled sampling event.

9.1.2.2 Non-CLP

EPA Field Sampling Contractors that procure non-CLP analytical services, or use their own corporate laboratory to analyze non-CLP samples or use the EPA regional laboratory for non-CLP analyses must follow the sample tracking procedures referenced in 9.1.3.2. Completed DQO Summary Forms must be submitted to the RSCC prior to the sampling event.

States, other Federal Agencies, PRPs and Federal Facilities are not required to report non-CLP sample tracking information to the EPA at this time. However, States, other Federal Agencies, PRPs and Federal Facilities should maintain non-CLP sample tracking information in their site files to assist EPA in tracking non-CLP data upon EPA's request.

9.1.3 Procedure

9.1.3.1 CLP

EPA Sampling Field Contractors, EPA Site Managers, States and other Federal Agencies requiring CLP services must contact the RSCC in accordance with The Regional Sampl Control Center Guidance for The Contract Laboratory Program (CLP) and Delivery of Analytical Services (DAS) Program for EPA-New England, July 1996 (Attachment P).

9.1.3.2 Non-CLP

EPA Field Sampling Contractors that procure their own non-CLP analytical services, or obtain non-CLP services from their corporate laboratory or from the EPA-NE regional laboratory must report the sample tracking information to the RSCC in accordance with The Regiona Sample Control Center Guidance for The Contract Laboratory Program (CLP) and Delivery of Analytical Services (DAS) Program for EPA-New England, July 1996 (Appendix P) and the DAS Sample Tracking and Scheduling Standard Operating Procedure, from the Region ARCS Delivery of Analytical Services Pilot Program, Final Report, Volume II. Appendices, 15 March 1994, (Attachment Q).

States and other Federal Agencies that procure non-CLP analytical services or obtain non-CLP services from their organizations' own laboratory should schedule and track samples in accordance with their organizations' procedures.

9.1.4 Action

9.1.4.1 CLP

CLP analysis requests by an EPA Field Sampling Contractor, State or other Federal Agency must be made by 5:00 p.m. the Tuesday before sampling. If a request is made later than this time, sample analysis slots cannot be guaranteed. Also, if DQO Summary Forms are not submitted prior to the sampling date, sample analysis slots cannot be guaranteed.

If an EPA Field Sampling Contractor consistently fails to allow for sufficient lead time in scheduling CLP samples and/or fails to accurately project quarterly CLP analytical needs, and/or fails to submit the associated DQO Summary Forms, corrective action will be required.

If a State or other Federal Agency performing Fund-lead work fails to allow for sufficient lead time in scheduling CLP samples and/or fails to submit the associated DQO Summary Forms, corrective action will be required.

9.1.4.2 Non-CLP

If an EPA Field Sampling Contractor, performing Fund-lead work or PRP/Federal Facility oversight, fails to provide the required non-CLP sample tracking information and/or the associated DQO Summary Form, corrective action will be required.

If States, other Federal Agencies, PRPs or Federal Facilities fail to schedule or track non-CLP samples correctly, corrective action should be initiated by that organization.

9.2 The Regional/Laboratory Communication Network

9.2.1 Objective

9.2.1.1 CLP

In January 1983, the CLP National Program Office established a system of direct communication between the regions and CLP laboratories as a routine method for regional data validation staff to obtain answers to technical questions concerning program data in the timeliest and most direct manner possible.

9.2.1.2 Non-CLP

EPA Field Sampling Contractors, States and other Federal Agencies performing Fund-lead work and/or PRP/Federal Facility oversight should establish a direct communication system with their contractor and/or subcontractor laboratories (as appropriate based upon privity of contract) to ensure timely resolution of technical issues.

For non Fund-lead sites, PRPs and Federal Facilities should also establish a direct communication system with their contractor and/or subcontractor laboratories (as appropriate based upon privity of contract) to ensure timely resolution of technical issues.

9.2.2 Requirements

The requirements for the CLP system are as follows:

a. Regional contact with CLP laboratories is permissible only after laboratory data submission.

b. Questions involving data delivery, contractual requirements, procedural recommendations, and other general CLP matters are to be referred to the RSCC, the NPO Sample Scheduling and Coordination Contract (currently named CLASS), or to program management (i.e., EPA-NE CLP-TPO) as appropriate.

c. Reanalysis requests originating from the data validator must be channeled by the EPA Field Sampling Contractor Lead Chemist through the EPA-NE CLP-TPO or EPA DV Chemist.

d. Only authorized personnel that are designated Regional CLP representatives may contact CLP laboratories, and they may contact only specified laboratory personnel.

To become a designated Regional CLP representative or alternate, the candidate's name and resume must be submitted to the CLP-TPO for review. Upon approval of the candidate, the CLP-TPO will notify CLASS for inclusion on the Region I CLP representatives list.

Similar requirements should exist for a non-CLP communication system.

9.2.3 Procedure

9.2.3.1 CLP

a. The entire data package should be assessed to determine if any of the four Action items listed below in Section 9.2.4 are a problem within the laboratory data package.

b. A list of required data resubmissions and analytical clarifications should be faxed to the laboratory prior to initiating the call.

c. The designated Regional CLP representative should call the laboratory, discuss each item on the faxed list, and establish a due date for resubmissions. The time frame for resubmission should be limited to seven days.

d. All conversations between the regional representatives and the CLP laboratories should be recorded by both the laboratories and the regional representatives on the Telephone Log or Regional/Laboratory Communication Form (Attachment J, Form iii).

e. The original Telephone Log/Communication Form is included in the Data Validation Report or Tier I Validation Cover Letter sent to the EPA Site Manager. One copy of the Telephone Log/Communication Form is forwarded by the EPA Field Sampling Contractor to each of the following:

- EPA NPO Sample Scheduling and Coordination Contract (currently named CLASS)
- The EPA-NE CLP-TPO (their copy to be included in the Data Validation Report or Tier I Validation Cover Letter)
- The CLP laboratory
- EPA-NE RSCC

f. Resubmitted data should be marked as "additional data" by the CLP laboratory. All resubmitted and/or omitted data should be submitted to the Region accompanied by a

revised DC-2 form.

g. If data resubmissions or verbal clarifications are not received within the specified timeframe, then the Regional CLP representative should contact the laboratory every day for 7 days.

h. If the information is still not received within the additional 7 days, then the Regional CLP representative should contact the CLP-TPO for follow-up action.

9.2.3.2 Non-CLP

a. For Fund-lead sites and PRP/Federal Facility oversight, all conversations between EPA personnel, EPA contractors, States, or other Federal Agencies with non-CLP laboratories should be recorded by both the non-CLP laboratories and the EPA/EPA Contractor/State/Other Federal Agency contacts.

b. For non Fund-lead sites, all conversations between PRPs, other Federal Agencies, or their contractors, with non-CLP laboratories should be recorded by both the non-CLP laboratories and the PRP/Other Federal Agency/Contractor contacts.

c. Copies of the Telephone Log or Regional/Laboratory Communication Form should be:

- Included in the Data Validation Report or Tier I Validation Cover Letter
- Sent to the laboratory
- Retained in the site file

9.2.4 Action

The four types of problems that require direct contact between the designated Regional representatives and the laboratory for resolution of laboratory data package problems are illustrated in Figures 2 and 3 and are described below:

a. In the case of missing or illegible deliverables, the validator should contact the laboratory through their designated Regional CLP representative to establish and record the expected due date for the requested deliverables.

b. i. When a CLP contract required reanalysis, is missing, the validator should check the CCS report, if available, to see if the problem was noted. If so, the designated Regional CLP representative should contact the laboratory to ascertain the expected due date. If the problem was not noted by CCS, the validator and/or Lead Chemist, in conjunction with the EPA Site Manager, must decide whether initiation of a reanalysis request would provide usable data (weighing a consideration of holding times, etc.). To initiate a CLP reanalysis request, the validator or Lead Chemist must first contact the CLP-TPO or EPA DV Chemist. If the TPO deems reanalysis appropriate, a reanalysis request form will be forwarded by the TPO to the CLP-APO for that laboratory.

ii. When a non-CLP contract required reanalysis for Fund-lead and PRP/Federal Facility Oversight work is missing, the EPA Field Sampling Contractors, States and other Federal Agencies should contact their contractor and/or subcontractor laboratory (as appropriate based upon privity of contract) to

ascertain the expected due date and ensure timely delivery of reanalysis results.

iii. When a non-CLP contract required reanalysis for non Fund-lead work is missing, the PRP, other Federal Agency, or their contractors should contact their contractor and/or subcontractor laboratory (as appropriate based upon privity of contract) to ascertain the expected due date and ensure timely delivery of reanalysis results.

c. Clarification of discrepancies or errors in the reported data usually requires correction and resubmission of results by the laboratory. If the laboratory does not agree with the error, then the validator should double check his/her work to ensure the accurate reporting and qualification of data. If the laboratory is still found to be in error but will not agree with the error, then the validator should use professional judgment to qualify the data.

d. In some cases, it may be necessary to have the laboratory provide certain explanations or detail conditions of analysis that do not correspond to any of the contract or method-required deliverables. In such cases, a verbal answer, documented in a Telephone Log/Communication Form by the designated Regional representative, is all that is contractually-required of the laboratory.

9.3 The CLP-TPO Communication Network

Similar to the communication networks described above, CLP-TPO communications involve contact with CLP Administrative Project Officers, CLP Contract Officers, CLP laboratories, the NPO contractors (CLASS and QATS) and the EPA Field Sampling Contractors' Lead Chemists. The CLP-TPO receives numerous QA reports from the NPO. Those which relate directly and specifically to CLP data validation will be forwarded to contractors responsible for data validation as appropriate.

Inter-regional questions or problems with CLP laboratory performance are referred to TPOs for resolution. For example, if a Region I data validator uncovers a possible contamination problem in a CLP laboratory assigned to Region II, the problem is first referred to the Region I CLP-TPO who then contacts the CLP-TPO in Region II to resolve the problem.

It is recommended that the CLP-TPO be notified of all problems and requirements for a particular case at one time. If there is an urgent requirement, the CLP-TPO may be contacted by phone to expedite corrective action. A copy of the Data Validation Report with the ORDA/IRDA Form as a cover page must be submitted to the CLP-TPO to provide documentation of the data validation and to facilitate resolution of inter-regional CLP laboratory performance problems.

10.0 THE TIER I VALIDATION COVER LETTER

10.1 Objective

The Tier I Validation Cover Letter documents that the data associated with a specific sample delivery group (SDG) were validated in accordance with the Region I Tier I Validation Guidance and justifies the use of a Tier I validation. The letter also documents the evaluation of PE sample results that were analyzed with the field samples, thereby providing a limited assessment of laboratory performance. Attachment M contains an example of a Tier I Validation Cover Letter.

10.2 Components of the Tier I Validation Cover Letter

10.2.1 Cover Letter

Tier I Validation Cover Letters that are generated for CLP Fund-lead and CLP PRP/Federal Facility oversight work, as well as EPA-generated non-CLP work, should be addressed and sent to the following:

- Christine Clark
  Regional Sample Control Center
  U.S. Environmental Protection Agency
  60 Westview Street
  Lexington, MA 02173
  cc: EPA Site Manager

- The subject heading of the Tier I Validation Cover Letter must include: the contractor work assignment number, the case number and SDG number (in that order), the laboratory name, the site name, the parameters evaluated, the total number of samples per sample matrix per parameter, (parenthetically identify the field duplicates), the sample matrix and field sample numbers analyzed for each parameter, the parameter, matrix and sample number for each type of blank, and the parameter, matrix, and sample number for each PE Sample. Note: Each sample number must be listed individually. (Refer to Attachment N for example of Data Validation Reports for exact Memorandum format to be used.)

- Only one SDG may be discussed in each Tier I Validation Cover Letter.

- Justification for Tier I validation. The validation Tier is based on project DQOs and is determined by the end users at the time of project scoping.

- Evaluation of PE sample results and potential impact on data

10.2.2 Attachments

10.2.2.1 Data Summary Tables - Unvalidated Data (CADRE-generated spreadsheets)

Data Summary Tables clearly marked "**NOT VALIDATED DATA**" should be included as an attachment for all Low/Medium CLP Organic and Inorganic data that have undergone CADRE review.

NOTE: Data Summary Tables are not required for data that have not undergone CADRE review.

10.2.2.2 Accuracy Check Worksheet- Data Validation Worksheet XI and PES Score Report/Vendor PES QC Acceptance Limits

All SDGs are required to have a parameter/matrix/concentration level associated PE sample, if one is available. The PE sample results should be evaluated based on Section XI in the appropriate VOA/SV, PEST/PCB or Inorganic Functional Guidelines (Parts II, III and IV of this document).

10.2.2.3 Support Documentation

10.2.2.3.1 Analytical Method for Non-CLP Methods

Copies of non-CLP methods and modifications to standard methods should be included in the Tier I Validation Cover Letter as support documentation and identified as such.

10.2.2.3.2 Copies of Telephone Logs/Communication Forms for the following must be included in the Tier I Validation Cover Letter:

- All communications with RSCC to track data packages and to resolve sample scheduling, tracking, and shipment questions
- All Regional/Laboratory communications to laboratories requesting resubmittal and/or clarification of data
- All communications with samplers to clarify sample numbers, locations, descriptions or preservation techniques and/or to alert them to significant field contamination
- All communications with the CLP-TPO/EPA DV Chemist to report contractually-deficient CLP data that will be recommended for data rejection or reduced payment
- All communications with the EPA Site Manager concerning possible data rejection
- All communications with the EPA Site Manager to authorize change in required data validation tier.

10.2.2.3.3 Copies of Data Supporting Recommendations for Reduced Payment

All non-compliant data that are of limited use to the end user are deemed to be of reduced worth by the region and should be recommended for reduced payment.

All non-compliances identified during a Tier I Validation that adversely affect data usability should be documented by attaching tabulated laboratory forms, raw data, or validator-prepared tabulations to substantiate the findings and conclusions presented in the Tier I Validation Cover Letter. For CLP data, support documentation attachments should be numbered and/or labelled and referenced accordingly in the text of the Tier I Validation Cover Letter. Similarly, support documentation for unusable non-CLP data should be attached to the Tier I Validation Cover Letter and recommendation for reduced payment noted. In addition, the validator should circle the specific items of concern located on these attachments.

10.2.2.3.4 Original Data Supporting Recommendations for Data Rejection/Zero Payment

All non-compliant original data that are unusable by the end user are deemed contractually unacceptable to the region, and, therefore, the laboratory should not be paid. Original CLP data should be attached to the Tier I Validation Cover Letter and sent to the CLP-TPO/EPA DV Chemist with a cover letter recommending data rejection. Similarly, unusable non-CLP data should be attached to the Tier I Validation Cover Letter and returned to the laboratory for non-payment.

10.2.2.3.5 Copies of Field Sampling Notes and/or Field Report

The field sampling notes and/or field report should be provided by the field sampler to the Lead Chemist or data validator to be included in the Tier I Validation Cover Letter as an attachment. In situations where sampling events extend over a period of weeks producing two or more SDGs and generate numerous pages of field log book notes, the field notes should be copied only once, included in one Data Validation Report and that Data Validation Report should be referenced by Case, SDG, and date of memorandum. The field sampling notes are included to provide complete documentation of the sampling event to substantiate site decisions made using the data and to support potential future litigation.

10.2.2.3.6 Copies of EPA-approved Amendments to QAPjP and/or SAP

Any EPA-approved amendments to the QAPjP and/or SAP that describe modified criteria used to validate site data should be included in the Tier I Validation Cover Letter as an attachment.

10.2.2.4 CSF Audit

Refer to Attachment C, Region I CSF Completeness Evidence Audit Program, July 3, 1991 or most recent revision.

10.2.2.5 DQO Summary Form

Copies of DQO Summary Forms previously submitted by the EPA Field Sampling Contractors, States and other Federal Agencies to the RSCC along with the quarterly CLP sample slot projections must be included with the Tier I Validation Cover Letter.

Copies of DQO Summary Forms for non-CLP sampling events previously submitted by the EPA Field Sampling Contractors to the RSCC prior to the sampling event, must be included with the Tier I Validation Cover Letter. Copies of DQO Summary Forms for non-CLP sampling events previously submitted by States and other Federal Agencies to the "Authorizing Organization" prior to the sampling event, should be included with site documents.

For proper distribution of the DQO Summary Forms refer to the DQO Summary Form Instructions (Attachment J, i).

The Draft DQO Summary Form (refer to Attachment J, i) should be used until such time as a Final version has been issued.

10.3 Initiating the Tier I Validation Procedure

a. Upon receipt of a data package, the data validator should ascertain the required data validation tier from the DQO Summary Form and/or EPA-approved QAPjP and/or SAP. If a Tier I validation is required, then the validator should determine if EPA or commercial PE samples were analyzed with the SDG. If an EPA PE sample was analyzed, then the PE Form I results should be faxed to the EPA PE Chemist for scoring. If PE samples were obtained from a commercial vendor, then the vendor' s PES QC acceptance limits should be utilized to evaluate PES results. If EPA or commercial PE samples were not included in the SDG, then the validator should note this and the reason why in the Tier I Validation Cover Letter.

b. The data validator should begin the Completeness Evidence Audit in accordance with the Region I CSF Completeness Evidence Audit Program, July 3, 1991 or most recent revision.

c. Once the PES Score Report is received, the data validator should evaluate the PE sample results in accordance with Section XI of Part II, III or IV of this document and complete the Section XI-Accuracy Check Worksheet.

d. The data validator should finalize the Completeness Evidence Audit.

e. If PE sample results indicate acceptable laboratory performance, then the validator should note this in the Tier I Validation Cover Letter.

f. If PE results indicate poor laboratory performance, then the data validator should note the specific laboratory performance problems and their impact on data quality. For example, "TCL MISSES" would indicate the possibility of false negatives, "TCL CONTAMINANTS" would indicate the possibility of false positives, and "ACTION LOW" and "ACTION HIGH" scores would indicate the possibility of negative and positive biases, respectively.

g. If PE results indicate poor laboratory performance, then the data validator should contact the EPA Site Manager to ascertain if a Tier II or Tier III validation is warranted. This call should be documented in a Telephone Log. **Only the EPA Site Manager can approve an upgrade in validation tier.**

h. The data validator should assemble the Tier I Validation Cover Letter with the all the required attachments as noted in Section 10.2.

11.0 THE DATA VALIDATION REPORT (Tiers II and III)

11.1 Objective

Data Validation Reports, generated for Tier II and Tier III validations, document that the data associated with a specific sample delivery group (SDG) were validated in accordance with the Region I Tier II and Tier III Validation Guidance, respectively. The Data Validation Report documents and discusses the rationale for any modifications to or deviations from the Region I Data Validation Guidance specified in this guidance document.

The findings of a Tier II and III validation are distributed to users for three distinct applications: (1) to make site decisions, (2) to provide oversight of CLP and non-CLP laboratory and method performance for contract management and payment recommendations, and (3) to provide EPA data validation oversight of the EPA Field Sampling Contractors.

For individuals involved in site-related decisions, it is imperative that the Data Validation Report present a clear explanation of those issues affecting the use of those data. The Report must provide the end users with an overview of analytical data quality and should also explain the qualitative confidence and quantitative "measurement error" associated with all sample results. In addition, the end users need Data Summary Tables that present all positive sample results, detection/quantitation limits, and associated qualifier codes.

On the other hand, the EPA individuals responsible for management and oversight of CLP and non-CLP laboratory performance and method performance require a presentation of issues related to laboratory non-compliance, poor laboratory practices that are not regulated in the contract, and any unusual method or analytical problems. For both contractual issues and problems affecting the usability of the data in making site decisions, support documentation must be sufficient to allow EPA to perform a full-scale review of the data validation in order to substantiate the Report's conclusions.

Data Validation Reports written by EPA Field Sampling Contractors are reviewed by the EPA-NE Quality Assurance Unit in accordance with the EPA-NE Data Validation Oversight/Methods Review Program. Data Validation Oversight Reports are provided to the EPA Site Managers and contract Project Officers. The contract Project Officer forwards the Data Validation Oversight Report to the EPA Field Sampling Contractor and requests corrective action. The continued effectiveness of the required corrective actions are monitored in subsequent validation oversights. Overall contractor data validation performance is monitored for each contract performance period.

11.2 Components of the Data Validation Report

In order to meet the varied needs of many end users, a six part **DATA VALIDATION REPORT** is generated. The report contains the following components in the order presented in this section. Each component should be completed in accordance with the following guidance. Attachment N includes two examples of Tier III Organic Data Validation Reports; a CLP Low/Medium organic soils SDG and a DAS low concentration surface waters SDG. Attachment J includes a copy of the following blank forms: DQO Summary Form, ORDA/IRDA Form, Telephone Log and Regional/Laboratory Communication Form, Data Validation Worksheets, Chain-of-Custody Form, and Traffic Report.

11.2.1 Organic/Inorganic Regional Data Assessment (ORDA/IRDA) Form

The ORDA/IRDA Form delineates issues relating to a laboratory's contractual non-compliance. The Form contains a checklist of items verified during validation. An ORDA/IRDA Form should be completed for all Tier II and III validations for CLP data validated by EPA Field Sampling Contractors, States, and other Federal Agencies for Fund-lead and PRP/Federal Facility oversight work. An ORDA/IRDA Form should also be completed for Tier II and III validations for non-CLP data performed by EPA Field Sampling Contractors for Fund-lead and PRP/Federal Facility oversight work.

For CLP data, "TPO/PO Action" should only be checked when contractual defects have resulted in reduced payment/data rejection recommendation letters to the TPO. All "TPO/PO Action" items should be detailed and documented in the "Action Items" line. Documentation supporting the "TPO/PO Action" items should be included in the Data Validation Report.

For EPA-generated non-CLP data, "TPO/PO Action" should only be checked when contractual defects have resulted in reduced payment/data rejection actions taken by the EPA Field Sampling Contractor. All "TPO/PO Action" items should be detailed and documented in the "Action Items" line. Supporting documentation should be included in the Data Validation Report. States, PRPs and other Federal Agencies are not required to submit ORDA/IRDA Forms for non-CLP data, but are encouraged to monitor the contractual performance of their contractor laboratories.

For both CLP and EPA-generated non-CLP data, refer to the back of the ORDA/IRDA Form for instructions on completing the form.

11.2.2 Data Validation Memorandum (DVM)

11.2.2.1 Narrative

This should briefly identify the scope of the analytical effort, provide a general overview of analytical quality, describe in detail and interpret all <u>specific problem areas that were identified in the worksheets</u>. Specific problems that impact the potential usability of the data should be emphasized. Data Validation Memoranda should be addressed and sent to the following:

- Christine Clark
  Regional Sample Control Center
  U.S. Environmental Protection Agency
  60 Westview Street
  Lexington, MA 02173
  cc: EPA Site Manager

  (Refer to Section 13.0 for proper distribution of Fund-lead, PRP/Federal

Facility Oversight and EPA-generated non-CLP Data Validation Report copies. Data Validation Reports generated by PRPs, States or other Federal Agencies for non Fund-lead sites should be distributed in accordance with those organizations' requirements.)

- The subject heading of the DVM must include: the contractor work assignment number, the case number and SDG number (in that order), the laboratory name, the site name, the parameters evaluated, the total number of samples per sample matrix per parameter, (parenthetically identify the field duplicates), the sample matrix and field sample numbers analyzed for each parameter, the parameter, matrix and sample number for each type of blank, and the parameter, matrix, and sample number for each PE Sample. Note: Each sample number must be listed individually. (Refer to Attachment N for example of Data Validation Reports for exact Memorandum format to be used.)

- Only one SDG may be discussed in each Data Validation Report.

- The first sentence of the first paragraph should state the validation tier used to validate the sample data. If different tiers were used to validate different subsets of the SDG, then this should be noted and the associated subsets and tiers identified.

- The first paragraph must also state that the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, July 1996 or most recent revision, was used to validate the data in accordance with the EPA-approved SAP and/or QAPjP. If validation criteria were modified to accommodate different QC criteria for non-CLP methods, then the modified criteria should be described in the first paragraph. If the EPA-approved SAP and/or QAPjP does not specify modified data validation criteria and the validator determines that modified criteria are necessary to properly evaluate the site data, then an amendment to the QAPjP and/or SAP describing the modified criteria must be submitted to EPA for approval prior to data validation. A copy of the amendment must be included in the support documentation for the Data Validation Report.

- The first paragraph must also identify the analytical methods used to analyze site samples.

- The second paragraph must list the QC parameters (checks) that were evaluated during validation. QC parameters that met criteria should be asterisked (*) in the left hand margin of the parameter name. Similarly, QC parameters that were not applicable to the analytical methods should be indicated by an "N/A" in the left hand margin of the parameter name. Note that worksheets should not be included for QC parameters that met criteria (except for Worksheet XII/XIII, Sample Quantitation) or were not applicable to the analytical method. (Refer to Attachment N for examples of a Tier II/III Data Validation Reports for exact memorandum format to be used.)

- "Potential Usability Issues" is the first parameter discussed in the DVM. The validator should discuss the potential impact of "measurement error" on data usability in terms of the project's Data Quality Objectives. The validator

should cite the usable aspects of the data and should identify problems as having either a major or minor impact on data usability.

- The DVM should identify for each QC parameter that did not meet criteria the affected samples, the analytical problem, and the recommended actions.

- Information should be presented in tabular format whenever possible, (see example DVMs in Attachment N). Narratives should be limited to discussions of complex analytical problems and justifications of actions taken based on professional judgment. The information should be conveyed in simple, concise language that an individual without an extensive background in analytical chemistry can understand.

- The DVM must clearly differentiate problems affecting the confidence concerning the presence/absence of a compound versus those involving quantitative error.

- The DVM should also differentiate between sampling issues (sampling error) and analytical issues (analytical error).

- The narrative should list or reference all changes that the validator has made to the laboratory's reported data, whether due to misidentification, errors in transcription or calculation.

- The last QC parameter discussed in the DVM is System Performance. This should include an overview of interrelated and/or multiplicative analytical problems that impact usability of the data.

- The narrative should list support documentation attachments and should include the validator's name and signature.

11.2.2.2 Data Summary

11.2.2.2.1 Qualifier Recommendation Summary Table-Table I

The purpose of Table I is to identify all qualifier codes applied to each sample per parameter, taking into account the multiplicative effects of various qualifiers. The validator should assess tendencies in bias.

11.2.2.2.2 Overall Evaluation of Data (Data Validation Worksheet)-Table II

The purpose of Table II is to identify and summarize the "analytical error" associated with the data as well the "sampling error" that was identified through validation. It also identifies potential usability issues associated with the data for the end user.

Since sampling variability must be assessed by the end user, that column remains blank on Table II throughout data validation.

11.2.2.2.3 Tentatively Identified Compound (TIC) Summary-Table III

Table III includes a list of TICs. TICs reported by the laboratory as "UNKNOWNS" without a compound class should not be included in the table.

11.2.2.2.4 Data Summary Tables-Spreadsheet

The purpose of the Data Summary Table is to provide a simple, condensed form of the analytical results (excluding PE sample results) for the end user, which enables a quick evaluation and comparison of the constituents identified at the various sampling locations.

Separate tables in "Lotus 1 2 3" are required for soil and water analyses and for organics and inorganics analyses. Additionally, separate tables are also required for volatile, semivolatile, and pesticide/PCB analytes for the organic analyses. Other database software may be used to generated Data Summary Tables as long as there is no deviation from the format and content requirements exhibited in Attachment N.

The Data Summary Tables must include: case number, CLP SDG number, site name, site location, matrix, parameter, concentration units, method-required detection/quantitation limits (CRDLs/CRQLs), EPA Sample (Traffic Report) numbers, sample locations/descriptions, laboratory sample numbers, all positive sample results, sample-specific and associated qualifier codes, dilution factors, % solids for soils, dates sampled, dates extracted, and dates analyzed. Examples of the Data Summary Tables are provided in Attachment N.

Only codes defined by this document are permitted to qualify data. Should it be necessary to include other codes, prior approval must be obtained from the EPA-NE CLP-TPO. If approval is given, complete definitions must be supplied in the key for the Data Summary Table. The standard data validation codes used in qualifying data in accordance with this guidance are:

U - The analyte was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit. The sample quantitation limit accounts for sample specific dilution factors and percent solids corrections or sample sizes that deviate from those required by the method.

J - The associated numerical value is an estimated quantity.

R - The data are unusable (analyte may or may not be present). Resampling and reanalysis is necessary for verification. The R replaces the numerical value or sample quantitation limit.

UJ - The analyte was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

EB, TB, BB - An analyte that was identified in an aqueous equipment blank, trip blank, or bottle blank that was used to assess field contamination associated with soil/sediment samples. **These qualifiers are to be applied to soil/sediment sample results only.** (For additional guidance refer to Blank Section V of Parts II, III or IV)

11.2.3 Standard Data Validation Worksheets

The data validation worksheets included in this document must be utilized to perform the data validation. Any modification to the worksheets must be documented in the QAPjP and/or SAP and be approved by EPA prior to sampling.

Worksheets should not be included for QC parameters that meet criteria or criteria that are not applicable to the analytical method, except for Worksheet XII/XIII-Sample Quantitation.

However, the data validator must complete page two of the Data Validation Worksheet Cover Page, and then sign and date the worksheet.

Copies of automated data review reports, i.e., CADRE, should be included in this section. Any automated data review reports, such as CADRE should be incorporated into the Data Validation Report according to the Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, February 1995 or most recent revision.

A completed Data Validation Worksheet Cover Page must precede the other worksheets.

11.2.4 Support Documentation

11.2.4.1 Analytical Method for Non-CLP Methods

Copies of non-CLP methods and modifications to standard methods should be included in the Data Validation Report as support documentation and identified as such.

11.2.4.2 Copies of Telephone Logs/Communication Forms for the following must be included in the Data Validation Report:

- All CLP "Records of Communications" with the RSCC to track data packages and to resolve sample scheduling, tracking, and shipment questions
- All Regional/Laboratory communications with laboratories requesting resubmittal and/or clarification of data
- All communications with samplers to clarify sample numbers, locations, descriptions or preservation techniques and/or to alert them to significant field contamination
- All communications with the CLP-TPO/EPA DV Chemist to report contractually-deficient CLP data that will be recommended for data rejection or reduced payment
- All communications with the EPA Site Manager concerning possible data rejection
- All communications with the EPA Site Manager to authorize change in required data validation tier.

11.2.4.3 Copies of Data Supporting Recommendations for Reduced Payment

All non-compliant data that are of limited use to the end user are deemed to be of reduced worth by the region and should be recommended for reduced payment.

All non-compliances, identified in the Data Validation Memorandum and/or on the ORDA/IRDA Form, that adversely affect data usability should be documented by attaching tabulated laboratory forms, raw data, or validator-prepared tabulations to substantiate the findings and conclusions presented in the text. Support documentation attachments should be numbered and/or labelled and referenced accordingly in the text of the DVM Narrative and on the ORDA/IRDA Form. In addition, the validator should circle the specific items of concern located on these attachments.

11.2.4.4 Original Data Supporting Recommendations for Data Rejection/Zero Payment

All non-compliant original data that are unusable by the end user are deemed contractually unacceptable and of no value to the region, and, therefore, the laboratory should not be paid. Original CLP data should be attached to the Data Validation Report and sent to the CLP-TPO/EPA DV Chemist with a cover letter recommending data rejection. Similarly, unusable,

non-compliant non-CLP data should be attached to the Data Validation Report and returned to the laboratory for non-payment.

11.2.4.5 Copies of Field Sampling Notes and/or Field Report

The field sampling notes and/or field report should be provided by the field sampler to the Lead Chemist or data validator to be incorporated in the Data Validation Report as an attachment. In situations where sampling events extend over a period of weeks producing two or more SDGs and generate numerous pages of field log book notes, the field notes should be copied only once, included in one Data Validation Report and that Data Validation Report should be referenced by Case, SDG, and date of Data Validation Report. The field sampling notes are included to provide complete documentation of the sampling event to substantiate site decisions made using the data and to support potential future litigation.

11.2.4.6 Copies of EPA-approved Amendments to QAPjP and/or SAP

Any EPA-approved amendments to the QAPjP and/or SAP that describe modified criteria used to validate site data should be included in the Data Validation Report as support documentation.

11.2.5 CSF Completeness Evidence Audit

Refer to Section 10.2.2.4

11.2.6 DQO Summary Form

Refer to Section 10.2.2.5

11.3 Initiating the Tier II and Tier III Data Validation Process

Once the various sources of information, as discussed in Section 8, are assembled, the data validator should begin the Tier II or Tier III validation in accordance with steps a., b., and c. outlined in Section 10.3. Next, the validator should review the Data Package Narrative and generate Data Summary Tables in spreadsheet format (i.e., Lotus or other database software) according to the following guidance.

11.3.1 Reviewing the CLP Data Package Narrative/Cover Page

Review of the Data Package Narrative in conjunction with the chain-of-custody forms, Traffic Reports and Log In sheets (CLP Organic SDG Narrative or CLP Inorganic Cover Page) should quickly familiarize the data validator with all QC, sample, shipment and/or analytical problems.

The CLP Data Package (SDG) Narrative/Cover Page must:

- Justify the use of flagged edits on organic CLP quantitation lists.
- Document all instances of manual integration in organic CLP cases.
- Differentiate between initial analyses and reanalyses for CLP and state if reanalysis is billable and why.

- List all pH determinations for VOAs.
- Document SOW number or method name and version date.
- Be signed by the Laboratory Manager authorizing the release of the data, and verifying the contents of the data and deliverables.

Note: Non-CLP laboratory data packages should provide similar sample analysis information in a narrative or cover page format..

Review the Data Package Narrative/Cover Page to determine if gross analytical and/or shipment problems occurred.

If holding times were exceeded and resulted in qualified data, the data validator should assess the reduced worth of the data. For CLP data packages, the validator should submit a reduced payment recommendation to the TPO in accordance with Attachment O, March 7, 1995 Memorandum to Heidi Horahan, ARCS DPO re: CLP-SOW OLM03.1-New Contract Requirements. If holding times were grossly violated, then data rejection may be warranted. The data rejection procedures specified in Attachment I should be followed. If VOA sample pH measurements indicate that samples were not acid preserved in the field, then the validator should contact the sampler to confirm that incorrect preservation techniques were used and document the finding as "sampling error" in the Data Validation Memorandum.

If other analytical and/or sampling related problems, i.e., shipment, were noted in the Data Package Narrative, then the validator should describe in the DVM those problems that impact the potential usability of the data.

11.3.2 Generating Data Summary Tables

Transcribe the results from the Form Is onto the Data Summary Tables. For organic analyses, do not transcribe the qualification codes used by the laboratory except for all "U"s for non-detects as well as "J"s for positive detects reported below the sample-specific CRQL. For inorganic analyses, do not transcribe the qualification codes used by the laboratory except for all "U"s for non-detects. For all inorganic positive detects that are less than or equal to 2x analyte IDLs, qualify sample results with a "J" code.

As appropriate, information will be added to or deleted from the Data Summary Tables during the course of data validation. PES and method blank results should not be reported on the Data Summary Tables.

Note that for CADRE validations, the Data Summary Tables are automatically generated. CADRE Data Summary Tables are provided to the EPA data validator for both validated and unvalidated data. For Tier I validations, Data Summary Tables with "**NOT VALIDATED DATA**" are included as an attachment to the Tier I Validation Cover Letter. For Tiers II and III, the validator must complete the validation in accordance with the Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, February 1995 or latest revision.

11.3.3 Usage of Qualifier Codes on the Data Summary Tables

The data qualifier codes, presented in Section 11.2.2.2.4, identify the degree of confidence concerning the presence or absence of reported compounds and identify results that are considered to be quantitatively inaccurate. These codes have been regionally standardized to

ensure that data validators throughout the region employ the same set of simple, concise definitions that are understandable to personnel within the various EPA offices. Therefore,

a. Only codes defined in Section 11.2.2.2.4 may be used to qualify or reject data. Should it be necessary to include other codes, prior approval must be obtained from the EPA-NE CLP-TPO.

b. In general, only one qualifier code is used with each reported result. The following hierarchy has been developed to ensure that only the most important code is used in situations where more than one quality control problem is associated with an analytical result:

- Codes relating to identification take precedence over codes related to quantitation. **If results are rejected, replace the numerical sample result or sample quantitation limit with an "R".** Thus, whenever a positive result is rejected "R", the "J" code will not be used. Also, whenever a non-detected result is rejected "R", the "U" or "UJ" code will not be used.

- Within each of the two categories of codes, the code that indicates a more serious problem with the data takes precedence. In all cases, the R code supersedes the J or EB, TB, BB codes.

- The J and the EB, TB, BB codes may be used together for soil/sediment samples.

c. The above restriction on the general use of multiple qualifiers for a single result is applicable only to the Data Summary Table and not to the narrative portion of the Data Validation Memorandum. The narrative should mention all problems, major and minor, associated with the individual sample results.

d. Parts II, III and IV of this document address the individual situations requiring the use of particular qualifier codes. Upon completion of the data validation, the validator should double check the Data Summary Tables for accuracy and completeness to ensure that the appropriate qualifier codes were added according to the requirements listed herein. The validator should also check that there are no discrepancies between the worksheets, Data Validation Memorandum narrative, the Qualifier Recommendation Summary Table, and the Data Summary Tables.

Once the data validation has been completed, the validator compiles the Data Validation Report and submits it for internal review within their organization.

12.0 INTERNAL REVIEW OF VALIDATION DOCUMENTS

12.1 Senior Validator Review

A Senior validator should review all Tier I Validation Cover Letters and Data Validation Reports to ensure the following:

a. All components of a Tier I Validation Cover Letter or Tier II or Tier III Data Validation Report are included.

b. Data validation has been performed in accordance with the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses and/or EPA-

approved modified or alternate validation criteria.

c. The data package has been evaluated for analytical quality and contractual compliance, and correct actions have been taken in the Data Validation Memorandum to address specific analytical deficiencies.

d. Compound names and concentrations reported on Data Summary Tables are consistent with Form I's or other laboratory tabulated report forms. All discrepancies should be justified in the Data Validation Memorandum.

e. Data qualifications identified in the worksheets are consistent with those in the Data Validation Memorandum narrative, the Qualifier Recommendation Summary Table and Data Summary Tables.

f. Non-compliant data that are unusable have been recommended for reduced payment/data rejection when applicable.

g. The project DQOs were used to determine if the degree of "measurement error" associated with the data potentially compromises the data usability.

12.2 Lead Chemist Review

As a final step in this process, it is important that the Lead Chemist check all outgoing reports for accuracy and completeness, due to the complexity of data validation and the importance of performing an accurate final assessment of data quality. The Lead Chemist must also review and concur with the final assessment of data quality and potential usability issues raised by the junior and senior validators.

The Lead Chemist should ensure that all accepted data are contractually compliant and usable.

The Lead Chemist must submit data rejection and reduced payment recommendation letters whenever appropriate.

The Lead Chemist must ensure that the final Data Validation Report is correctly distributed.

13.0 DISTRIBUTION OF DATA VALIDATION REPORTS AND TIER I VALIDATION COVER LETTERS

The following distribution table is applicable to Data Validation Reports and Tier I Validation Cover Letters generated by EPA Field Sampling Contractors for CLP and non-CLP Fund-lead and CLP PRP/Federal Facility oversight work.

The CLP Data Validation Reports generated by States or other Federal Agencies performing Fund-lead work under Cooperative and Interagency Agreements, respectively, should be sent to the EPA-NE RSCC for purposes of contract administration. A copy of the CLP Data Validation Reports and/or Final Project Reports should also be sent to the EPA Site Manager.

Copies of non-CLP Data Validation Reports generated by States, other Federal Agencies, PRPs, or Federal Facilities are not required to be forwarded to the EPA-NE RSCC. However, States, other Federal Agencies, PRPs, or Federal Facilities should forward a copy of the non-CLP Data Validation Report and/or the Final Project Report to the EPA Site Manager.

Table of Deliverables

Document	REGIONAL RECIPIENTS		NATIONAL RECIPIENTS
	EPA-NE		Regions II-X
	CLP-TPO/RSCC (For Central Files)	EPA SITE MANAGER	CLP-TPO
TIER I VALIDATION COVER LETTER with attachments	X	X	
DATA VALIDATION REPORT			
ORDA/IRDA Form	X		X*
DV Memo (including narrative, Tables I, II, III, and Data Summary Tables)	X	X	X*
Worksheets	X	X	
Support Documentation	X	X	X*
CSF Completeness Audit	X	X	
DQO Summary Form	X	X	
CSF - DATA PACKAGE		X**	

* CLP Data Validation Memoranda only (EPA-generated non-CLP Data Validation Memoranda are not distributed nationally)

Note: Telephone Logs/Communication Forms for the CLP Regional/Laboratory communication program should be forwarded to the NPO Sample Scheduling and Coordination Contractor, RSCC, CLP-TPO (their copy to be included in the Data Validation Report or the Tier I Cover Letter), and the CLP laboratory.

**All data packages/CSFs are ultimately archived in the EPA-NE Administrative Records Center.

14.0 EPA DATA VALIDATION OVERSIGHT/METHODS REVIEW PROGRAM

The regional QA Unit of OEME reviews and comments upon contractor-prepared Data Validation Reports and Tier I Validation Cover Letters. This oversight program serves a dual purpose. First, the QA Unit evaluates the contractor's ability to accurately perform data validation in accordance with this regional policy. Secondly, the QA Unit assesses the use of current, new and/or modified analytical methods in order to make needed method revisions based on scientific data. Resubmission of Data Validation Reports may be required in cases where the required format and procedures were not followed, or when clarifications or corrections are needed. The EPA Field Sampling Contractor is responsible for implementing and monitoring the effectiveness of all corrective actions recommended by EPA during oversight for validations performed by the prime contractor and any subcontractors. When critical deficiencies and/or problems have been identified during EPA Oversight, the EPA Field Sampling Contractor may be required to prepare a separate Corrective Action response letter to resolve those deficiencies and/or problems.

References

1. User's Guide to the Contract Laboratory Program, EPA/540/P-91/002, January 1991.

2. EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, August 1994, DRAFT INTERIM FINAL.

3. NEIC Policies and Procedures, EPA-330/9-78-001-R, May 1978, revised May 1986.

4. Data Quality Objectives Process for Superfund, EPA/540/R-93/071, September 1993, INTERIM FINAL.

5. Test Methods for Evaluating Solid Waste, Physical/Chemical Method (EPA Pub. SW-846, Third Edition) and updates.

6. USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, ILMO4.0, EPA/540/R-95/121.

7. USEPA Contract Laboratory Program Statement of Work for Organic Analysis, OLMO3.1, EPA/540/R-94/073.

8. Region I Tiered Organic and Inorganic Data Validation Guidelines, July 1, 1993, DRAFT.

9. Region I CSF Completeness Evidence Audit Program, July 3, 1991.

10. Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, 2/01/88, modified 11/01/88.

11. Region I Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, 6/13/88, modified 2/89.

12. Specifications and Guidance for Contaminant-Free Sample Containers - Publication 9240.0-05A, EPA/540/R-93/051, December 1992.

13. Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies, EPA/600/R-92/128, July 1992.

14. Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, February 1995.

15. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, EPA/540/R-94/012, February 1994.

16. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA/540/R-94/013, February 1994.

17. EPA Region I Performance Evaluation Program Guidance, July 1996 Revision.

18. Standard Operation Procedures for Submitting Data for Reduced Payment/Data Rejection, September 9, 1991.

19. Training Manual for Reviewing Laboratory Data Package Completeness, June 1994.

Figure 1: CLP Data Validation - Roles & Responsibilities

Figure 2: Overview of the Data Validation Process for CLP Data

Figure 3: Overview of the Data Validation Process for Non-CLP Data Generated for EPA

Figure 4: Data Validation Criteria Flow Chart for EPA Superfund Data

For hardcopy of Figures 1, 2, 3, and 4 contact:

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Part I - Data Validation Manual: The Data Quality System

ATTACHMENTS

The following attachments are referenced in Part I of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses. Guidance in some of the documents is superseded by the more recent guidance provided in Part I.

Attachment A	"Quality Assurance for Superfund Environmental Data Collection Activities" - Publication 9200.2-16FS, February 1993, and "EPA Order 5360.1, Draft 1995 Quality Assurance Order".
Attachment B	"Region I Tiered Organic and Inorganic Data Validation Guidelines", July 1, 1993, DRAFT.
Attachment C	"Region I CSF Completeness Evidence Audit Program", July 1991.
Attachment D	"Specifications and Guidance for Contaminant-Free Sample Containers" - Publication 9240.0-05A, EPA/540/R-93/051, December 1992.
Attachment E	"User's Guide to the Contract Laboratory Program", EPA/540/P-91/002, January 1991.
Attachment F	Region I Short Sheets and EPA CLP Information Sheets.
Attachment G	"Training Manual for Reviewing Laboratory Data Package Completeness", June 1994.
Attachment H	"EPA Region I Performance Evaluation Program Guidance", July 1996, Revision.
Attachment I	"Standard Operating Procedures for Submitting Data for Reduced Payment/Data Rejection", September 9, 1991.
Attachment J	Data Validation Report - Blank Forms i. DQO Summary Form ii. ORDA/IRDA Form iii. Telephone Log or Regional/Laboratory Communication Form iv. Data Validation Worksheets v. Chain-of-Custody Form vi. Traffic Report
Attachment K	Example of Contract Compliance Screening (CCS) Report
Attachment L	"Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review", February 1995.
Attachment M	Example Tier I Validation Cover Letter
Attachment N	Example Tier III Data Validation Reports
Attachment O	"March 7, 1995 Memorandum to Heidi Horahan, ARCS DPO re: CLP-SOW OLM03.1-New Contract Requirements."
Attachment P	"The Regional Sample Control Center Guidance for the Contract Laboratory Program (CLP) and Delivery of Analytical Services (DAS) Program for EPA-New England", November 1996.
Attachment Q	"Region I ARCS Delivery of Analytical Services Pilot Program, Final Report Volume II. Appendices", 15 March 1994.

Attachment A

"Quality Assurance for Superfund Environmental Data Collection Activities" - Publication 9200.2-16FS, February 1993, and "EPA Order 5360.1, Draft 1995 Quality Assurance Order"

For hardcopy of Attachment A contact:

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For electronic copies of Order 5360.1 see:

http://www.epa.gov/region07/newsinfo/qaorder.html

Attachment B

"Region I Tiered Organic and Inorganic Data Validation Guidelines",
July 1, 1993, DRAFT

REGION I

TIERED ORGANIC AND INORGANIC DATA VALIDATION GUIDELINES

JULY 1, 1993

INTRODUCTION

Historically, Region I has required that analytical data for Superfund sites undergo full validation according to the Region I Laboratory Data Validation Functional Guidelines documents.

Full validation, however, does not always meet the Data Quality Objectives (DQOs) for each site activity, and it can contribute to high costs and missed deadlines. To address this problem, Region I's Environmental Services Division (ESD) has created a tiered approach to data validation which accomplishes the following:

- enables data users to select the level of validation necessary to meet their DQOs
- saves time and money
- promotes consistent evaluation of data quality between Superfund sites

Three tiers have been established and are described in the next section. Tier III is equivalent to the full validation currently performed in Region I, and includes the procedures performed under Tiers I and II.

TIERED APPROACH TO DATA VALIDATION

The inorganic and organic data validation process can be broken down into three distinct levels: Tier I, Tier II, and Tier III.

Tier I: A completeness evidence audit is performed to ensure that all laboratory data and documentation are present. Completeness evidence audits are performed in accordance with procedures contained in the Region I CSF Completeness Evidence

Audit Program, dated 7/3/91. (This document is the currently used procedure as referenced in the memorandum titled "Region I CSF Completeness Evidence Audit Program" from the Region I CLP-TPOs to Region I Contractors, dated 7/7/91.)

Tier II: A Tier I completeness evidence audit is performed, and, in addition, the results of all Quality Control (QC) checks and procedures are evaluated and used to assess and qualify sample results. Tier II data validation is performed primarily from information contained on the tabulated data reporting forms. It has been estimated by ESD that Tier II validation takes 50% of the time required to perform a Tier III validation.

Tier III: A full data validation is performed. Tier III includes Tier I and Tier II procedures plus an in-depth examination of all raw data to check for technical, calculation, analyte identification/analyte quantitation, and transcription errors. Tier III data validation is performed in accordance with the Region I CSF Completeness Evidence Audit Program and the Region I Laboratory Data Validation Functional Guidelines.

At a minimum, all data should be carried through Tiers I or II. Tier I is mandatory, regardless of the immediate intended use of the data, to ensure that all laboratory documents have been obtained for future data validation, potential litigation, and/or to defend site decisions. Validation requirements must always be documented in an approved QAPP prior to sampling. Several examples of when a Tier I or Tier II validation may suffice to meet DQOs are as follows:

- Design run data which are collected during a treatability study. Data used to support the final design parameters, however, should undergo Tier III validation.

- Long-term monitoring data which have only "minimal changes" in constituent concentrations from the previous round. The magnitude of these allowable changes, as well as the procedures to be followed if QAPP requirements are not met, must be documented in an approved QAPP prior to sampling. (If QAPP requirements are not met, a Tier II or Tier III validation should be performed.)

- EPA oversight split data which "compare well" with PRP data. The comparison criteria, as well as procedures to be followed if QAPP requirements are not met, must be documented in an approved QAPP prior to sampling. (If QAPP requirements are not met, a Tier II or Tier III validation should be performed.)

Full validation (Tier III) can always be performed at a later date as long as Tiers I or II have been initially completed. The entire data package (Tier III) or just individual parameters, matrices, sample locations, and/or risk compounds (partial Tier III) could then be specified for full validation. If a subset of the entire data package was targeted for full validation, then a Tier II validation would be performed on the entire data package (if it hadn't already) and a partial Tier III validation would be performed for individual parameters, etc. (whatever was to comprise the subset validation). The first paragraph of the data validation memorandum must explicitly document the level of validation performed, i.e. Tier II plus partial Tier III validation for benzene, Tier II plus partial Tier III validation for sample location MW-100, Tier II plus partial Tier III validation for volatile organics, etc.

In certain circumstances, full validation (Tier III) may be deemed necessary from the start of a project. Several examples of when full validation is needed are as follows:

- Only one set of data for a particular sample location, type and/or parameter is available and a decision of whether to remediate will be based on this sample. An example of this is background data.

- The data will be used to define a critical site boundary.

- The data will be used to determine compliance with clean-up goals.

TIER II DATA VALIDATION PROCEDURE

To perform a Tier II data validation, a Tier I review is completed and the results of all QC checks and procedures are evaluated and used to assess and qualify sample results. During a Tier II review,

the raw data for field samples and QC checks are not evaluated (with a few exceptions, i.e. pH check for volatile organics, metals, and cyanide to verify proper sample preservation). The goal is to validate data using information contained mainly on the tabulated data reporting forms and chain-of-custody (COC) forms. Tier II assumes that all results are reported by the laboratory and that all reported results are correct.

Prior to performing a Tier II validation, conduct the Tier I completeness evidence audit according to the requirements contained in the Region I CSF Completeness Evidence Audit Program, dated 7/3/91, and request the missing deliverables from the laboratory. Begin the Tier II validation while waiting for any missing deliverables.

To perform a Tier II inorganic validation, the reviewer must have all data reporting forms for field sample and QC sample results (Forms I through XIV), as well as the COC forms in the data package. Validation is performed according to requirements contained in the attached table (Attachment I) and in conjunction with the Region I Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, dated 6/13/88 (modified 2/89). This guidance is also applicable to inorganic analyses performed in accordance with the ILM01.0, ILM02.0, and ILM03.0 versions of the U.S. EPA CLP Statement of Work (SOW). Tier II reporting and deliverable requirements are the same as those for full validation (Tier III); only the actual validation procedures contained in Section 3 of the Region I Functional Guidelines have been modified to minimize examination of the raw data and to eliminate the recalculation of results.

To perform a Tier II organic validation, the reviewer must have all data reporting forms for field sample and QC sample results (Forms I through X), as well as the COC forms in the data package. Validation is performed according to guidance contained in the attached table (Attachment II) and in conjunction with the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, dated 2/1/88 (modified 11/1/88). This guidance is also applicable to organics analyses performed in accordance with the OLM01.0 SOW, even though the 11/1/88 Region I Functional Guidelines document has not yet been modified to accomodate pesticide/PCB method changes contained in the OLM01.0 SOW. Tier II reporting and deliverable

requirements for data validation are the same as for full validation (Tier III); only the actual validation procedures contained in Sections 3 and 4 of the Region I Functional Guidelines have been modified to minimize examination of the raw data and to eliminate the recalculation of results.

The results for each QC parameter, specified in Attachments I and II, must be evaluated using the data reporting forms provided by the laboratory. The data provided on the forms are not verified with the raw data. Information contained on the forms should be used to verify that QC samples were analyzed with the correct analytes at the proper frequency and concentration, that the QC limits were met, and required corrective actions were taken. The QC parameters of System Performance and Compound Identification for the volatile and semivolatile fractions are not evaluated during the Tier II review as it would require that a substantial review of the raw data be performed.

As a result of the Tier II evaluation, the field sample results may be accepted, qualified as estimated, or rejected. <u>In circumstances where the entire data package or data for multiple samples must be rejected or will be significantly qualified based upon the Tier II results, the reviewer must first consider the impact of rejected results and/or discrepant information on the data needs of the specific project. If the data are critical to the project needs, then examination of the raw data is strongly recommended to prevent faulty site decisions based on technical, transcription, and/or calculation errors. The EPA Remedial Project Manager (RPM) or Site Assessment Manager (SAM) must be contacted to approve a partial or complete Tier III validation prior to its initiation</u>. If the RPM or SAM decides that no further validation is warranted based on the objectives of the sampling event and the nature of the data qualification, then the reviewer should document this decision in the <u>first paragraph</u> of the data validation (DV) memorandum. The nature of the data problem, the extent of data qualification, and the level of validation performed must also be documented in the DV memorandum. It is expected that raw data review might be required more frequently for pesticide/PCB data, since identification and quantitation of pesticides and PCBs is based solely on gas chromatography data with no mass spectral confirmation/quantitation.

The attached tables, Attachment I (Tier II Inorganic Data Validation)

and Attachment II (Tier II Organic Data Validation), consist of four columns which identify the specific QC criteria to be checked, the laboratory reporting form(s) to review, the specific sections of the Region I Functional Guidelines to follow, and the adjustments needed for the specific sections of the Region I Functional Guidelines to perform a Tier II validation.

ATTACHMENT I

TIER II INORGANIC DATA VALIDATION

TIER II INORGANIC DATA VALIDATION

QC CRITERIA	DATA REPORTING FORMS TO REVIEW	APPLICABLE SECTIONS IN FUNCTIONAL GUIDELINES[3]	COMMENTS
Data Completeness	! Complete SDG File (CSF) 1. Original Sample Data Package including Cover Page, Forms I through XIV, DC-1, DC-2, raw data 2. Original shipping and receiving documents 3. All original lab records of sample transfer, preparation and analysis, as well as telephone contact logs.	! I., p. 21	! Perform a Tier I completeness evidence audit according to procedures in the Region I CSF Completeness Evidence Audit Program, dated July 3, 1991, to ensure that all laboratory data and documentation are present. Request missing deliverables from the laboratory following appropriate procedures.
Holding Times	! Forms I, XIII, XIV ! Chain-of-Custody/Traffic Report ! Sample Digestion/Distillation Logs	! II. A through D, pp. 21-22	! Examine Chain-of-Custody/Traffic Report Forms to determine if samples were properly preserved in the field. ! To verify sample pH upon laboratory receipt, review sample digestion logs as this information is not included on the forms.
Calibration	! Forms IIA, IIB, XIV	! III. A through B, pp. 22-23 C.1-3, pp. 23-24 C.5 and 6, p. 24 C.8 and 9, p. 24 D.1-3, pp. 24-25 D.5-8, pp. 25-26	! Calibration correlation coefficients for AA, Hg, and CN are not reviewed since this information is not included on the forms.
Blanks	! Forms I, III, X, XIII, XIV ! Chain-of-Custody/Traffic Report	! IV. A through D, pp. 26-28	! Review data reporting forms only. Do not verify with raw data.
ICP Interference Check Sample	! Forms I, IV, X, XI, XIV	! V. A through B, p. 28 C.1 and 2, p. 28 C.4, p. 29 D, pp. 29-31	! Review data reporting forms only. Do not verify with raw data. ! Paragraph C.4: For evidence of results with an absolute value >2xIDL for those analytes which are not present in the ICS A solution, evaluate Form IV. Do not check the raw data.

SEE NOTE ON PAGE 3 OF 3.

[3]REGION I LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES, 6/13/88, MODIFIED 2/89

TIER II INORGANIC DATA VALIDATION

QC CRITERIA	DATA REPORTING FORMS TO REVIEW	APPLICABLE SECTIONS IN FUNCTIONAL GUIDELINES[3]	COMMENTS
Matrix Spike Sample Analysis	! Forms VA, VB, XIII ! Chain-of-Custody/Traffic Report	! VI. A through B, pp. 31-32 C.1, p. 32 C.3-5, p. 32 D, pp. 32-33	! Review data reporting forms only. Do not verify with raw data. ! Review Chain-of-Custody/Traffic Report Forms to verify that samples identified as field blanks are not used for spiked sample analysis.
Laboratory Duplicate Sample Analysis	! Forms VI, XIII ! Chain-of-Custody/Traffic Report	! VII. A through B, p. 33 C.1, p. 33 C.3 and 4, p. 34 D, p. 34	! Review data reporting forms only. Do not verify with raw data. ! Review Chain-of-Custody/Traffic Report Forms to verify that samples identified as field blanks are not used for duplicate sample analysis.
Field Duplicates	! Form Is ! Chain-of-Custody/Traffic Report	! VIII. A through D, pp. 34-35	! No change from current procedures.
Laboratory Control Sample Analysis (LCS)	! Forms VII, XIII	! IX. A through B, p. 35 C.1, p. 35 C.3, p. 36 D, p. 36	! Review data reporting forms only. Do not verify with raw data.
Furnace Atomic Absorption Analysis	! Forms I, VIII, XIII, XIV	! X. A through B, p. 37 C.1 and 2, p. 37 C.4, p. 37 D, pp. 37-38	! Review data reporting forms only. Do not verify with raw data. ! Review Form Is for the presence/absence of "M" flags indicating the failing/passing of the duplicate injection precision criteria for field samples. ! Do not verify post-digestion spike recoveries reported on Form XIV with the raw data. ! To verify that the Furnace Atomic Absorption Analysis Scheme was followed, evaluate Form XIV for spike recoveries not within 85-115%, initial and reanalyses, and dilution factors. In addition to Form XIV, evaluate Form I for sample concentrations to verify that an MSA analysis was not required for any result quantitated directly from the calibration curve and for which spike recoveries were not within 85-115%.
ICP Serial Dilution Analysis	! Forms IX, X, XIV	! XI. A through B, pp. 38-39 C.1, p. 39 C.3, p. 39 D, p. 39	! Review data reporting forms only. Do not verify with raw data. ! Paragraph C.3: For evidence of negative interference, evaluate Form IX. Do not check the raw data.

SEE NOTE ON PAGE 3 OF 3.

[3]REGION I LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES, 6/13/88, MODIFIED 2/89

TIER II INORGANIC DATA VALIDATION

QC CRITERIA	DATA REPORTING FORMS TO REVIEW	APPLICABLE SECTIONS IN FUNCTIONAL GUIDELINES[3]	COMMENTS
Detection Limits	! Forms I, X, XIII, XIV	! XII. A through D, pp. 39-40	! Paragraph C.3: To verify that sample weights, volumes, and dilutions are taken into account when reporting sample quantitation limits, evaluate Forms I, X, XIII, and XIV.
Sample Result Verification	! Forms I, XII, XIII, XIV	! XIII. A through B, pp. 40-41 C.3, p. 41 D, p. 41	! Review data reporting forms only. Do not verify with raw data. ! For any result reported on Form I for which the sample result is greater than the linear range for ICP (Form XII) and greater than the calibrated range for non-ICP parameters (Form XIV), verify that the result was reported from a diluted sample analysis (Form XIV) and that the diluted sample result falls within the respective ranges. Dilution and preparation factors are found on Forms XIII and XIV. Do not check the raw data.
Overall Assessment of Data for a Case		! XIV., p. 42	! Limit to the sections evaluated during Tier II review.

NOTE: IN CIRCUMSTANCES WHERE THE ENTIRE DATA PACKAGE OR DATA FOR MULTIPLE SAMPLES MUST BE REJECTED OR WILL BE SIGNIFICANTLY QUALIFIED BASED UPON THE TIER II RESULTS, THE REVIEWER MUST FIRST CONSIDER THE IMPACT OF REJECTED RESULTS AND/OR DISCREPANT INFORMATION ON THE DATA NEEDS OF THE SPECIFIC PROJECT. IF THE DATA ARE CRITICAL TO THE PROJECT NEEDS, THEN EXAMINATION OF THE RAW DATA IS STRONGLY RECOMMENDED TO PREVENT FAULTY SITE DECISIONS BASED ON TECHNICAL, TRANSCRIPTION, AND/OR CALCULATION ERRORS. THE EPA REMEDIAL PROJECT MANAGER (RPM) OR SITE ASSESSMENT MANAGER (SAM) MUST BE CONTACTED TO APPROVE A PARTIAL OR COMPLETE TIER III VALIDATION PRIOR TO ITS INITIATION.

[3]REGION I LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES, 6/13/88, MODIFIED 2/89

ATTACHMENT II

TIER II ORGANIC DATA VALIDATION

TIER II ORGANIC DATA VALIDATION

QC CRITERIA	DATA REPORTING FORMS TO REVIEW	APPLICABLE SECTIONS IN FUNCTIONAL GUIDELINES[3]	COMMENTS
Data Completeness	! Complete SDG File (CSF) 1. Original Sample Data Package including Cover Page, Forms I through X, DC-1, DC-2, raw data 2. Original shipping and receiving documents 3. All original lab records of sample transfer, preparation and analysis, as well as telephone contact logs.		! Perform a Tier I completeness evidence audit according to procedures in the Region I CSF Completeness Evidence Audit Program, dated July 3, 1991, to ensure that all laboratory data and documentation are present. Request missing deliverables from the laboratory following appropriate procedures.
Holding Times VOA & SVOA	! Form Is ! Chain of Custody / Traffic Report ! SDG Narrative	! I. A through D, pp. 21-22	! Examine Chain-of-Custody/Traffic Report Forms to determine if samples were properly preserved in the field. ! To verify sample pH upon laboratory receipt, review the SDG Narrative as this information is not included on the forms.
Pest/PCB		! I. A through D, p. 48	
GC/MS Tuning VOA & SVOA	! Form Vs	! II. A through B, pp. 22-23 C.3.a and c, p. 23 D, pp. 24-26	! Review data reporting forms only. Do not verify with raw data and do not recalculate reported values.
Calibration VOA & SVOA	! Forms IV, VI, VII	! III. A through B, pp. 26-27 C.1.a.2, p. 27 C.1.b.2, p. 28 C.2.a.1, p. 28 C.2.b.2, p. 29 D, pp. 29-30	! Review data reporting forms only. Do not verify with raw data. Do not recalculate %RSD, RRF or %D values. ! Review Form IV to determine the samples associated with each calibration.
Instrument Performance/Calibration Pest/PCB	! Forms VI, VII, VIII, IX	! II. A, p. 49 B.1-4, pp. 49-51 C through D, pp. 51-54 ! III. A through B, pp. 54-55 C.1.c and e, pp. 55-56 C.2, p. 56 D, p. 56	! Review data reporting forms only. Do not verify with raw data and do not recalculate reported values.

SEE NOTE ON PAGE 3 OF 3.

[3]REGION I LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ORGANICS ANALYSES, 2/1/88, MODIFIED 11/1/88

TIER II ORGANIC DATA VALIDATION

QC CRITERIA	DATA REPORTING FORMS TO REVIEW	APPLICABLE SECTIONS IN FUNCTIONAL GUIDELINES[3]	COMMENTS
Blanks VOA & SVOA	! Forms I, IV ! Chain of Custody / Traffic Report	! IV. A through B, p. 30 C.2, pp. 30-31 D, pp. 31-33	! Review data reporting forms only. Do not verify with raw data.
Pest/PCB		! IV. A through B, p. 57 C.2 and 3, p. 57 D, pp. 57-59	
Surrogate Recovery VOA & SVOA	! Form IIs	! V. A through B, pp. 33-34 C.2.a-c, p. 34 C.3.a-c, p. 34 D, pp. 34-35	! Review data reporting forms only. Do not verify with raw data.
Pest/PCB		! V. A through B, p. 59 D, pp. 59-60	
Matrix Spike & Matrix Spike Duplicate VOA & SVOA	! Forms I, III	! VI. A through B, pp. 35-36 C.1 and 3, p. 36 D, pp. 36-37	! Review data reporting forms only. Do not verify with raw data.
Pest/PCB		! VI. A through B, p. 60 C.1 and 3, pp. 60-61 D, p. 61	
Field Duplicates VOA & SVOA	! Form Is ! Chain of Custody / Traffic Report	! VII. A through D, pp. 37-38	! No change from current procedures.
Pest/PCB		! VII. A through D, pp. 61-62	
Internal Standards Performance VOA & SVOA	! Form VIIIs	! VIII. A through B, p. 38 C.2 and 3, p. 38 D, pp. 38-39	! Review data reporting forms only. Do not verify with raw data.
Compound Identification VOA & SVOA	---	---	! Not evaluated during Tier II review.
Pest/PCB	! Forms I, X	! VIII. A, B, pp. 62, 63 C, D, pp. 63, 64	! Review data reporting forms only. Do not verify with raw data.

SEE NOTE ON PAGE 3 OF 3.

[3]REGION I LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ORGANICS ANALYSES, 2/1/88, MODIFIED 11/1/88

TIER II ORGANIC DATA VALIDATION

QC CRITERIA	DATA REPORTING FORMS TO REVIEW	APPLICABLE SECTIONS IN FUNCTIONAL GUIDELINES[3]	COMMENTS
Compound Quantitation & Reported Detection Limits VOA & SVOA	! Form Is ! SDG Narrative	! X. C.4, p. 41 D, p. 41	! Only reported quantitation limits can be evaluated during a Tier II review. ! Review the SDG Narrative to identify and explain any anomalies on the Form Is. Qualify data accordingly. ! Review data reporting forms only. Do not verify with raw data.
Pest/PCB		! IX. C.2, p. 64 D, pp. 64-65	
Tentatively Identified Compounds VOA & SVOA	! Form Is	---	! Verify that target compounds are not reported as TICs in another fraction.
System Performance VOA & SVOA	---	---	! Not evaluated during Tier II review.
Overall Assessment of Data for a Case			! Limit to the sections evaluated during Tier II review.

NOTE: IN CIRCUMSTANCES WHERE THE ENTIRE DATA PACKAGE OR DATA FOR MULTIPLE SAMPLES MUST BE REJECTED OR WILL BE SIGNIFICANTLY QUALIFIED BASED UPON THE TIER II RESULTS, THE REVIEWER MUST FIRST CONSIDER THE IMPACT OF REJECTED RESULTS AND/OR DISCREPANT INFORMATION ON THE DATA NEEDS OF THE SPECIFIC PROJECT. IF THE DATA ARE CRITICAL TO THE PROJECT NEEDS, THEN EXAMINATION OF THE RAW DATA IS STRONGLY RECOMMENDED TO PREVENT FAULTY SITE DECISIONS BASED ON TECHNICAL, TRANSCRIPTION, AND/OR CALCULATION ERRORS. THE EPA REMEDIAL PROJECT MANAGER (RPM) OR SITE ASSESSMENT MANAGER (SAM) MUST BE CONTACTED TO APPROVE A PARTIAL OR COMPLETE TIER III VALIDATION PRIOR TO ITS INITIATION.

[3]REGION I LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES FOR EVALUATING ORGANICS ANALYSES, 2/1/88, MODIFIED 11/1/88

Attachment C

"Region I CSF Completeness Evidence Audit Program", July 1991

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I
60 WESTVIEW STREET, LEXINGTON MA 02173

MEMORANDUM

DATE: August 7, 1991

SUBJ: Region I CSF Completeness Evidence Audit Program

FROM: Moira M. Lataille
Deborah A. Szaro
Region I CLP TPOs

TO: Lead Chemists
Region I Contractors

THRU: Heidi Horahan
ARCs DPO

The attached Region I CSF Completeness Evidence Audit Program/July 3, 1991 replaces the currently used procedure described by CEAT-Techlaw in EPA Regional CSF Completeness Evidence Audit Guidelines. Begin using the Region I CSF CEAP on the next CSF you receive. Note that the forms supplied by CEAT-Techlaw during the Complete SDG File Training seminar held on February 20, 1991 will no longer be utilized. These are replaced by the **EPA Region I Complete SDG File Receipt/Transfer Form** and the **DC-2 Forms.**

To assist you in implementing this new CSF Program, we have set up a CSF Hotline number, (617) 229-2050, at the Region I Weston/ESAT office. Primary contact is Pam Rose and secondary contact is Kate Schweitzer. All questions received by ESAT will be documented with telephone conversation logs. Questions requiring clarification will be forwarded by ESAT to the TPOs and/or NEIC. You will receive an answer to your question within 24 hours or be informed that the question is being researched by the TPO/NEIC and that clarification will be provided as soon as possible. In an effort to save the Lead Chemists' time and reduce the number of repeated questions, a copy of questions and answers received from all Lead Chemists will be provided to each Lead Chemist in a monthly report. Please take the time to read the monthly reports.

Please note the following:

o All CSF data must have the Region I CSF Completeness Evidence Audit performed even if those data are not to be validated at this time.

o Only Lead Chemists may call the CSF Hotline; please identify yourself when you call.

o The Hotline is to be used to resolve technical/legal questions and specific audit

questions after you have read and become familiar with the Region I CSF CEAP. The ESAT contacts will not walk you through an audit.

If you are repeatedly unable to reach either the primary or secondary ESAT contact at the CSF Hotline, call either Deborah Szaro or Moira Lataille at (617) 860-4312.

cc: Carol Wood, QAO
Scott Clifford, ESAT DPO

REGION I CSF COMPLETENESS
EVIDENCE AUDIT PROGRAM

July 3, 1991

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1.0 INTRODUCTION

Evidence audits are conducted to ensure that laboratory documentation and data will be admissible in potential litigation. Prior to the implementation of the OLM01.0 Organic and ILM01.0 Inorganic Statements of Work, evidence audits for all Routine Analytical Services case files were performed by CEAT-Techlaw. However, under the ILM01.0 and OLM01.0 Inorganic and Organic Statements of Work, laboratories must now develop Complete Sample Delivery Group Files (CSFs). The CSFs consist of the original Sample Data Package and all related documentation. Laboratories operating under the new contracts will submit the CSFs directly to the regions, who will now be responsible for conducting the evidence audits. This process allows the EPA to quickly monitor the quality of the laboratory documentation.

To easily integrate the evidence audit into the validation procedure, the Region I Quality Assurance Office has developed the Region I CSF Completeness Evidence Audit Program. The program addresses two fundamental areas of responsibility necessary to ensure the admissibility of laboratory-generated documentation and analytical data as evidence. First, the integrity of the CSF must be maintained during all transfers. Second, the completeness of the CSF documentation must be assured through the evidence audit process.

The Region I CSF Completeness Evidence Audit Program replaces the procedure described by CEAT-Techlaw in EPA Regional CSF Completeness Evidence Audit Guidelines. None of the forms supplied by CEAT-Techlaw at the Complete SDG File Training seminar held on February 20, 1991 will be necessary to complete the Region I CSF Completeness Evidence Audit or to perform the CSF tracking procedures.

A flowchart outlining the Region I CSF Completeness Evidence Audit Program is included in Attachment I.

2.0 COMPONENTS OF THE CSF

The CSF consists of the original Sample Data Package and all related documentation. The laboratory is required to assemble the CSF and submit it directly to the Region (as specified in Exhibit B, Section II, B-22 of OLM01.0 and Exhibit B, Section II,B-13 of ILM01.0). The laboratory submits a Complete SDG File (CSF) Inventory Sheet, **DC-2 Form**, (inorganic pages 1-2, organic pages 1-4), which indexes all

documents submitted in the CSF. In addition to the original Sample Data Package, the CSF consists of the following original documents:

- **!** A completed, signed, and dated Complete SDG File (CSF) Inventory Sheet, **DC-2 Form**;
- **!** All original shipping documents including the EPA chain of custody records, airbills, EPA traffic reports, and sample tags sealed in plastic bags;
- **!** All original receiving documents, including the sample log-in sheet (DC-1 Form), and other receiving forms or copies of receiving logbooks;
- **!** All original laboratory records, not already submitted in the Sample Data Package, concerning internal laboratory sample transfer/tracking, preparation and analysis;
- **!** All other original SDG-specific documents in the laboratory's possession including telephone contact logs, copies of personal logbook pages, and hand written case-specific notes.

3.0 THE CSF TRACKING PROCEDURE

3.1 Tracking Overview

To comply with evidence requirements, signed and dated custody seals must be affixed to the CSF whenever it is transferred. The CSF is considered transferred whenever it changes location upon shipment or hand-delivery. This occurs when the CSF is shipped from the laboratory to the Regional Sample Control Center (RSCC), from the RSCC to the Prime Contractor, from the Prime Contractor to the Data Validation Subcontractor, from the Data Validation Subcontractor to the Prime Contractor, whenever the CSF is requested for oversight by the Region I EPA Quality Assurance Office, or any other time the CSF must change custody.

Data Validation Subcontractors will not be responsible for conducting evidence audits; however, they must be informed of and adhere to the Region I CSF Completeness Evidence Audit Program, CSF Tracking Procedures. The Prime Contractors are responsible for ensuring that all Data Validation Subcontractors are properly trained in the procedures outlined in the tracking procedure.

The CSF Tracking Procedure is initiated when the CSF is received at the RSCC by the Sample Control Coordinator (SCC). The SCC will initiate the **CSF Receipt/Transfer Form**, which will remain with the CSF through every transfer. The purpose of the **CSF Receipt/Transfer Form** is to document the presence and condition of custody seals, which must be affixed to the data package in compliance with evidence audit requirements during all transfers. Examples of blank and completed **CSF Receipt/Transfer Form**s are included in Attachment IIA and IIB.

3.2 CSF Tracking Procedure

The CSF is received at the RSCC from the laboratory under custody seal. The SCC initiates a **CSF Receipt/Transfer Form**, which will remain with the CSF with every transfer. For each transfer, the following protocol for CSF tracking and completion of the **CSF Receipt/Transfer Form** must be followed:

1. Inspect the unopened CSF shipment. Determine if custody seals are present or absent. If present, determine if custody seals are intact or broken.

2. Open the CSF shipment and complete the **CSF Receipt/Transfer Form**. The case number, SDG number, and data package number will be completed by the SCC.

   - **Receipt Date** - Enter the date that the contractor/validator received the CSF;

   - **Received By** - Enter the name and initials of the contractor/validator who has opened the CSF, and list the affiliation, i.e. RSCC, Weston/ESAT, NUS/ARCS, Dynamac, EPA, etc.;

   - **CSF Activity** - List the CSF activity. For example, the SCC will list the activity as "CSF Receipt". The contractor/validator will list the activity as "validation", "resubmittals", "data validation oversight" or "CSF storage";

   - **Custody Seals** - Indicate whether the custody seals were present and intact;

- **Released** - If the CSF must be transferred to a new location, identify which organization the package will be released to and the date of release, i.e. shipment date or hand-delivery date.

3.3 Laboratory Resubmittal Tracking

All laboratory resubmittals requested during the evidence audit and/or data validation must be shipped under custody seal. The Prime Contractor Lead Chemist is the only one authorized to request and receive resubmittals. The Data Validation Subcontractor cannot request or receive resubmittals. The laboratory may send resubmittals to either the RSCC or the Prime Contractor.

If the laboratory sends resubmittals to the RSCC, a new **CSF Receipt/Transfer Form** will be initiated by the SCC. The resubmittals and new **CSF Receipt/Transfer Form** will be shipped to the Prime Contractor Lead Chemist as stated in section 3.2. The Prime Contractor will complete the appropriate section of the new **CSF Receipt/Transfer Form** and will indicate the "CSF Activity" as "Resubmittals". The Prime Contractor will then forward the resubmittals to the Data Validation Subcontractor under custody seal.

However, if the laboratory sends resubmittals directly to the Prime Contractor, a new **CSF Receipt/Transfer Form** will be initiated by the Prime Contractor. The Prime Contractor will complete the appropriate section of the new **CSF Receipt/Transfer Form** and will indicate the "CSF Activity" as "Resubmittals". The Prime Contractor will then forward the resubmittals to the Data Validation Subcontractor under custody seal.

If the Prime Contractor receives resubmittals from both the laboratory and the RSCC, the Prime Contractor must verify that the resubmittals received from the RSCC are identical to those received directly from the laboratory. The Prime Contractor may then discard and recycle the set of resubmittals received from the RSCC. If the two sets of resubmittals are not identical, the Prime Contractor must contact the laboratory to determine which set of resubmittals is correct.

Upon receipt of the resubmittals, the Data Validation Subcontractor will complete the appropriate section of the new **CSF Receipt/Transfer Form**. Under "Released", the Data

Validation Subcontractor should indicate "Included with CSF". All **CSF Receipt/Transfer Forms** and laboratory resubmittals must be kept with the CSF.

3.4 Data Validation Oversight

If the QA Office requests a CSF for data validation oversight, the Prime Contractor must complete the appropriate sections of the **CSF Receipt/Transfer Form** and ship the CSF under custody seal to the EPA. When the data validation oversight is complete, the EPA will complete the appropriate sections of the **CSF Receipt/Transfer Form** and ship the CSF under custody seal to the Prime Contractor.

4.0 THE CSF AUDIT PERFORMANCE PROCEDURE

4.1 CSF Audit Overview

The purpose of the evidence audit is to determine completeness of the CSF as shipped from the laboratory. **The auditor must verify that all documents are present as stated by the laboratory on the DC-2 Form and that <u>all</u> pages in the CSF are accounted for on the DC-2 Form.** All evidentiary documents must be clearly identified with the case number and SDG number, and must be signed and dated where required. The accuracy of the Sample Data Package submitted as part of the CSF is determined during the normal data validation procedure and is not part of the evidentiary audit.

The CSF Audit Performance Procedure outlines the protocol that Prime Contractors must follow to complete the evidence audit. The evidence audit must be completed by Prime Contractors only. Data Validation Subcontractors performing data validation will not be responsible for conducting the evidence audit, although they will be required to adhere to all CSF tracking procedures. The Prime Contractor will perform the evidence audit by reviewing the **DC-2 Form**, which is submitted by the laboratory as part of the CSF. Examples of blank organic and inorganic **DC-2 Form**s are included in Attachment IIIA. Examples of laboratory-completed organic and inorganic **DC-2 Form**s are included in Attachment IIIB. Examples of laboratory-completed and Prime Contractor-completed organic and inorganic **DC-2 Form**s are included in Attachment IIIC.

4.2 Inorganic Completeness Evidence Audit

The following describes the Region I guidelines for

conducting completeness evidence audits of inorganic CSFs. The CSF will be shipped to the Prime Contractor Lead Chemist by the RSCC. A **CSF Receipt/ Transfer Form**, initiated by the SCC, will be shipped with the CSF.

The Prime Contractor Auditor/Validator will perform the evidence audit using a photocopy of each completed and signed **DC-2 Form** which is submitted by the laboratory as part of the CSF or which is submitted with resubmitted documents. The Prime Contractor Auditor/Validator must not write on the original **DC-2 Form**, which will remain with the CSF, unmodified.

When resubmittals are requested, the Prime Contractor Auditor/Validator should request that the laboratory number the resubmitted pages so that they may be appended to the end of the CSF. Pages should not be inserted into the CSF, and original pages in the CSF should not be replaced by resubmitted pages.

When the laboratory resubmittals are received, photocopy the new **DC-2 Form** and perform the evidence audit for the resubmitted sections only. The Prime Contractor Auditor/Validator must not write on the original **DC-2 Form**, which will remain with the CSF, unmodified.

The Prime Contractor Auditor/Validator must generate telephone communication logs whenever the laboratory is contacted for resubmittals or clarification.

Complete the evidence audit according to the following protocol:

1. Inspect the package for custody seals and follow the protocol outlined in the CSF Tracking Procedure. After completing the appropriate sections of the **CSF Receipt/Transfer Form**, proceed with the evidence audit.

2. Locate the CSF Inventory Sheet, **DC-2 Form**, submitted by the laboratory. Make one photocopy of this **DC-2 Form** to perform the evidence audit. At the top of the first page, label the photocopy "Evidence Audit Photocopy". The original **DC-2 Form** submitted by the laboratory must remain with the CSF, unmodified.

   If the **DC-2 Form** is not included with the CSF, contact the laboratory for submittal and complete

a telephone communication log. Resubmittal of just the **DC-2 Form** is not required to be under custody seal. Proceed with the evidence audit after the **DC-2 Form** has been submitted by the laboratory and photocopied by the Prime Contractor Auditor/Validator.

3. Review the documents in the CSF. Compare the document page numbers to the page numbers listed on the **DC-2 Form**. Ensure that all documents are accounted for and legible. If extra pages were included with the CSF but were not listed on the **DC-2 Form**, or if page numbers listed on the **DC-2 Form** were incorrect, request that a corrected **DC-2 Form** be submitted. Complete a telephone communication log.

4. For items 1-27 on the **DC-2 Form,** if the information is accurate and legible, place a check in the EPA column for those items.

   If any pages are missing, inaccurate, or illegible, do not put a check in the EPA column. Request resubmittal of the pages from the laboratory and complete a telephone communication log.

5. For item 28, check whether the traffic report is present. If no, leave EPA column blank, request resubmittal of the pages from the laboratory and complete a telephone communication log.

   Check whether the traffic report was signed and dated. If yes, place a check in the EPA column. If no, leave EPA column blank and indicate the non-compliance directly next to item 28 on the **DC-2 Form**. Do not request a laboratory resubmittal of the traffic report if it was present but not signed or dated.

6. Proceed to item 29. Check whether airbills, chain of custody records, sample tags, sample log-in sheets (DC-1 Form and/or lab form), and the SDG cover sheet are present. If no, leave EPA column blank, request resubmittals from the laboratory, and complete a telephone communication log.

   Check whether the airbills, chain of custody records and SDG cover sheets were signed and

dated. If yes, place a check in the EPA column. If no, leave EPA column blank and indicate the non-compliance directly next to item 29 on the **DC-2 Form**. Do not request laboratory resubmittals of these documents if they were present but not signed and dated.

Check whether the sample log-in sheet/ DC-1 Form are complete and accurate. If yes, place a check in the EPA column. If no, leave EPA column blank and indicate the non-compliance directly next to item 29 on the **DC-2 Form**. Do not request laboratory resubmittals of these documents if they were present but not complete or accurate.

7. Items 30, 31, and 32 concern laboratory documentation including miscellaneous shipping/receiving records, telephone logs, internal laboratory sample transfer/ tracking sheets, and sample preparation and analysis records. Confirm that EPA sample numbers, SDG numbers, and Case numbers are correctly referenced to this particular Case and SDG on all documents submitted by the laboratory. If yes, place a check in the EPA columns. If no, leave EPA columns blank, request that the laboratory resubmit the correct documents and complete a telephone communication log.

8. If there are documents listed in item 33, confirm that EPA sample numbers, SDG numbers, and Case numbers are correctly referenced to this particular Case and SDG on all documents submitted by the laboratory. If yes, place a check in the EPA columns. If no, leave EPA columns blank, request that the laboratory resubmit the correct documents, and complete a telephone communication log.

9. The evidence auditor should sign the "Audited by" section at the bottom of each photocopied **DC-2 Form**. The evidence auditor's printed name, title, and date should also be completed. In addition, the evidence auditor should indicate their company name/contract below the "Printed Name/Title" line.

10. Since resubmittals may be requested during validation, hold all **DC-2 Form**s until the data validation is complete before proceeding with the

distribution of the forms.

11. When requested resubmittals and new **DC-2 Form** are received from the laboratory, make a photocopy of the new **DC-2 Form**. At the top of the first page, label the photocopy "Evidence Audit Photocopy". The original **DC-2 Form** submitted by the laboratory must remain with the CSF, unmodified. Perform the evidence audit for the resubmitted sections on the photocopy of the new **DC-2 Form**. The column on the photocopied **DC-2 Form** for the original data package, which was left blank during the evidence audit pending resubmittals, remains blank.

4.3 Organic Completeness Evidence Audit

The following describes the Region I guidelines for conducting completeness evidence audits of organic CSFs. The CSF will be shipped to the Prime Contractor Lead Chemist by the RSCC. A **CSF Receipt/ Transfer Form**, initiated by the SCC, will be shipped with the CSF.

The Prime Contractor Auditor/Validator will perform the evidence audit using a photocopy of each completed and signed **DC-2 Form** which is submitted by the laboratory as part of the CSF or which is submitted with resubmitted documents. The Prime Contractor Auditor/Validator must not write on the original **DC-2 Form** which will remain unmodified with the CSF.

When resubmittals are requested, the Prime Contractor Auditor/Validator should request the laboratory to number the resubmitted pages so that they may be appended to the end of the CSF. Pages should not be inserted into the CSF and original pages in the CSF should not be replaced by resubmitted pages.

When the laboratory resubmittals are received, photocopy the new **DC-2 Form** and perform the evidence audit for the resubmitted sections only. The Prime Contractor Auditor/Validator must not write on the original **DC-2 Form** which will remain with the CSF, unmodified.

The Prime Contractor Auditor/Validator must generate telephone communication logs whenever the laboratory is contacted for resubmittals or clarification.

Complete the evidence audit according to the following protocol:

1. Inspect the package for custody seals and follow the protocol outlined in the CSF Tracking Procedure. After completing the appropriate sections of the **CSF Receipt/Transfer Form**, proceed with the evidence audit.

2. Locate the CSF Inventory Sheet, **DC-2 Form**, submitted by the laboratory. Make one photocopy of this **DC-2 Form** to perform the evidence audit. At the top of the first page, label the photocopy "Evidence Audit Photocopy". The original **DC-2 Form** submitted by the laboratory must remain with the CSF, unmodified.

   If the **DC-2 Form** is not included with the CSF, contact the laboratory for submittal and complete a telephone communication log. Resubmittal of just the **DC-2 Form** is not required to be under custody seal. Proceed with the evidence audit after the **DC-2 Form** has been submitted by the laboratory and photocopied by the Prime Contractor Auditor/Validator.

3. Review the documents in the CSF. Compare the document numbers to the page numbers listed on the **DC-2 Form**. Ensure that all documents are accounted for and legible.
   If extra pages were included with the CSF but were not listed on the **DC-2 Form**, or if page numbers listed on the **DC-2 Form** were incorrect, request that a corrected **DC-2 Form** be submitted. Complete a telephone communication log.

4. For items 2, 4, 5, and 6 on the **DC-2 Form**, if the information is accurate and legible, place a check in the EPA column for those items.

   If any pages are missing, inaccurate, or illegible, do not check off the EPA column. Request resubmittals from the laboratory and complete a telephone communication log.

5. For item 3, check whether the traffic report is present. If no, leave EPA column blank, request resubmittal of the form, and complete a telephone communication log.

Check whether the traffic report was signed and dated. If yes, place a check in the EPA column. If no, leave the EPA column blank and indicate the non-compliance directly next to item 3 on the **DC-2 Form**. Do not request a laboratory resubmittal of the traffic report if it was present but not signed or dated.

6. Item 7 concerns laboratory documentation including internal laboratory sample transfer/tracking sheets, sample preparation and analysis logbook pages, screening records, and all instrument output, including strip charts from screening activities. Confirm that EPA sample numbers, SDG numbers, and Case numbers are correctly referenced to this particular Case and SDG on all documents submitted by the laboratory. If yes, place a check in the EPA columns. If no, leave the EPA column blank, request that the laboratory resubmit the correct documents, and complete a telephone communication log.

7. Proceed to item 8. Check whether airbills, chain of custody records, sample tags, sample log-in sheets (DC-1 Form and/or lab form), the SDG cover sheet, and miscellaneous shipping/receiving records are present. If no, leave the EPA column blank, request resubmittals from the laboratory, and complete a telephone communication log.

   Check whether the airbills, chain of custody records and SDG cover sheets were signed and dated. If yes, place a check in the EPA column. If no, leave EPA column blank and indicate the non-compliance directly next to item 8 on the **DC-2 Form**. Do not request laboratory resubmittals of these documents if they were present but not signed and dated.

   Check whether the sample log-in sheet/DC-1 Form are complete and accurate. If yes, place a check in the EPA column. If no, leave EPA column blank and indicate the non-compliance directly next to item 8 on the **DC-2 Form**. Do not request laboratory resubmittals of these documents if they were present but not complete or accurate.

8. Item 9 lists all internal laboratory sample transfer records and tracking sheets. Confirm

that EPA sample numbers, SDG numbers, and Case numbers are correctly referenced by the laboratory. If yes, place a check in the EPA columns. If no, leave EPA columns blank, request resubmittals from the laboratory, and complete a telephone communication log.

9. If there are documents listed in item 10, confirm that EPA sample numbers, SDG numbers, and Case numbers are correctly referenced to this particular Case and SDG on all documents submitted by the laboratory. If yes, place a check in the EPA columns. If no, leave EPA columns blank, request resubmittals from the laboratory, and complete a telephone communication log.

10. The evidence auditor should sign the "Audited by" section at the bottom of each photocopied **DC-2 Form**. The evidence auditor's printed name, title, and date should also be completed. In addition, the evidence auditor should indicate their company name/contract below the "Printed Name/Title" line.

11. Since resubmittals may be requested during validation, hold all **DC-2 Forms** until the data validation is complete before proceeding with the distribution of the forms.

12. When requested resubmittals and new **DC-2 Form** are received from the laboratory, make a photocopy of the new **DC-2 Form**. At the top of the first page, label the photocopy "Evidence Audit Photocopy". The original **DC-2 Form** submitted by the laboratory must remain with the CSF, unmodified. Perform the evidence audit for the resubmitted sections on the photocopy of the new **DC-2 Form**. The column on the photocopied **DC-2 Form** for the original data package, which was left blank during the evidence audit pending resubmittals, remains blank.

5.0 POTENTIAL PROBLEMS WITH THE CSF AUDIT PROCESS

The following is a list of guidelines to aid the auditor in determining the appropriate action to take when a CSF or DC-2 deviates from the required format. Examples of situations which would and would not require contacting the laboratory for resubmittals are also included.

5.1 Guidelines for Contacting the Laboratory

The laboratory must be contacted for any problem that affects the completeness or accuracy of the CSF. For example:

- **!** If the CSF contains pages identified with only a laboratory identifier, such as a LIMS project number, the laboratory must be contacted. All pages of the CSF must reference the CLP Case Number and SDG to maintain data completeness. Any pages with only a laboratory or LIMS project number must be resubmitted.

- **!** If the laboratory mistakenly indicates "Not Applicable" for an item and it is obvious that the item is applicable, i.e. the document is present in the CSF, the laboratory must be contacted. For example, if the laboratory mistakenly indicates that the airbills are "NA", then the laboratory must be contacted and the revised DC-2 Form must be resubmitted to indicate the exact page number of the airbills.

- **!** If the DC-2 Form used by the laboratory does not itemize all pages present in the CSF, the laboratory must be contacted. The laboratory may use their own version of the DC-2 Form as long as all items/pages are listed. If the DC-2 Form does not accurately reflect the contents of the CSF, then the laboratory must resubmit the DC-2 Form.

- **!** If the laboratory submits photocopied documentation instead of original documentation, and if the location of the originals is not noted on each photocopy, then the laboratory must be contacted. The entire CSF must be submitted with all original documentation, or the location of the originals must be noted on each photocopy.

  For example, sample tags and air bills must be original documentation. Sample preparation logs and standard preparation logs, which are usually in bound logbooks, may be photocopies.

5.2 Guidelines for <u>Not</u> Contacting the Laboratory

The laboratory does not need to be contacted if problems

do not affect the completeness or accuracy of the CSF. For example:

- If the laboratory uses a different DC-2 Form than the one included in the Region I program (i.e. individual items on the DC-2 Form have slightly different headers than those on the CLP forms), the laboratory does not need to be contacted. As long as all documents are accurately inventoried on the laboratory DC-2 Form and the DC-2 Form accurately reflects the contents of the CSF, then the laboratory does not need to be contacted.

- If the Traffic Report includes the Chain of Custody form, as is the case with the new Traffic Reports, the laboratory does not need to be contacted. The laboratory may list them individually. The duplication of page numbers is inevitable.

- If the laboratory has inserted resubmitted pages into the CSF, the laboratory does not need to be contacted. The laboratory has the option to add the requested resubmittals in an addendum, insert additional pages in the package and renumber the pages or resubmit the page with the original page number.

- If other inconsistencies are found on the DC-2 Form, but the integrity of the package is not affected, then complete the audit and note the deficiency. For example, some laboratories may not check each item individually on the DC-2 Form, but may instead draw a continuous arrow down the column to indicate that all items were checked. If, however, an item that is not applicable to the case is indicated as present by the continuous arrow, note the inconsistency on the DC-2 Form.

- If the laboratory listed <u>both</u> the original and photocopied pages of the shipping documents on the DC-2 Form, the laboratory does not need to be contacted. The laboratory may have listed the photocopied documents under the "Traffic Report" and "EPA Shipping/Receiving Documents" sections and the original documents under "Other Records". As long as the original documentation is included with the CSF, it is not necessary for the

laboratory to resubmit the DC-2 Form with the original documents listed under the "Traffic Report" and "EPA Shipping/Receiving Documents" sections.

6.0 COMPLETION OF EVIDENCE AUDIT AND DISTRIBUTION OF AUDIT FORMS

The audit is complete after data validation has been performed and when all **DC-2 Form**s have been received and audited. Even if data validation is performed by a Data Validation Subcontractor, the Prime Contractor is still responsible for obtaining any resubmittals required by the validation and new **DC-2 Form**s following the protocol outlined above for CSF tracking and auditing.

The photocopied **DC-2 Form**s completed by the evidence auditor, the original laboratory-submitted **DC-2 Form**, and the **CSF Receipt/Transfer Form** should remain with the CSF. The evidence auditor should make a copy of all **DC-2 Form**s that were previously photocopied and completed during the audit procedure. These copies, along with copies of the telephone communication logs, should be sent to:

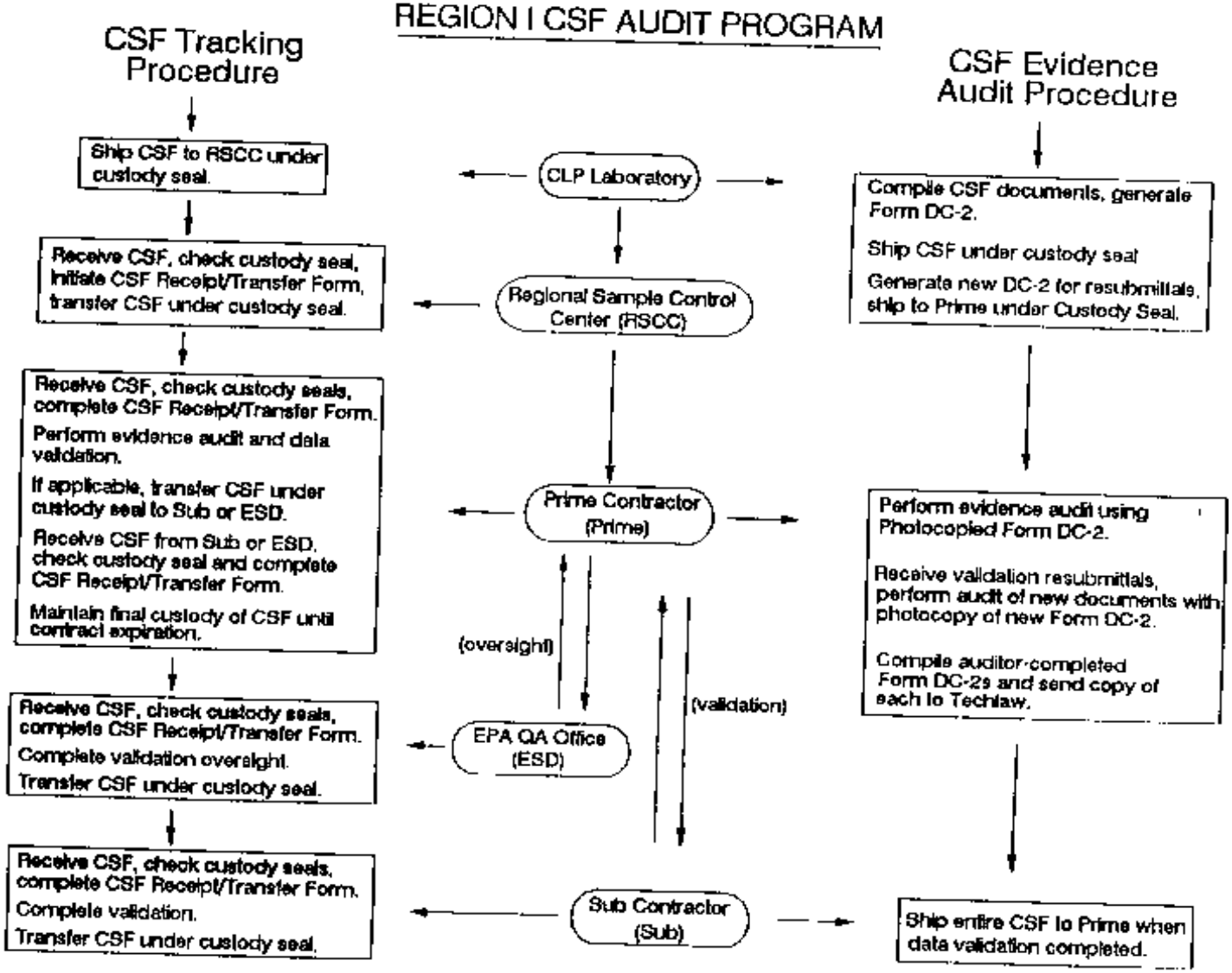
Contract Evidence Audit Team (CEAT-TechLaw)
12600 West Colfax Avenue
Suite C-310
Lakewood, Colorado 80215
Attn: Kerri Luka, Project Leader

When the validation and evidence audit procedures are completed, the CSF remains with the Prime Contractor until contract expiration or until further use of the CSF is required by Region I.

Attachment I

Flowchart of Region I CSF Evidence Audit Program

REGION I CSF AUDIT PROGRAM



Attachment IIA

Blank CSF Receipt/Transfer Form

EPA REGION I
COMPLETE SDG FILE
RECEIPT/TRANSFER FORM

Case #: _____ SDG: _____ Data Package #: _____

Receipt Date	Received by: Name	Init.	Affiliation	CSF Activity	Custody Present (On	Seals: / Intact Receipt)	Released: To	Date
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		

Attachment IIB

Completed CSF Receipt/Transfer Form

EPA REGION I
COMPLETE SDG FILE
RECEIPT/TRANSFER FORM

Case #: 12348 SDG: AA001 Data Package #: 9006040

Receipt Date	Received by: Name	Init.	Affiliation	CSF Activity	Custody Present (On	Seals: / Intact Receipt)	Released: To	Date
2/20/91	Mg Himmel	MH	RSCC Weston/SAT	resubmittals	(Y) N	(Y) N	US/ARCS	2/20/91
2/23/91	Mary Smith	MS	NJS/ARCS	resubmittals	(Y) N	(Y) N	Dynamac	2/23/91
2/23/91	Linda Jones	LJ	Dynamac	resubmittals	(Y) N	(Y) N	included with CSF	
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		

EPA REGION I
COMPLETE SDG FILE
RECEIPT/TRANSFER FORM

Case #: 12348 SDG: AA001 Data Package #: 4006040

Receipt Date	Received by: Name	Init.	Affiliation	CSF Activity	Custody Present (On	Seals: / Intact Receipt)	Released: To	Date
2/3/91	Meg Himmel	MH	RSCC Weston/ESAT	CSF Receipt	(Y) N	(Y) N	NUS/ARCS	2/3/91
2/5/91	Mary Smith	MS	NUS/ARCS	Validation	(Y) N	(Y) N	Dynamac	2/6/91
2/9/91	Linda Jones	LJ	Dynamac	Validation	(Y) N	(Y) N	NUS/ARCS	2/27/91
3/3/91	Mary Smith	MS	NUS/ARCS	CSF Stream	(Y) N	(Y) N	EPA	3/27/91
3/29/91	Steve Stebola	SS	EPA	Data Validation Complete	(Y) N	(Y) N	NUS/ARCS	4/18/91
4/21/91	Mary Smith	MS	NUS/ARCS	CSF Stream	(Y) N	(Y) N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		
					Y N	Y N		

Attachment IIIA

Blank Organic and Inorganic DC-2 Forms

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____ CITY/STATE _____

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ SAS NO. _____

CONTRACT NO. _____ SOW NO. _____ IFB NO. _____

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II F AND SECTION III H)

	PAGE NOs FROM	TO	CHECK LAB	KY
1. Inventory Sheet (Form DC-2) (Do not number)				
2. SDG Case Narrative				
3. Traffic Report				
4. Volatiles Data				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II VOA)				
MS/MSD Duplicate Summary (Form III VOA)				
Method Blank Summary (Form IV VOA)				
Tuning and Mass Calibration (Form V VOA)				
b. Sample Data				
TCL Results - (Form I VOA)				
Tentatively Identified Compounds (Form I VOA-TIC)				
Reconstructed total ion chromatograms (RIC) for each sample				
For each sample:				
Raw spectra and of background-subtracted mass spectra of target compounds identified				
Mass spectra of all reported TIC's with three best library matches				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI VOA)				
RICs and Quan Reports for all Standards				
Continuing Calibration (Form VII VOA)				
RICs and Quan Reports for all Standards				
Internal Standard Area Summary (Form VIIIA VOA)				
d. Raw QC Data				
BFB				
Blank Data				
Matrix Spike Data				
Matrix Spike Duplicate Data				
5. Semivolatile Data				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II SV)				
MS/MSD Summary (Form III SV)				
Method Blank Summary (Form IV SV)				
Tuning and Mass Calibration (Form V SV)				

ORGANIC COMPLETE EDG FILE (CSF) INVENTORY SHEET (Contd)

CASE NO. _____ EDG NO. _____ EDG NOS. TO FOLLOW _____ SDG NO. _____

	PAGE NOs FROM	TO	CHECK LAB	EV
3. Semivolatiles Data (Contd)				
b. Sample Data				
TCL Results (Form I SV)				
Tentatively Identified Compounds (Form I SV-TIC)				
Reconstructed total ion chromatograms (RIC) for each sample				
For each sample:				
Raw spectra and background-subtracted mass spectra of TCL compounds				
Mass spectra of TIC's with 3 best library matches				
GPC chromatograms (if GPC performed)				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI SV)				
RICs and Quan Reports for all standards				
Continuing Calibration (Form VII SV)				
RICs and Quan Reports for all standards				
Internal Standard Area Summary (Form VIII SV)				
Internal Standard Area Summary (Form VIII SV)				
d. Raw QC Data				
DFTPP				
Blank Data				
Matrix Spike Data				
Matrix Spike Duplicate Data				
6. Pesticides				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II Pest)				
MS/MSD Duplicate Summary (Form III Pest)				
Method Blank Summary (Form IV Pest)				
b. Sample Data				
TCL Results - Organic Analysis Data Sheet (Form I Pest)				
Chromatograms (Primary Column)				
Chromatograms from second GC column confirmation				
GC Integration report or data system printout and calibration plots				
Manual work sheets				
UV traces from GPC (if available)				
For pesticides/PCBs confirmed by GC/MS, copies of raw spectra and copies of background-subtracted mass spectra of target compounds (samples & standards)				

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ SAS NO. _____

	PAGE NOS FROM	TO	CHECK LAB	X
6. Pesticides (Contd)				
c. Standards Data				
Pesticides Evaluation Standards Summary (Form VIII, Pest-1)				
Pesticides Evaluation Standards Summary (Form VIII, Pest-2)				
Pesticide/PCB Standards Summary (Form II, Pest)				
Pesticide/PCB Identification (Form X, Pest)				
Standard chromatograms and data system printout for all standards				
For pesticides/PCBs confirmed by GC/MS, copies of spectra for standards used				
d. Raw QC Data				
Blank Data				
Matrix Spike Data				
Matrix Spike Duplicate Data				
7. Miscellaneous Data				
Original preparation and analysis forms or copies of preparation and analysis logbook pages				
Internal sample and sample extract transfer chain-of-custody records				
Screening records				
All instrument output, including strip charts from screening activities (describe or list)				
8. EPA Shipping/Receiving Documents				
Airbills (No. of shipments ___)				
Chain-of-Custody Records				
Sample Tags				
Sample Log-In Sheet (Lab & DCI)				
SDG Cover Sheet				
Miscellaneous Shipping/Receiving Records (describe or list)				
9. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list)				

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont'd)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ SAS NO. _____

	PAGE Nos FROM	TO	CHECK LAB	EP
10. Other Records (describe or list) Telephone Communication Log				

11. Comments: _____

Completed by: (CLP Lab) _____ (Signature) _____ (Printed Name/Title) _____ (Date)

Audited by: (EPA) _____ (Signature) _____ (Printed Name/Title) _____ (Date)

INORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____ CITY/STATE _____

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ SAS NO. _____

CONTRACT NO. _____ SOW NO. _____ IFB NO. _____

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II G AND SECTION III D)

	PAGE NOs FROM	TO	CHECK LAB	KP
1. Inventory Sheet (Form DC-2) (Do not number)				
2. Cover Page				
3. Inorganic Analysis Data Sheet (Form I)				
4. Initial and Continuing Calibration Verification (Form II, Part 1)				
5. CRDL Standard for AA and ICP (Form II, Part 2)				
6. Blanks (Form III)				
7. ICP Interference Check Sample (Form IV)				
8. Spike Sample Recovery (Form V, Part 1)				
9. Post Digest Spike Sample Recovery (Form V, Part 2))				
10. Duplicates (Form VI)				
11. Laboratory Control Sample (Form VII)				
12. Standard Addition Results (Form VIII)				
13. ICP Serial Dilutions (Form IX)				
14. Instrument Detection Limits, Quarterly (Form X)				
15. ICP Interelement Correction Factors, Annually (Form XI, Part 1)				
16. ICP Interelement Correction Factors, Annually (Form XI, Part 2)				
17. ICP Linear Ranges Quarterly (Form XII)				
18. Preparation Log (Form XIII)				
19. Analysis Run Log (Form XIV)				
20. ICP Raw Data				
21. Flame AA Raw Data				
22. Furnace AA Raw Data				
23. Mercury Raw Data				
24. Cyanide Raw Data				
25. Percent Solids Calculations				

INORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ _____ _____ SAS NO. _____

	PAGE NOS. FROM	TO	CHECK LAB	EPA
26. Distillation Logs (Cyanides Only)				
27. Digestion Logs				
28. Traffic Report				
29. EPA Shipping/Receiving Documents				
Airbills (No. of shipments ___)				
Chain-of-Custody Records				
Sample Tags				
Sample Log-In Sheet (Lab & Form DC-1)				
SDG Cover Sheet				
30. Miscellaneous Shipping/Receiving Records (list all individual records)				
Telephone Logs				
_____				
_____				
31. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list)				
_____				
_____				
32. Internal Original Sample Preparation and Analysis Records (describe or list)				
Preparation records _____				
Analysis records _____				
Description _____				
33. Other Records (describe or list)				
_____				
_____				
_____				

34. Comments:

_____

_____

Completed by: (CLP Lab) _____ (Signature) _____ (Printed Name/Title) _____ (Date)

Audited by: (EPA) _____ (Signature) _____ (Printed Name/Title) _____ (Date)

Attachment IIIB

Laboratory-Completed Organic and Inorganic DC-2 Forms

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME: ABC Lab CITY/STATE: Boston, Mass.

CASE NO. 2344 SDG NO. A001 SDG NOS. TO FOLLOW: No

MAX NO. OF SDGS: 10

CONTRACT NO. 34329 SOW NO. 190 LFS NO. 04/08/0

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION IX F AND SECTION III E)

	Page Nos. FROM	TO	Check	
1. Inventory Sheet (Form DC-2) (Do not number)				
2. SDG Case Narrative	1	3	✓	—
3. Traffic Report	4	5	✓	—
4. Volatiles Data	6	6	✓	—
a. QC Summary				
Surrogate Percent Recovery Summary (Form II VOA)	7	9	✓	—
MS/MSD Duplicate Summary (Form III VOA)	10	14	✓	—
Method Blank Summary (Form IV VOA)	15	19	✓	—
Tuning and Mass Calibration (Form V VOA)	20	23	✓	—
b. Sample Data				
TCL Results - (Form I VOA)	24	24	✓	—
Tentatively Identified Compounds (Form I VOA-TIC)			✓	—
Reconstructed total ion chromatograms (RIC) for each sample			✓	—
For each sample:				
Raw spectra and of background-subtracted mass spectra of target compounds identified			✓	—
Mass spectra of all reported TIC's with three best library matches			✓	—
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI VOA)	153	153	✓	—
RICs and Quan Reports for all Standards	154	162	✓	—
Continuing Calibration (Form VII VOA)	163	170	✓	—
RICs and Quan Reports for all Standards	171	190	✓	—
Internal Standard Area Summary (Form VIIIA VOA)	191	196	✓	—
d. Raw QC Data				
BFB				
Blank Data	197	209	✓	—
Matrix Spike Data	210	220	✓	—
Matrix Spike Duplicate Data	221	221	✓	—
	222	240	✓	—
5. Semivolatiles Data				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II SV)	241	260	✓	—
MS/MSD Summary (Form III SV)	261	270	✓	—
Method Blank Summary (Form IV SV)	271	278	✓	—
Tuning and Mass Calibration (Form V SV)	279	287	✓	—

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont'd)

CASE No. 2346 SDG No. [illegible] SDG NOS. TO FOLLOW N/A

SAS No. N/A

	PAGE NOS. FROM	TO	CHECK LAB	X
5. Semivolatiles Data (Cont'd)				
b. Sample Data				
TCL Results (Form I SV)	285	288	✓	
Tentatively Identified Compounds (Form I SV-TIC)			✓	
Reconstructed total ion chromatograms (RIC) for each sample			✓	
For each sample:				
Raw spectra and background-subtracted mass spectra of TCL compounds			✓	
Mass spectra of TIC's with 3 best library matches			✓	
GPC chromatograms (if GPC performed)			✓	
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI SV)	391	402	✓	
RICs and Quan Reports for all standards				
Continuing Calibration (Form VII SV)	403	422	✓	
RICs and Quan Reports for all standards				
Internal Standard Area Summary (Form VIIIE SV)	423	435	✓	
Internal Standard Area Summary (Form VIIIC SV)	436	451	✓	
d. Raw QC Data				
DFTPP				
Blank Data	452	470	✓	
Matrix Spike Data	471	486	✓	
Matrix Spike Duplicate Data	484	497	✓	
	498	510	✓	
6. Pesticides				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II Pest)	511	521	✓	
MS/MSD Duplicate Summary (Form III Pest)	521	531	✓	
Method Blank Summary (Form IV Pest)	530	532	✓	
b. Sample Data				
TCL Results - Organic Analysis Data Sheet (Form I Pest)	535	539	✓	
Chromatograms (Primary Column)			✓	
Chromatograms from second GC column confirmation			✓	
GC Integration report or data system printout and calibration plots			✓	
Manual work sheets			✓	
UV traces from GPC (if available)			✓	
For pesticides/PCBs confirmed by GC/MS, copies of raw spectra and copies of background-subtracted mass spectra of target compounds (samples & standards)			✓	

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CASE NO. 12346 SDG NO. 24001 SDG NOS. TO FOLLOW N/A

PAGE NO. 6/7

	PAGE NOS. FROM	TO	CHECK LAB	X
6. Pesticides (Contd)				
c. Standards Data				
Pesticides Evaluation Standards Summary (Form VIII, Pest-1)	583	584	✓	
Pesticides Evaluation Standards Summary (Form VIII, Pest-2)	595	598	✓	
Pesticide/PCB Standards Summary (Form IX, Pest)	600	615	✓	
Pesticide/PCB Identification (Form X, Pest)	616	619	✓	
Standard chromatograms and data system printout for all standards	620	632	✓	
For pesticides/PCBs confirmed by GC/MS, copies of spectra for standards used	633	640	✓	
d. Raw QC Data				
Blank Data	641	652	✓	
Matrix Spike Data	653	670	✓	
Matrix Spike Duplicate Data	671	677	✓	
7. Miscellaneous Data				
Original preparation and analysis forms or copies of preparation and analysis logbook pages	678	690	✓	
Internal sample and sample extract transfer chain-of-custody records	691	701	✓	
Screening records	702	707	✓	
All instrument output, including strip charts from screening activities (describe or list)				
Strip Charts	708	740	✓	
8. RPA Shipping/Receiving Documents				
Airbills (No. of shipments 1)	741	750	✓	
Chain-of-Custody Records	751	753	✓	
Sample Tags	754	771	✓	
Sample Log-In Sheet (Lab & DCI)	772	773	✓	
SDG Cover Sheet	774	774	✓	
Miscellaneous Shipping/Receiving Records (describe or list)				
N/A				
9. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list)				
N/A				

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont'd)

CASE NO. 13148 SDG NO. [illegible] SDG NOS. TO FOLLOW - N/A

SAS NO.

	PAGE NOS. FROM	TO	CHECK YES	N
10. Other Records (describe or list) Telephone Communication Log	775	782	✓	

11. Comments:

Completed by: (CLP Lab) John Smith (Signature) John Smith / Sample Control Custodian (Printed Name/Title) 2/2/91 (Date)

Audited by: (RPA) (Signature) (Printed Name/Title) (Date)

INORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME - ABC LAB CITY/STATE BOSTON - MASS

CASE NO. 1234 SDG NO. [illegible] SDG NOS. TO FOLLOW N/A

CONTRACT NO. 5 4921 SOW NO. 3/90 IFB NO. [illegible] RAS NO. 13

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II G AND SECTION III D)

	PAGE NOS FROM	TO	CHECK LAB	EPA
1. Inventory Sheet (Form DC-2) (Do not number)			✓	
2. Cover Page	1	1	✓	
3. Inorganic Analysis Data Sheet (Form I)	2	20	✓	
4. Initial and Continuing Calibration Verification (Form II, Part 1)	21	27	✓	
5. CRDL Standard for AA and ICP (Form II, Part 2)	27	35	✓	
6. Blanks (Form III)	36	47	✓	
7. ICP Interference Check Sample (Form IV)	48	59	✓	
8. Spike Sample Recovery (Form V, Part 1)	60	65	✓	
9. Post Digest Spike Sample Recovery (Form V, Part 2))	66	69	✓	
10. Duplicates (Form VI)	70	75	✓	
11. Laboratory Control Sample (Form VII)	76	90	✓	
12. Standard Addition Results (Form VIII)	91	101	✓	
13. ICP Serial Dilutions (Form IX)	102	109	✓	
14. Instrument Detection Limits, Quarterly (Form X)	110	135	✓	
15. ICP Interelement Correction Factors, Annually (Form XI, Part 1)	136	141	✓	
16. ICP Interelement Correction Factors, Annually (Form XI, Part 2)	142	150	✓	
17. ICP Linear Ranges Quarterly (Form XII)	151	170	✓	
18. Preparation Log (Form XIII)	171	190	✓	
19. Analysis Run Log (Form XIV)	191	190	✓	
20. ICP Raw Data	191	195	✓	
21. Flame AA Raw Data	196	210	✓	
22. Furnace AA Raw Data	211	219	✓	
23. Mercury Raw Data	220	227	✓	
24. Cyanide Raw Data	228	240	✓	
25. Percent Solids Calculations	241	250	✓	

INORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CLIENT NO. 12348 SDG NO. [illegible] SDG NOS. TO FOLLOW NA CASE NO. NA

	PAGE NOS. FROM	PAGE NOS. TO	CHECK LAB	CHECK EPA
26. Distillation Logs (Cyanides Only)	251	256	✓	
27. Digestion Logs	257	280	✓	
28. Traffic Report	281	281	✓	
29. EPA Shipping/Receiving Documents				
Airbills (No. of shipments)	282	285	✓	
Chain-of-Custody Records	286	290	✓	
Sample Tags	291	315	✓	
Sample Log-In Sheet (Lab & Form DC-1)	316	317	✓	
SDG Cover Sheet	318	318	✓	
30. Miscellaneous Shipping/Receiving Records (list all individual records)				
Telephone Logs	319	319	✓	
31. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list) NA				
32. Internal Original Sample Preparation and Analysis Records (describe or list)				
Preparation records	320	322	✓	
Analysis records	323	340	✓	
Description				
33. Other Records (describe or list) NA				
34. Comments:				

Completed by: (CLP Lab) John Smith (Signature) John Smith / Sample Control Custodian (Printed Name/Title) 2/2/9 (Date)

Audited by: (EPA) (Signature) (Printed Name/Title) (Date)

FORM DC-2-2

Attachment IIIC

Laboratory and Contractor-Completed
Organic and Inorganic DC-2 Form

EVIDENCE AUDIT PHOTO

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME: AEC LAB CITY/STATE: Boston Mass

CASE NO. 2346 SDG NO. M001 SDG NOS. TO FOLLOW: NA SAS NO. NA

CONTRACT NO. 54329 SOW NO. 3190 IFB NO. [illegible]

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II F AND SECTION III B)

	Page Nos. From	To	Check Lab	DCI
1. Inventory Sheet (Form DC-2) (Do not number)			✓	✓
2. SDG Case Narrative	1	3	✓	✓
3. Traffic Report	4	5	✓	✓
4. Volatiles Data	6	6	✓	✓
a. QC Summary				
Surrogate Percent Recovery Summary (Form II VOA)	7	9	✓	✓
MS/MSD Duplicate Summary (Form III VOA)	10	14	✓	✓
Method Blank Summary (Form IV VOA)	15	19	✓	✓
Tuning and Mass Calibration (Form V VOA)	20	23	✓	✓
b. Sample Data				
TCL Results - (Form I VOA)	24	24	✓	✓
Tentatively Identified Compounds (Form I VOA-TIC)			✓	✓
Reconstructed total ion chromatograms (RIC) for each sample			✓	✓
For each sample:				
Raw spectra and of background-subtracted mass spectra of target compounds identified			✓	✓
Mass spectra of all reported TIC's with three best library matches			✓	✓
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI VOA)	153	153	✓	✓
RICs and Quan Reports for all Standards	154	162	✓	✓
Continuing Calibration (Form VII VOA)	163	170	✓	✓
RICs and Quan Reports for all Standards	171	190	✓	✓
Internal Standard Area Summary (Form VIII VOA)	191	196	✓	✓
d. Raw QC Data				
BFB				
Blank Data	197	209	✓	✓
Matrix Spike Data	210	220	✓	✓
Matrix Spike Duplicate Data	221	221	✓	✓
	222	240	✓	✓
5. Semivolatiles Data				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II SV)	241	260	✓	✓
MS/MSD Summary (Form III SV)	261	270	✓	✓
Method Blank Summary (Form IV SV)	271	278	✓	✓
Tuning and Mass Calibration (Form V SV)	279	287	✓	✓

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CASE NO. 2346 SDC NO. [illegible] SDG NOS. TO FOLLOW N/A

SAS NO. N/A

	PAGE NOS. FROM	TO	CHECK LAB	REV
5. Semivolatiles Data (Contd)				
b. Sample Data				
TCL Results (Form I SV)	255	255	✓	✓
Tentatively Identified Compounds (Form I SV-TIC)			✓	✓
Reconstructed total ion chromatograms (RIC) for each sample			✓	✓
For each sample:				
Raw spectra and background-subtracted mass spectra of TCL compounds			✓	✓
Mass spectra of TIC's with 3 best library matches			✓	✓
GPC chromatograms (if GPC performed)			✓	✓
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI SV)	391	402	✓	✓
RICs and Quan Reports for all standards				
Continuing Calibration (Form VII SV)	403	412	✓	✓
RICs and Quan Reports for all standards				
Internal Standard Area Summary (Form VIIIB SV)	413	435	✓	✓
Internal Standard Area Summary (Form VIIIC SV)	436	451	✓	✓
d. Raw QC Data				
DFTPP				
Blank Data	452	470	✓	✓
Matrix Spike Data	471	496	✓	✓
Matrix Spike Duplicate Data	466	497	✓	✓
	498	510	✓	✓
6. Pesticides				
a. QC Summary				
Surrogate Percent Recovery Summary (Form II Pest)	511	521	✓	✓
MS/MSD Duplicate Summary (Form III Pest)	521	531	✓	✓
Method Blank Summary (Form IV Pest)	530	535	✓	✓
b. Sample Data				
TCL Results - Organic Analysis Data Sheet (Form I Pest)	535	535	✓	✓
Chromatograms (Primary Column)			✓	✓
Chromatograms from second GC column confirmation			✓	✓
GC Integration report or data system printout and calibration plots			✓	✓
Manual work sheets			✓	✓
UV traces from GPC (if available)			✓	✓
For pesticides/PCBs confirmed by GC/MS, copies of raw spectra and copies of background-subtracted mass spectra of target compounds (samples & standards)			✓	✓

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CASE NO. 12398 SDG NO. A8001 SDG NOS. TO FOLLOW NA

FILE NO. N

	PAGE NOs FROM	TO	LAB	CONTR
6. Pesticides (Contd)				
c. Standards Data				
Pesticides Evaluation Standards Summary (Form VIII, Part-1)	588	594	✓	—
Pesticides Evaluation Standards Summary (Form VIII, Part-2)	595	599	—	—
Pesticide/PCB Standards Summary (Form IX, Part)	600	615	✓	—
Pesticide/PCB Identification (Form X, Part)	616	619	✓	—
Standard chromatograms and data system printout for all standards	620	632	—	—
For pesticides/PCBs confirmed by GC/MS, copies of spectra for standards used	633	640	—	—
d. Raw QC Data				
Blank Data	641	652	—	—
Matrix Spike Data	653	670	✓	✓
Matrix Spike Duplicate Data	671	677	✓	✓
7. Miscellaneous Data				
Original preparation and analysis forms or copies of preparation and analysis logbook pages	678	690	✓	✓
Internal sample and sample extract transfer chain-of-custody records	691	701	✓	✓
Screening records	702	717	✓	✓
All instrument output, including strip charts from screening activities (describe or list)				
SDG Log-ins	718	740	—	—
	—	—	—	—
8. EPA Shipping/Receiving Documents				
Airbills (No. of shipments 1)				
Chain-of-Custody Records — not signed	741	750	✓	—
Sample Tags	751	753	✓	—
Sample Log-In Sheet (Lab & Dec)	754	771	✓	✓
SDG Cover Sheet	772	773	✓	✓
Miscellaneous Shipping/Receiving Records (describe or list)	774	774	—	✓
NA	—	—	—	—
9. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list)				
NA	—	—	—	—

ORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont'd)

CASE NO. 12345 SDG NO. 4444 SDG NOS. TO FOLLOW UA

DAS NO. A

	PAGE NOS FROM	TO	CHECK LAB	R
10. Other Records (describe or list) Telephone Communication Log				
11. Comments:				

Completed by: (CLP Lab) John Smith (Signature) John Smith/Source Control Curator (Printed Name/Title) 2/9/91 (Date)

Audited by: (RPA) Mary Smith (Signature) Mary Smith/Validator (Printed Name/Title) 2/23/9 (Date)

NLS/ARCS

EVIDENCE AUDIT PHOTOC

INORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME: ABC LAB CITY/STATE: BOSTON MASS

CASE NO. 1234 SDG NO. ABC01 SDG NOS. TO FOLLOW N/A

CONTRACT NO. 5492 SOW NO. 3/90 SYS NO. 1-ABC10

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II G AND SECTION III D)

	PAGE NOs FROM	TO	CHECK LAB	EPA
1. Inventory Sheet (Form DC-2) (Do not number)			✓	✓
2. Cover Page	1	1	✓	✓
3. Inorganic Analysis Data Sheet (Form I)	2	20	✓	✓
4. Initial and Continuing Calibration Verification (Form II, Part 1)	21	27	✓	✓
5. CRDL Standard for AA and ICP (Form II, Part 2)	27	35	✓	✓
6. Blanks (Form III)	36	47	✓	✓
7. ICP Interference Check Sample (Form IV)	48	59	✓	✓
8. Spike Sample Recovery (Form V, Part 1)	60	65	✓	✓
9. Post Digest Spike Sample Recovery (Form V, Part 2))	66	69	✓	✓
10. Duplicates (Form VI)	70	75	✓	✓
11. Laboratory Control Sample (Form VII)	76	90	✓	✓
12. Standard Addition Results (Form VIII)	91	101	✓	✓
13. ICP Serial Dilutions (Form IX)	102	109	✓	✓
14. Instrument Detection Limits, Quarterly (Form X)	110	135	✓	✓
15. ICP Interelement Correction Factors, Annually (Form XI, Part 1)	136	141	✓	
16. ICP Interelement Correction Factors, Annually (Form XI, Part 2)	142	150	✓	✓
17. ICP Linear Ranges Quarterly (Form XII)	151	170	✓	✓
18. Preparation Log (Form XIII)	171	180	✓	✓
19. Analysis Run Log (Form XIV)	181	190	✓	✓
20. ICP Raw Data	191	195	✓	✓
21. Flame AA Raw Data	196	210	✓	✓
22. Furnace AA Raw Data	211	219	✓	✓
23. Mercury Raw Data	220	227	✓	✓
24. Cyanide Raw Data	228	240	✓	✓
25. Percent Solids Calculations	241	250	✓	✓

INORGANIC COMPLETE SDG FILE (CSF) INVENTORY SHEET (Contd)

CLP No. 12348 SDG No. A9CC07 SDG Nos. To Follow N/A SAS No. N/A

	Page Nos. From	Page Nos. To	Check Lab	Check EPA
26. Distillation Logs (Cyanides Only)	251	256	✓	—
27. Digesting Logs	257	280	✓	—
28. Traffic Report	281	287	✓	—
29. EPA Shipping/Receiving Documents				
Airbills (No. of shipments 1)	282	285	✓	✓
Chain-of-Custody Records not signed	286	290	✓	—
Sample Tags	291	315	✓	✓
Sample Log-In Sheet (Lab & Form DC-1)	316	317	✓	✓
SDG Cover Sheet	318	318	✓	—
30. Miscellaneous Shipping/Receiving Records (list all individual records)				
Telephone Logs	319	319	✓	—
31. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list)				
NA				
32. Internal Original Sample Preparation and Analysis Records (describe or list)				
Preparation records	320	327	✓	✓
Analysis records	328	340	✓	✓
Description				
33. Other Records (describe or list)				
NA				
34. Comments:				

Completed by: (CLP Lab) John Smith (Signature) John Smith / Sample Control Custodian (Printed Name/Title) 2/2/91 (Date)

Audited by: (EPA) Mary Smith (Signature) Mary Smith / Validator (Printed Name/Title) 2/5/91 (Date)

NUS/ARCS

FORM DC-2-2

Attachment D

“Specifications and Guidance for Contaminant-Free Sample Containers”-
Publication 9240.0-05A, EPA/540/R-93/051
December 1992

For hardcopy of Attachment D contact:

National Technical Information Service

703-605-6000

Attachment E

“User’s Guide to the Contract Laboratory Program”
EPA/504/P-91/002
January 1991

For hardcopy of Attachment E contact:

National Technical Information Service

703-487-4650

Attachment F

Region I Short Sheets and CLP Information Sheets

For hardcopy of Attachment F contact:

Steve Stodola, U.S. EPA Region I
TEL: 781-860-4634
EMAIL: stodola.steve@epamail.epa.gov

For electronic copies of related documents see:

http://www.epa.gov/superfund/oerr/aoc/

(Click on CLP Products and Services)

USEPA
CONTRACT LABORATORY PROGRAM
SHORT SHEETS

AVAILABLE SHORT SHEETS

TITLE	DOCUMENT DATE OR NUMBER		SHORT SHEET REVISION NUMBER
USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, Multi-Concentration	2/88		1.0
USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, Multi-Concentration	OLM01.0	1.0	
USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, Multi-Concentration	OLM01.9	3.0	
USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, Multi-Concentration	OLM02.1	2.0	
USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, High-Concentration	9/88		1.0
USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, Multi-Concentration	7/88		1.0
USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, Multi-Concentration	ILM01.0	1.0	
USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, Multi-Concentration	ILM02.1	2.0	
USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, Multi-Concentration	ILM03.0	2.0	
USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, High-Concentration	IHC01.2	1.0	

AVAILABLE SHORT SHEETS

TITLE	DOCUMENT DATE OR NUMBER	SHORT SHEET REVISION NUMBER
USEPA Contract Laboratory Program Statement of Work for Analysis of Polychlorinated Dibenzo-p-Dioxins (PCDD) and PolychlorinatedDibenzofurans (PCDF) Multi-Media, Multi-Concentration	DFLM01.2	3.0
Superfund Analytical Methods for Low Concentration Water for Organics Analysis	10/92	2.0
USEPA Contract Laboratory Program Water Quality Parameters in Multi-Concentration Water (WQP)	6/93	2.0
USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, Multi-Concentration	ILM04.0	0.0
USEPA Contract Laboratory Program Statement of Work for Organic Analysis Multi-Media, Multi-Concentration	OLM03.2	0.0

SOW SHORT SHEET GUIDELINES

The following is a description of the categories included in the Statement of Work (SOW) Short Sheets.

! **Title**: Name of the document as it appears on the cover page of the document.

! **Document Number**: Number of the document as it appears on the cover of the document. Not all SOWs have associated document numbers. A SOW may be referenced by either the document date or the document number, depending on which one is applicable.

! **Document Date**: Date the document was initially issued according to the cover page of the document. Not all SOWs have associated document dates. A SOW may be referenced by either the document date or the document number, depending on which one is applicable.

! **Effective Dates**: Range of dates for which the Regions can submit samples for analysis under a particular SOW. If contracts have not been awarded yet, then the expected award date is listed.

! **Concentration**: Range of sample concentrations for which the SOW is applicable, such as low to medium, high, or > 20 mg/kg.

! **Data Turnaround**: Number of days the laboratory has to submit the complete data package after sample receipt.

! **Matrices**: Sample matrices for which the SOW is applicable, such as aqueous, soil, sediment, multi-phase, etc.

! **Significant Features**: Information about the SOW which distinguishes it from other SOWs. This section highlights critical items such as holding times, concentrations and matrices which may be different.

! **Revisions/Modifications**: Revisions from the previous SOW which may significantly affect data useability.

! **Recommended Uses**: Explanation of appropriate Superfund activities for which the SOW may be utilized.

! **Analytes/CRQLs**: References Attachment I which lists the parameters included in the analysis and their respective CRQLs or CRDLs. The aqueous and soil CRQLs and CRDLs are listed. "Notes" are provided at the bottom of each Attachment to document deviations from the CRQLs and CRDLs listed.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	Not Applicable
DOCUMENT DATE:	February 1988
EFFECTIVE DATES:	January 20, 1989 through September 10, 1991
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- Volatiles and semivolatiles are analyzed by GC/MS; pesticides/PCBs are analyzed by GC/ECD.
- Major Tentatively Identified Compounds (TICs) are reported for GC/MS analyses.
- Second column confirmation by GC/ECD is required for all pesticides/PCBs. Pesticides/PCBs which are identifie concentrations above 10 ng/uL are confirmed by GC/MS analysis.

REVISIONS/MODIFICATIONS

The 9/88 and 4/89 revisions to the 2/88 SOW do not significantly affect data useability.

RECOMMENDED USES

This Routine Analytical Services (RAS) method is recommended for broad spectrum analysis to define the na of potential site contamination during SSI, LSI, and RI/FS activities. This method is suitable when a thirty five day tu results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential co be present at significant risk levels.

* Sediment samples with high moisture content should be solicited as RAS + SAS (Special Analytical Service) achieve the CRQLs.

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
FEBRUARY, 1988

TARGET COMPOUND LIST - VOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Chloromethane	10	10
Bromomethane	10	10
Vinyl Chloride	10	10
Chloroethane	10	10
Methylene Chloride	5	5
Acetone	10	10
Carbon Disulfide	5	5
1,1-Dichloroethene	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene(total)	5	5
Chloroform	5	5
1,2-Dichloroethane	5	5
2-Butanone	10	10
1,1,1-Trichloroethane	5	5
Carbon Tetrachloride	5	5
Vinyl Acetate	10	10
Bromodichloromethane	5	5

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
Trichloroethene	5	5
Dibromochloromethane	5	5
1,1,2-Trichloroethane	5	5
Benzene	5	5
trans-1,3-Dichloropropene	5	5
Bromoform	5	5
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	5	5
Toluene	5	5
1,1,2,2-Tetrachloroethene	5	5
Chlorobenzene	5	5
Ethyl Benzene	5	5
Styrene	5	5
Total Xylenes	5	5

NOTE:

- **!** THE SAMPLE-SPECIFIC CRQLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHE ABOVE.
- **!** MEDIUM LEVEL SOIL CRQL = 125 x AQUEOUS CRQL REPORTED IN UG/KG.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
FEBRUARY, 1988

TARGET COMPOUND LIST - SEMIVOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl alcohol	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
bis(2-Chloroisopropyl) ether	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-dipropylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic acid	50	1600
bis(2-Chloroethoxy) methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol (para-chloro-meta-cresol)	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	50	1600
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1600
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
3-Nitroaniline	50	1600
Acenaphthene	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	50	1600
4,6-Dinitro-2-methylphenol	50	1600
N-nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

NOTE:

! THE SAMPLE-SPECIFIC CRQLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER TH

! MEDIUM LEVEL SOIL CRQL = 1980 x AQUEOUS CRQL REPORTED IN UG/KG.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
FEBRUARY, 1988

TARGET COMPOUND LIST - PESTICIDES/PCBs

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
alpha-BHC	0.05	8.0
beta-BHC	0.05	8.0
delta-BHC	0.05	8.0
gamma-BHC(Lindane)	0.05	8.0
Heptachlor	0.05	8.0
Aldrin	0.05	8.0
Heptachlor epoxide	0.05	8.0
Endosulfan I	0.05	8.0
Dieldrin	0.10	16.0
4,4'-DDE	0.10	16.0
Endrin	0.10	16.0
Endosulfan II	0.10	16.0
4,4'-DDD	0.10	16.0
Endosulfan sulfate	0.10	16.0

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
4,4'-DDT	0.10	16.0
Methoxychlor	0.5	80.0
Endrin ketone	0.10	16.0
alpha-Chlordane	0.5	80.0
gamma-Chlordane	0.5	80.0
Toxaphene	1.0	160.0
Aroclor-1016	0.5	80.0
Aroclor-1221	0.5	80.0
Aroclor-1232	0.5	80.0
Aroclor-1242	0.5	80.0
Aroclor-1248	0.5	80.0
Aroclor-1254	1.0	160.0
Aroclor-1260	1.0	160.0

NOTE:

! THE SAMPLE-SPECIFIC CRQLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER T
ABOVE.

! MEDIUM LEVEL SOIL CRQL = 15 x LOW LEVEL SOIL CRQL REPORTED IN UG/KG.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	OLM01.0
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	September 28, 1990 through February 1994
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment *

SIGNIFICANT FEATURES

- **!** The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- **!** Volatiles and semivolatiles are analyzed by GC/MS, pesticides/PCBs are analyzed by GC/ECD.
- **!** Major Tentatively Identified Compounds (TICs) are reported for GC/MS analyses.
- **!** Second column confirmation by GC/ECD is required for all pesticides/PCBs. Pesticides/PCBs which are identified by GC/EC above 10 ng/uL are confirmed by GC/MS analysis.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the 2/88 SOW that are incorporated in the OLM01.0 SOW:

- **!** Selected volatile CRQLs have been raised; pesticide/PCB low soil CRQLs have been lowered; and selected pesticide/PCB aqu changed.
- **!** Target Compound List (TCL) changes include the elimination of vinyl acetate from the volatile TCL; the elimination of benzy acid from the semivolatile TCL; the addition of carbazole to the semivolatile TCL; and the addition of endrin aldehyde to the p semivolatile TCL compound bis(2-chloroisopropyl)ether was renamed 2,2'-oxybis(1-chloropropane).
- **!** A new method for analysis of pesticides/PCBs is used. Changes include the use of wide bore capillary columns; new surrogate techniques.
- **!** Pesticide/PCB quantitation is performed using both the primary and secondary columns. The lower value is reported by the lal

The only significant change in the OLM01.1 (December, 1990) and OLM01.1.1 (February, 1991) revisions to the OLM01.(lowering of selected semivolatile CRQLs. The significant changes in the OLM01.1 through OLM01.7 revisions to the OLM01.0 S(selected semivolatile CRQLs and options for either a 14 day or 35 day data turnaround.

RECOMMENDED USES

This Routine Analytical Services (RAS) method is recommended for broad spectrum analysis to define the nature and extent contamination during SSI, LSI, and RI/FS activities. This method is suitable when a fourteen day or thirty five day turnaround for r recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at signific

* Sediment samples with high moisture content should be solicited as RAS + SAS (Special Analytical Service) in order to ac

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Attachment 1

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM01.0

TARGET COMPOUND LIST - VOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Chloromethane	10	10
Bromomethane	10	10
Vinyl Chloride	10	10
Chloroethane	10	10
Methylene Chloride	10*	10*
Acetone	10	10
Carbon Disulfide	10*	10*
1,1-Dichloroethene	10*	10*
1,1-Dichloroethane	10*	10*
1,2-Dichloroethene (total)	10*	10*
Chloroform	10*	10*
1,2-Dichloroethane	10*	10*
2-Butanone	10	10
1,1,1-Trichloroethane	10*	10*
Carbon Tetrachloride	10*	10*
Bromodichloromethane	10*	10*
1,2-Dichloropropane	10*	10*

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
cis-1,3-Dichloropropene	10*	10*
Trichloroethene	10*	10*
Dibromochloromethane	10*	10*
1,1,2-Trichloroethane	10*	10*
Benzene	10*	10*
trans-1,3-Dichloropropene	10*	10*
Bromoform	10*	10*
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	10*	10*
Toluene	10*	10*
1,1,2,2-Tetrachloroethene	10*	10*
Chlorobenzene	10*	10*
Ethyl Benzene	10*	10*
Styrene	10*	10*
Xylenes (total)	10*	10*

* CRQLs previously 5 ug/L and 5 ug/Kg in 2/88 SOW.

NOTE:

! THE SAMPLE-SPECIFIC CRQLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER TH
! MEDIUM LEVEL SOIL CRQL = 120 x AQUEOUS CRQL REPORTED IN UG/KG.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM01.0

TARGET COMPOUND LIST - SEMIVOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
2,2'-oxybis(1-Chloropropane)	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-dipropylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
bis(2-Chloroethoxy) methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	25*	800*
2-Chloronaphthalene	10	330
2-Nitroaniline	25*	800*
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	25*	800*

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Acenaphthene	10	330
2,4-Dinitrophenol	25*	800*
4-Nitrophenol	25*	800*
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	25*	800*
4,6-Dinitro-2-methylphenol	25*	800*
N-nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	25*	800*
Phenanthrene	10	330
Anthracene	10	330
Carbazole	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	10**	330**
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

* CRQLs previously 50 ug/L and 1600 ug/Kg in 2/88 SOW ** CRQLs previously 20 ug/L and 660 ug/Kg in 2/88 SOW.

NOTE:

! THE SAMPLE-SPECIFIC CRQLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER THAN THOSE LISTED ABOVE.

! MEDIUM LEVEL SOIL CRQL = 1000 x AQUEOUS CRQL REPORTED IN UG/KG.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM01.0

TARGET COMPOUND LIST - PESTICIDES/PCBs

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil** CRQL (ug/Kg,ppb)
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
gamma-BHC (Lindane)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil** CRQL (ug/Kg,ppb)
4,4'-DDT	0.10	3.3
Methoxychlor	0.5	17.0
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.05*	1.7
gamma-Chlordane	0.05*	1.7
Toxaphene	5.0*	170.0
Aroclor-1016	1.0*	33.0
Aroclor-1221	2.0*	67.0
Aroclor-1232	1.0*	33.0
Aroclor-1242	1.0*	33.0
Aroclor-1248	1.0*	33.0
Aroclor-1254	1.0	33.0
Aroclor-1260	1.0	33.0

Aqueous CRQLs changed from 2/88 SOW to the following:

* Aqueous CRQLs (ug/L) - alpha- and gamma- Chlordane from 0.5 to 0.05;
Toxaphene from 1.0 to 5.0;
Aroclors-1016, 1232, 1242, and 1248 from 0.5 to 1.0;
Aroclor-1221 from 0.5 to 2.0.

All low soil CRQLs changed from 2/88 SOW to the following:

** Low Soil CRQLs (ug/Kg) -alpha-BHC through Endosulfan I from 8.0 to 1.7;
Dieldrin through 4,4'-DDT and Endrin ketone from 16.0 to 3.3;
Methoxychlor from 80.0 to 17.0;
alpha- and gamma-Chlordane from 80.0 to 1.7;
Toxaphene from 160.0 to 170.0;
Aroclor-1016, 1232, 1242, and 1248 from 80.0 to 33.0;
Aroclor-1221 from 80.0 to 67.0;
Aroclor-1254 and 1260 from 160.0 to 33.0.

NOTE**:**

! THE SAMPLE-SPECIFIC CRQLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER ABOVE.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	OLM01.9
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	July 1993 through February 1995
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- Volatiles and semivolatiles are analyzed by GC/MS, pesticides/PCBs are analyzed by GC/ECD.
- Major Tentatively Identified Compounds (TICs) are reported for GC/MS analyses.
- Second column confirmation by GC/ECD is required for all pesticides/PCBs. The lower of the two concentrations detected on both columns is reported. Pesticides/PCBs which are detected at concentrations above 10 ng/uL are confirmed by GC/M

REVISIONS/MODIFICATIONS

The only significant revisions to the OLM01.0 SOW in the OLM01.8 revision were changes in the format and content of the Agency Standard diskette deliverable.

The following is a list of the significant changes from the OLM01.8 revision that are incorporated in the OLM01.9 rev

- MS/MSD analysis is not required for SDGs containing only equipment/trip blanks or PE samples.
- Specific instructions are given regarding resolution of problems with reduced sample volume and MS/MSD sample

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent of potential site contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remedial Design/Remedial Action (RD/RA) activities comply with pre-determined clean-up standards. This method is suitable when a 14 day or 35 day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* This method is not applicable to sediment samples with high moisture content.

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Attacl

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM01.9

TARGET COMPOUND LIST - VOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Chloromethane	10	10
Bromomethane	10	10
Vinyl chloride	10	10
Chloroethane	10	10
Methylene chloride	10	10
Acetone	10	10
Carbon disulfide	10	10
1,1-Dichloroethene	10	10
1,1-Dichloroethane	10	10
1,2-Dichloroethene (total)	10	10
Chloroform	10	10
1,2-Dichloroethane	10	10
2-Butanone	10	10
1,1,1-Trichloroethane	10	10
Carbon tetrachloride	10	10
Bromodichloromethane	10	10
1,2-Dichloropropane	10	10

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg/ppb)
cis-1,3-Dichloropropene	10	10
Trichloroethene	10	10
Dibromochloromethane	10	10
1,1,2-Trichloroethane	10	10
Benzene	10	10
trans-1,3-Dichloropropene	10	10
Bromoform	10	10
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	10	10
1,1,2,2-Tetrachloroethane	10	10
Toluene	10	10
Chlorobenzene	10	10
Ethylbenzene	10	10
Styrene	10	10
Xylenes (Total)	10	10

NOTE:

! The sample-specific CRQLs for soil samples will be adjusted for percent moisture and will be higher than those listed above.

! Medium level soil CRQL = 120 x aqueous CRQL reported in ug/kg.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM01.9

TARGET COMPOUND LIST - SEMIVOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Phenol	10	330
bis(2-chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
2,2'-oxybis(1-chloropropane)	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
bis-(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	25	800
2-Chloronaphthalene	10	330
2-Nitroaniline	25	800
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	25	800

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Acenaphthene	10	330
2,4-Dinitrophenol	25	800
4-Nitrophenol	25	800
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenylether	10	330
Fluorene	10	330
4-Nitroaniline	25	800
4,6-Dinitro-2-methylphenol	25	800
N-Nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	25	800
Phenanthrene	10	330
Anthracene	10	330
Carbazole	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	10	330
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenzo(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

NOTE:

! The sample-specific CRQLs for soil samples will be adjusted for percent moisture and will be higher than those listed above.

! Medium level soil CRQL = 1000 x aqueous CRQL in ug/Kg.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM01.9

TARGET COMPOUND LIST - PESTICIDE/PCB

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
gamma-BHC(Lindane)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor Epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/L,ppb)
4,4'-DDT	0.10	3.3
Methoxychlor	0.5	17.0
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.05	1.7
gamma-Chlordane	0.05	1.7
Toxaphene	5.0	170.0
Aroclor-1016	1.0	33.0
Aroclor-1221	2.0	67.0
Aroclor-1232	1.0	33.0
Aroclor-1242	1.0	33.0
Aroclor-1248	1.0	33.0
Aroclor-1254	1.0	33.0
Aroclor-1260	1.0	33.0

NOTE:

! The sample-specific CRQLs for soil samples will be adjusted for percent moisture and will be higher than those listed above.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	OLM02.1
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	No contracts have been awarded
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- Volatiles and semivolatiles are analyzed by GC/MS, pesticides/PCBs are analyzed by GC/ECD.
- Major Tentatively Identified Compounds (TICs) are reported for GC/MS analyses.
- Second column confirmation by GC/ECD is required for all pesticides/PCBs. The lower of the two concentrations detected on both columns is reported. Pesticides/PCBs which are detected at concentrations above 10 ng/uL are confirmed by GC/MS analysis.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the OLM01.9 SOW that are incorporated in the OLM02.0 SOW (including revision (

- For volatiles analysis, if gaseous compounds 1) fail to exhibit narrow symmetrical peak shape, 2) are not separated from the solvent front, or 3) are not resolved greater than 90% from each other; then a subambient oven controller must be used and the initial temperature must be $\leq 10^{o}C$.
- Background subtraction must be performed utilizing a spectrum obtained no greater than 20 scans prior to the elution of BFB or DFTPP.
- The final column temperature must be held for three minutes following the elution of the last BNA target compound.
- Samples which contain alkane series in the TICs will be evaluated using the mass chromatograms of m/z 43, 57, and 71 and the alkane series will be reported as one TIC along with a total estimated concentration.
- Within 72 hours of detecting a multi-component pesticide/PCB in a field sample, a standard must be analyzed.
- The number of TICs for volatiles have been raised from 10 to 30 and for semivolatiles have been raised from 20 to 30.

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent of potential site contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remedial Design/Remedial Action (RD/RA) activities comply with the pre-determined clean-up standards. This method is suitable when a 14 day or 35 day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* This method is not applicable to sediment samples with high moisture content.

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Attachment 1.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM02.1

TARGET COMPOUND LIST - VOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Chloromethane	10	10
Bromomethane	10	10
Vinyl chloride	10	10
Chloroethane	10	10
Methylene chloride	10	10
Acetone	10	10
Carbon disulfide	10	10
1,1-Dichloroethene	10	10
1,1-Dichloroethane	10	10
1,2-Dichloroethene (total)	10	10
Chloroform	10	10
1,2-Dichloroethane	10	10
2-Butanone	10	10
1,1,1-Trichloroethane	10	10
Carbon tetrachloride	10	10
Bromodichloromethane	10	10
1,2-Dichloropropane	10	10

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg/ppb)
cis-1,3-Dichloropropene	10	10
Trichloroethene	10	10
Dibromochloromethane	10	10
1,1,2-Trichloroethane	10	10
Benzene	10	10
trans-1,3-Dichloropropene	10	10
Bromoform	10	10
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	10	10
1,1,2,2-Tetrachloroethane	10	10
Toluene	10	10
Chlorobenzene	10	10
Ethylbenzene	10	10
Styrene	10	10
Xylenes (Total)	10	10

NOTE:

! The sample-specific CRQLs for soil samples will be adjusted for percent moisture and will be higher than those listed above.

! Medium level soil CRQL = 120 x aqueous CRQL reported in ug/kg.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM02.1

TARGET COMPOUND LIST - SEMIVOLATILES

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Phenol	10	330
bis(2-chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
2,2'-oxybis(1-chloropropane)	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
bis-(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	25	800
2-Chloronaphthalene	10	330
2-Nitroaniline	25	800
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	25	800

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
Acenaphthene	10	330
2,4-Dinitrophenol	25	800
4-Nitrophenol	25	800
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenylether	10	330
Fluorene	10	330
4-Nitroaniline	25	800
4,6-Dinitro-2-methylphenol	25	800
N-Nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	25	800
Phenanthrene	10	330
Anthracene	10	330
Carbazole	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	10	330
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenzo(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

NOTE:

! The sample-specific CRQLs for soil samples will be adjusted for percent moisture and will be higher than those listed above.

! Medium level soil CRQL = 1000 x aqueous CRQL in ug/Kg.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM02.1

TARGET COMPOUND LIST - PESTICIDE/PCB

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/Kg,ppb)
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
gamma-BHC(Lindane)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor Epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3

Compound	Aqueous CRQL (ug/L,ppb)	Low Soil CRQL (ug/L,ppb)
4,4'-DDT	0.10	3.3
Methoxychlor	0.5	17.0
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.05	1.7
gamma-Chlordane	0.05	1.7
Toxaphene	5.0	170.0
Aroclor-1016	1.0	33.0
Aroclor-1221	2.0	67.0
Aroclor-1232	1.0	33.0
Aroclor-1242	1.0	33.0
Aroclor-1248	1.0	33.0
Aroclor-1254	1.0	33.0
Aroclor-1260	1.0	33.0

NOTE:

! The sample-specific CRQLs for soil samples will be adjusted for percent moisture and will be higher than those listed above.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, HIGH CONCENTRATION

DOCUMENT NUMBER:	Not Applicable
DOCUMENT DATE:	September 1988
EFFECTIVE DATES:	June 7, 1989 through December 26, 1991
CONCENTRATION:	High: Greater than 20 ppm
DATA TURNAROUND:	35 Days
MATRICES:	Liquid/Solid/Multi-phase

SIGNIFICANT FEATURES

- No holding times are designated for high concentration samples.
- The analyses are suitable for highly contaminated samples (>20 mg/Kg).
- The analyses are acceptable for liquid, solid, or multiphase samples. Multi-phase samples are separated into water miscible liquid, water immiscible liquid, or solid phases. Each phase is analyzed separately.
- Volatile, extractable (semivolatiles and pesticides), and multicomponent extractable (Aroclors and Toxaphene) co
- Volatiles and extractables are analyzed by GC/MS; Aroclors and Toxaphene are analyzed by GC/ECD.
- Second column confirmation by GC/ECD is required for Aroclors and Toxaphene.
- Major Tentatively Identified Compounds (TICs) are reported for GC/MS analyses.

REVISIONS/MODIFICATIONS

The 1/89 and 4/89 revisions to the 9/88 SOW do not significantly affect data useability.

RECOMMENDED USES

This Routine Analytical Services (RAS) method is recommended for pre-remedial, remedial, or removal projects where high concentrations of organic contaminants (greater than 20 mg/Kg) are suspected and a thirty five day turnaround for results is adequate. It is recommended for samples obtained from drummed material, waste pits or lagoons, piles of waste, tanker trucks, onsite tanks, or apparent contaminated soil areas. The waste material may be industrial process waste, byproducts, raw materials, intermediates and contaminated products. Samples may be spent oil, spent solvents, paint wastes, metal treatment wastes, and polymer formulations.

The method is suitable for solids, liquids, or multiphase samples, a phase being either water miscible liquid, water immiscible liquid, or solid. Various methods of phase separation may be utilized depending on the number and types of phases in a sample

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in

TARGET COMPOUND LIST - VOLATILES

Compound	CRQL (mg/Kg,ppm)
Chloromethane	5.0
Bromomethane	5.0
Vinyl Chloride	5.0
Chloroethane	5.0
Methylene Chloride	2.5
Acetone	5.0
Carbon Disulfide	2.5
1,1-Dichloroethene	2.5
1,1-Dichloroethane	2.5
1,2-Dichloroethene (total)	2.5
Chloroform	2.5
1,2-Dichloroethane	2.5
2-Butanone	5.0
1,1,1-Trichloroethane	2.5
Carbon Tetrachloride	2.5
Vinyl Acetate	5.0
Bromodichloromethane	2.5

Compound	CRQL (mg/Kg,ppm)
1,2-Dichloropropane	2.5
cis-1,3-Dichloropropene	2.5
Trichloroethene	2.5
Dibromochloromethane	2.5
1,1,2-Trichloroethane	2.5
Benzene	2.5
trans-1,3-Dichloropropene	2.5
Bromoform	2.5
4-Methyl-2-pentanone	5.0
2-Hexanone	5.0
Tetrachloroethene	2.5
1,1,2,2-Tetrachloroethane	2.5
Toluene	2.5
Chlorobenzene	2.5
Ethylbenzene	2.5
Styrene	2.5
Xylene (Total)	2.5

NOTE:

! ALL CRQLs ARE BASED ON WET WEIGHT AND APPLY TO SOLID AND LIQUID SAMPLES.
! RESULTS FOR BOTH SOLID AND LIQUID SAMPLES ARE REPORTED AS MG/KG, WET WEIGHT.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, HIGH CONCENTRATION
SEPTEMBER, 1988

TARGET COMPOUND LIST - EXTRACTABLES

Compound	CRQL (mg/Kg,ppm)
Phenol	20
bis(2-Chloroethyl)ether	20
2-Chlorophenol	20
1,3-Dichlorobenzene	20
1,4-Dichlorobenzene	20
Benzyl alcohol	20
1,2-Dichlorobenzene	20
2-Methylphenol	20
bis(2-Chloroisopropyl) ether	20
4-Methylphenol	20
N-Nitroso-di-n-dipropylamine	20
Hexachloroethane	20
Nitrobenzene	20
Isophorone	20
2-Nitrophenol	20
2,4-Dimethylphenol	20
Benzoic acid	100
bis(2-Chloroethoxy) methane	20
2,4-Dichlorophenol	20
1,2,4-Trichlorobenzene	20
Naphthalene	20
4-Chloroaniline	20
Hexachlorobutadiene	20
4-Chloro-3-methylphenol (para-chloro-meta-cresol)	20
2-Methylnaphthalene	20
Hexachlorocyclopentadiene	20
2,4,6-Trichlorophenol	20
2,4,5-Trichlorophenol	100
2-Chloronaphthalene	20
2-Nitroaniline	100
Dimethylphthalate	20

Compound	CRQL (mg/Kg,ppm)
Acenaphthylene	20
2,6-Dinitrotoluene	20
3-Nitroaniline	100
Acenaphthene	20
2,4-Dinitrophenol	100
4-Nitrophenol	100
Dibenzofuran	20
2,4-Dinitrotoluene	20
Diethylphthalate	20
4-Chlorophenyl-phenylether	20
Fluorene	20
4-Nitroaniline	100
4,6-Dinitro-2-methylphenol	100
N-nitrosodiphenylamine	20
4-Bromophenyl-phenylether	20
alpha-BHC	20
Hexachlorobenzene	20
beta-BHC	20
Pentachlorophenol	100
gamma-BHC (Lindane)	20
Phenanthrene	20
Anthracene	20
delta-BHC	20
Heptachlor	20
Aldrin	20
Di-n-butylphthalate	20
Fluoranthene	20
Heptachlor epoxide	20
Monochlorobiphenyl	100
Dichlorobiphenyl	100
Trichlorobiphenyl	100

NOTE:

- ALL CRQLs ARE BASED ON WET WEIGHT AND APPLY TO SOLID AND LIQUID SAMPLES.
- RESULTS FOR BOTH SOLID AND LIQUID SAMPLES ARE REPORTED AS MG/KG, WET WEIGHT.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, HIGH CONCENTRATION
SEPTEMBER, 1988

TARGET COMPOUND LIST - EXTRACTABLES (CONTINUED)

Compound	CRQL (mg/Kg,ppm)
Tetrachlorobiphenyl	100
Pyrene	20
gamma-Chlordane	20
Endosulfan I	20
alpha-Chlordane	20
4,4,'-DDE	20
Dieldrin	20
Hexachlorobiphenyl	100
Pentachlorobiphenyl	100
Endrin	20
Endosulfan II	20
4,4'-DDD	20
Heptachlorobiphenyl	100
Butylbenzylphthalate	20
Endosulfan sulfate	20
4,4'-DDT	20

Compound	CRQL (mg/Kg,ppm)
Endrin ketone	20
Benzo(a)anthracene	20
Methoxychlor	20
Chrysene	20
Octachlorobiphenyl	200
3,3'-Dichlorobenzidine	40
bis(2-Ethylhexyl)phthalate	20
Nonachlorobiphenyl	200
Decachlorobiphenyl	200
Di-n-octylphthalate	20
Benzo(b)fluoranthene	20
Benzo(k)fluoranthene	20
Benzo(a)pyrene	20
Indeno(1,2,3-cd)pyrene	20
Dibenz(a,h)anthracene	20
Benzo(g,h,i)perylene	20

TARGET COMPOUND LIST - MULTICOMPONENT EXTRACTABLES

Compound	CRQL (mg/Kg,ppm)
Toxaphene	50
Aroclor 1016	10
Aroclor 1221	10
Aroclor 1232	10
Aroclor 1242	10
Aroclor 1248	10
Aroclor 1254	10
Aroclor 1260	10

NOTE:

- ALL CRQLs ARE BASED ON WET WEIGHT AND APPLY TO SOLID AND LIQUID SAMPLES.
- RESULTS FOR BOTH SOLID AND LIQUID SAMPLES ARE REPORTED AS MG/KG, WET WEIGHT.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	Not Applicable
DOCUMENT DATE:	July 1988
EFFECTIVE DATES:	May 23, 1989 through March 29, 1992
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	35 Days
MATRICES:	Aqueous/Soil/Sediment *

SIGNIFICANT FEATURES

! The analyses are suitable for aqueous, soil or sediment samples at low to medium concentration levels.

REVISIONS/MODIFICATIONS

2/89 - Method for Total Cyanide (CN) Analysis by Midi Distillation Method 335.2 CLP-M was added.

6/89 - Revisions to the 7/88 SOW do not significantly affect data useability.

RECOMMENDED USES

This Routine Analytical Services (RAS) method is recommended for broad spectrum analysis to define the nature and extent of potential site contamination during SSI, LSI, and RI/FS activities. This method is suitable when a thirty five day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* Sediment samples with high moisture content should be solicited as RAS + SAS (Special Analytical Service) in order to achieve the CRDLs.

ANALYTES/CRDLs

The parameters included in the analysis and the Contract Required Detection Limits (CRDLs) are listed in Attachm

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
JULY, 1988

TARGET ANALYTE LIST

Analyte	AQUEOUS CRDL (ug/L,ppb)	SOIL CRDL (mg/Kg,ppm)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6

Analyte	AQUEOUS CRDL (ug/L,ppb)	SOIL CRDL (mg/Kg,ppm)
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2

NOTE:

! THE SAMPLE-SPECIFIC CRDLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER THAN THOSE LISTED ABOVE.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	ILM01.0
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	September 7, 1990 through September 26, 1993
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	35 Days
MATRICES:	Aqueous/Soil/Sediment *

SIGNIFICANT FEATURES

- The analyses are suitable for aqueous, soil or sediment samples at low to medium concentration levels.
- This Statement of Work includes the midi distillation for cyanide analysis and the microwave digestion for GFAA and ICP analyses. These two sample preparation procedures require less sample volume than the traditional Statement of Work sample preparation procedures.

REVISIONS/MODIFICATIONS

None to Date

RECOMMENDED USES

This Routine Analytical Services (RAS) method is recommended for broad spectrum analysis to define the nature and extent of potential site contamination during SSI, LSI, and RI/FS activities. This method is suitable when a thirty five day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* Sediment samples with high moisture content should be solicited as RAS + SAS (Special Analytical Service) in order to achieve the CRDLs.

ANALYTES/CRDLs

The parameters included in the analysis and the Contract Required Detection Limits (CRDLs) are listed in Att

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
ILM01.0

TARGET ANALYTE LIST

Analyte	Aqueous CRDL (ug/L,ppb)	Soil CRDL (mg/Kg,ppm)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6

Analyte	Aqueous CRDL (ug/L,ppb)	Soil CRDL (mg/Kg,ppm)
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2

NOTE:

! THE SAMPLE-SPECIFIC CRDLs FOR SOIL SAMPLES WILL BE ADJUSTED FOR PERCENT MOISTURE AND WILL BE HIGHER THAN THOSE LISTED ABOVE.

TITLE: **USEPA CONTRACT LABORATORY PROGRAM**
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	ILM02.1
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	March 1993 through October 1994
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- The analyses are suitable for aqueous, soil, or sediment samples at low to medium concentration levels.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the ILM01.0 SOW that are incorporated in the ILM02.1 SOW:

- Specific analysis instructions are presented when samples are received for dissolved metals analysis.
- Requirements for contract reports/deliverables distribution are included for 14 day turnaround contracts.
- SOPs are now required to be distributed by the laboratories to EPA-NEIC.
- Microwave digestion for soil/sediment samples is not appropriate for quantitative recovery of antimony.

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent of potential site contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remedial Design/Remedial Action (RD/RA) activities comply with pre-determined clean-up standards. This method is suitable when a 14 day or 35 day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* This method is not applicable to sediment samples with high moisture content.

ANALYTES/CRDLs

The parameters included in the analysis and the Contract Required Detection Limits (CRDLs) are listed in Attachm

ATTACHMENT 1

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
ILM02.1

TARGET ANALYTE LIST

Analyte	Aqueous CRDL (ug/L,ppb)	Soil CRDL (mg/Kg,ppm)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6

Analyte	Aqueous CRDL (ug/L,ppb)	Soil CRDL (mg/Kg,ppm)
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2

NOTE:

! The sample-specific CRDLs for soil samples will be adjusted for percent moisture and will be higher than those listed a

TITLE: **USEPA CONTRACT LABORATORY PROGRAM**
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	ILM03.0
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	May 1993 through March 1996
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- The analyses are suitable for aqueous, soil, or sediment samples at low to medium concentration levels.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the ILM02.1 SOW that are incorporated in the ILM03.0 SOW:

- An analytical spike and an aqueous Laboratory Control Sample (LCS) are not required when analyzing field samples fo
- Terminology was added to require that sample coolers be returned to the appropriate sampling office within 14 days following shipment receipt.
- Additional instrumentation requirements were added for greater than 500 samples per month capacity.
- For cyanide water analysis, the LCS requirement was changed from "not required" to "using the distilled ICV as the LC

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent of potential site contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remedial Design/Remedial Action (RD/RA) activities comply with pre-determined clean-up standards. This method is suitable when a 14 day or 35 day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* This method is not applicable to sediment samples with high moisture content.

ANALYTES/CRDLs

The parameters included in the analysis and the Contract Required Detection Limits (CRDLs) are listed in Attachment 1

ATTACHMENT 1

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
ILM03.0

TARGET ANALYTE LIST

Analyte	Aqueous CRDL (ug/L,ppb)	Soil CRDL (mg/Kg,ppm)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6

Analyte	Aqueous CRDL (ug/L,ppb)	Soil CRDL (mg/Kg,ppm)
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2

NOTE:

! The sample-specific CRDLs for soil samples will be adjusted for percent moisture and will be higher than those listed a

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, HIGH CONCENTRATION

DOCUMENT NUMBER:	IHC01.2
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	May 15, 1991 through November 30, 1993
CONCENTRATION:	High
DATA TURNAROUND:	35 Days
MATRICES:	Liquid/Solid/Multi-phase

SIGNIFICANT FEATURES

- The analyses are suitable for highly contaminated samples.
- The analyses are acceptable for liquid, solid, or multiphase samples. Multi-phase samples are separated into water miscible liquid, water immiscible liquid, or solid phases. Each phase is analyzed separately.
- The analyses include conductivity and pH; potassium is not included.

REVISIONS/MODIFICATIONS

The IHC01.1 and IHC01.2 revisions to the IHC01.0 SOW do not significantly affect data useability.

RECOMMENDED USES

This Routine Analytical Services (RAS) method is recommended for pre-remedial, remedial, or removal projects where high concentrations of inorganic contaminants are suspected and a thirty five day turnaround for results is adequate. It is recommended for samples obtained from drummed material, waste pits or lagoons, piles of waste, tanker trucks, onsite tanks, or apparent contaminated soil areas. The waste material may be industrial process waste, byproducts, raw materials, intermediates and contaminated products. Samples may be spent oil, spent solvents, paint wastes, metal treatment wastes, and polymer formulations.

The method is suitable for solids, liquids, or multiphase samples, a phase being either water miscible liquid, water immiscible liquid, or solid. A phase separation step is applied prior to digestion. Each phase is analyzed and reported as a separat

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANIC ANALYSIS
MULTI-MEDIA, HIGH CONCENTRATION
IHC01.2

TARGET ANALYTE LIST

Analyte	CRQL (mg/Kg, ppm)	**Analyte**	CRQL (mg/Kg, ppm)
Aluminum	80	Manganese	10
Antimony	20	Mercury	0.3
Arsenic	5	Nickel	20
Barium	80	Selenium	5
Beryllium	5	Silver	10
Cadmium	10	Sodium	80
Calcium	80	Thallium	20
Chromium	10	Vanadium	20
Cobalt	20	Zinc	10
Copper	40	Cyanide	1.5
Iron	20	pH	NA
Lead	10	Conductivity	3.0 (µmhos/cm)
Magnesium	80		

NOTE:

! ALL CRQLs ARE BASED ON WET WEIGHT AND APPLY TO SOLID AND LIQUID SAMPLES.
! RESULTS FOR BOTH SOLID AND LIQUID SAMPLES ARE REPORTED AS MG/KG, WET WEIGHT.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ANALYSIS OF POLYCHLORINATED DIBENZO-p-DIOXINS (PCDD)
AND POLYCHLORINATED DIBENZOFURANS (PCDF)
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	DFLMO1.2
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	May 1992 through May 1994
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	45 days
MATRICES:	Aqueous/Soil/Fly Ash/Chemical Waste

SIGNIFICANT FEATURES

- The parameters include 2,3,7,8-substituted Tetra-, Penta-, Hexa-, Hepta-, and Octachlorinated dibenzo-p-dioxins (PCDDs) and diben
- Total homologue concentrations are reported for a given level of chlorination (i.e. Total TCDD, Total PCDF, etc.).
- 2,3,7,8-TCDD toxic equivalence is determined using all 2,3,7,8-substituted isomers.
- The dioxins and furans are analyzed by High Resolution Gas Chromatography and Low Resolution Mass Spectrometry (HRGC/LRM
- Second column confirmation is required if the toxic equivalence is greater than or equal to 0.7ppb (soil or fly ash), 7ppt (aqueous) or
- Chemical waste includes oils, stillbottoms, oily sludge, wet fuel oil, and surface water heavily contaminated with oils. Soil samples i

REVISIONS/MODIFICATIONS

Revisions DFLMO1.1 (September 1991) and DFLMO1.2 (April 1992) do not significantly affect data useability.

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for analysis of polychlorinated dioxins and furans to define the nat site contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remec (RD/RA) activities comply with pre-determined clean-up standards. This method is suitable when a forty five day turnaround for results recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant ri applicable to sediment samples with high moisture content.

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Table 1.

TABLE 1

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ANALYSIS OF POLYCHLORINATED
DIBENZO-p-DIOXINS (PCDD) AND POLYCHLORINATED DIBENZOFURANS (PCDF)
MULTI-MEDIA, MULTI-CONCENTRATION
DFLMO1.2

TARGET COMPOUND LIST

Compound	Aqueous CRQL (ng/L,ppt)	Low Soil CRQL[A] (ug/Kg,ppb)	Fly Ash CRQL[A] (ug/Kg,ppb)	Chemical Waste CRQL (ug/Kg,ppb)	TEFs[B]
2,3,7,8-TCDD	10	1.0	1.0	10	1.0
2,3,7,8-TCDF	10	1.0	1.0	10	0.10
1,2,3,7,8-PeCDF	25	2.5	2.5	25	0.05
1,2,3,7,8-PeCDD	25	2.5	2.5	25	0.50
2,3,4,7,8-PeCDF	25	2.5	2.5	25	0.50
1,2,3,4,7,8-HxCDF	25	2.5	2.5	25	0.10
1,2,3,6,7,8-HxCDF	25	2.5	2.5	25	0.10
1,2,3,4,7,8-HxCDD	25	2.5	2.5	25	0.10
1,2,3,6,7,8-HxCDD	25	2.5	2.5	25	0.10
1,2,3,7,8,9-HxCDD	25	2.5	2.5	25	0.10
2,3,4,6,7,8-HxCDF	25	2.5	2.5	25	0.10
1,2,3,7,8,9-HxCDF	25	2.5	2.5	25	0.10
1,2,3,4,6,7,8-HpCDF	25	2.5	2.5	25	0.01
1,2,3,4,6,7,8-HpCDD	25	2.5	2.5	25	0.01
1,2,3,4,7,8,9-HpCDF	25	2.5	2.5	25	0.01
OCDD	50	5.0	5.0	50	0.001
OCDF	50	5.0	5.0	50	0.001

Homologue Compounds	Number of Possible Isomers	Number of 2,3,7,8-Substituted Isomers
TOTAL TCDD	22	1
TOTAL TCDF	38	1
TOTAL PeCDD	14	1
TOTAL PeCDF	28	2
TOTAL HxCDD	10	3
TOTAL HxCDF	16	4
TOTAL HpCDD	2	1
TOTAL HpCDF	4	2
OCDD	1	1
OCDF	1	1

NOTE:

A. The sample-specific CRQLs for soils and fly ash samples will be adjusted for percent moisture and will be higher than those listed above

B. TEF = 2,3,7,8-TCDD Toxic Equivalence Factors (TEFs) for the PCDDs/PCDFs.

TITLE: SUPERFUND ANALYTICAL METHODS FOR LOW CONCENTRATION WATER FOR ORGANICS

DOCUMENT NUMBER:	Not Applicable
DOCUMENT DATE:	October 1992
EFFECTIVE DATES:	December 1992 through June 1994
CONCENTRATION:	Low
DATA TURNAROUND:	14 Days
MATRICES:	Aqueous

SIGNIFICANT FEATURES

- The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- Volatiles and semivolatiles are analyzed by GC/MS; pesticides/PCBs are analyzed by GC/ECD.
- All parameters require significantly reduced CRQLs as compared to the OLM01.9 SOW.
- A 25mL aliquot of sample is purged for volatiles analysis.
- Analysis of a Laboratory Control Sample (LCS) for each parameter is required.
- Semivolatile and pesticide/PCB samples must be extracted by continuous liquid-liquid extraction procedures.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the 6/91 Low Concentration SOW:

- The requirement for a diskette deliverable was removed.
- Technical acceptance criteria for the volatile LCS were established.
- Potential action against a laboratory for Performance Evaluation (PE) scores below 75% was added.

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and level organic* contamination in water supplies during Site Investigation (SI) and Remedial Investigation/Feasibility Study verify that Remedial Design/Remedial Action (RD/RA) activities comply with pre-determined clean-up standards. This m detection limits than SOW OLM01.9 and can aid in the determination of low level contamination in public drinking water samples are expected to be from drinking water and well/ground water sources around Superfund sites. This method is sui turnaround for results is adequate.

* This method should not be used for low concentration volatile organics analyses in Region I when comparison to the dr Contaminant Levels (MCLs) is required. The Region I EPA 524.2 standard specifications should be utilized for this pu

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Attachme

SUPERFUND ANALYTICAL METHODS FOR LOW CONCENTRATION
WATER FOR ORGANICS ANALYSIS
10/92

TARGET COMPOUND LIST - VOLATILES

COMPOUND	CRQL (ug/L, ppb)
Chloromethane	1
Bromomethane	1
Vinyl chloride	1
Chloroethane	1
Methylene chloride	2
Acetone	5
Carbon disulfide	1
1,1-Dichloroethene	1
1,1-Dichloroethane	1
cis-1,2-Dichloroethene	1
trans-1,2-Dichloroethene	1
Chloroform	1
1,2-Dichloroethane	1
2-Butanone	5
Bromochloromethane	1
1,1,1-Trichloroethane	1
Carbon tetrachloride	1
Bromodichloromethane	1
1,2-Dichloropropane	1
cis-1,3-Dichloropropene	1

COMPOUND	CRQL (ug/L, ppb)
Trichloroethene	1
Dibromochloromethane	1
1,1,2-Trichloroethane	1
Benzene	1
trans-1,3-Dichloropropene	1
Bromoform	1
4-Methyl-2-pentanone	5
2-Hexanone	5
Tetrachloroethene	1
1,1,2,2-Tetrachloroethane	1
1,2-Dibromoethane	1
Toluene	1
Chlorobenzene	1
Ethylbenzene	1
Styrene	1
Xylenes (Total)	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
1,2-Dichlorobenzene	1
1,2-Dibromo-3-chloropropane	1

SUPERFUND ANALYTICAL METHODS FOR LOW CONCENTRATION
WATER FOR ORGANICS ANALYSIS
10/92

TARGET COMPOUND LIST - SEMIVOLATILES

COMPOUND	CRQL (ug/L, ppb)
Phenol	5
bis-(2-chloroethyl)ether	5
2-Chlorophenol	5
2-Methylphenol	5
2,2'-oxybis(1-chloropropane)	5
4-Methylphenol	5
N-Nitroso-di-n-propylamine	5
Hexachloroethane	5
Nitrobenzene	5
Isophorone	5
2-Nitrophenol	5
2,4-Dimethylphenol	5
bis-(2-Chloroethoxy)methane	5
2,4-Dichlorophenol	5
1,2,4-Trichlorobenzene	5
Naphthalene	5
4-Chloroaniline	5
Hexachlorobutadiene	5
4-Chloro-3-methylphenol	5
2-Methylnaphthalene	5
Hexachlorocyclopentadiene	5
2,4,6-Trichlorophenol	5
2,4,5-Trichlorophenol	20
2-Chloronaphthalene	5
2-Nitroaniline	20
Dimethylphthalate	5
Acenaphthylene	5
2,6-Dinitrotoluene	5
3-Nitroaniline	20
Acenaphthene	5

COMPOUND	CRQL (ug/L, ppb)
2,4-Dinitrophenol	20
4-Nitrophenol	20
Dibenzofuran	5
2,4-Dinitrotoluene	5
Diethylphthalate	5
4-Chlorophenyl-phenylether	5
Fluorene	5
4-Nitroaniline	20
4,6-Dinitro-2-methylphenol	20
N-Nitrosodipenylamine	5
4-Bromophenyl-phenylether	5
Hexachlorobenzene	5
Pentachlorophenol	20
Phenanthrene	5
Anthracene	5
Di-n-butylphthalate	5
Fluoranthene	5
Pyrene	5
Butylbenzylphthalate	5
3,3'-Dichlorobenzidine	5
Benzo(a)anthracene	5
Chrysene	5
bis(2-ethylhexyl)phthalate	5
Di-n-octylphthalate	5
Benzo(b)fluoranthene	5
Benzo(k)fluoranthene	5
Benzo(a)pyrene	5
Indeno(1,2,3-cd)pyrene	5
Dibenzo(a,h)anthracene	5
Benzo(g,h,i)perylene	5

SUPERFUND ANALYTICAL METHODS FOR LOW CONCENTRATION
WATER FOR ORGANICS ANALYSIS
10/92

TARGET COMPOUND LIST - PESTICIDE/PCB

PARAMETER	CRQL (ug/L, ppb)
alpha-BHC	0.01
beta-BHC	0.01
delta-BHC	0.01
gamma-BHC (Lindane)	0.01
Heptachlor	0.01
Aldrin	0.01
Heptachlor epoxide	0.01
Endosulfan I	0.01
Dieldrin	0.02
4,4'-DDE	0.02
Endrin	0.02
Endosulfan II	0.02
4,4'-DDD	0.02
Endosulfan sulfate	0.02

PARAMETER	CRQL (ug/L, ppb)
4,4'-DDT	0.02
Methoxychlor	0.10
Endrin ketone	0.02
Endrin aldehyde	0.02
alpha-Chlordane	0.01
gama-Chlordane	0.01
Toxaphene	1.0
Aroclor-1016	0.2
Aroclor-1221	0.4
Aroclor-1232	0.2
Aroclor-1242	0.2
Aroclor-1248	0.2
Aroclor-1254	0.2
Aroclor-1260	0.2

TITLE: **USEPA CONTRACT LABORATORY PROGRAM**
WATER QUALITY PARAMETERS (WQP) IN MULTI-CONCENTRATION WATER

DOCUMENT NUMBER:	Not Applicable
DOCUMENT DATE:	June 1993
EFFECTIVE DATES:	July 1993 through June 1994
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous

SIGNIFICANT FEATURES

- The parameters include Alkalinity, Ammonia-Nitrogen, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), Chloride, Nitrate/Nitrite, Total Phosphorous, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), and Sulfate.
- Ion chromatography may be used in place of conventional methods for the determination of Chloride, Nitrate/Nitrite, Phosphorous, and Sulfate.
- The laboratory IDL for a parameter may exceed the CRDL if the sample concentrations are greater than 5xIDL.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the 2/93 version that are incorporated in the 6/93 SOW:

- The CRDL for Total Dissolved Solids was elevated from 4,000 ug/L to 10,000 ug/L.
- The analysis procedure for Total Organic Carbon (TOC) was changed from following a step-by-step procedure for calibrating and standardizing the TOC analyzer to performing these functions according to the manufacturers specifications.

RECOMMENDED USES

These Contract Laboratory Program (CLP) methods are intended for use only with aqueous samples. They are recommended for analysis of selected water quality parameters to define the nature and extent of potential site contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remedial Design/Remedial Action (RD/RA) activities comply with pre-determined clean-up standards. These methods may also be used for monitoring the wastewater treatment processes of pretreatment plants and Publicly Owned Treatment Works (POTWs). This method is suitable when a 14 day or 35 day turnaround for results is adequate. It is recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

ANALYTES/CRDLs

The parameters included in the analysis and the Contract Required Detection Limits (CRDLs) are listed in Attachment

ATTACHMENT 1

USEPA CONTRACT LABORATORY PROGRAM
WATER QUALITY PARAMETERS (WQP) IN MULTI-CONCENTRATION WATER
6/93

PARAMETER LIST - WATER QUALITY PARAMETERS

PARAMETER	**CRDL (ug/L, ppb)**
Alkalinity	2000
Ammonia-Nitrogen	1000
Total Organic Carbon (TOC)	100
Chemical Oxygen Demand (COD)	3000
Chloride	2000
Nitrate/Nitrite	100
Total Phosphorous	100
Total Dissolved Solids (TDS)	10000
Total Suspended Solids (TSS)	4000
Sulfate	2000

TITLE: **USEPA CONTRACT LABORATORY PROGRAM**
STATEMENT OF WORK FOR INORGANICS ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	ILM04.0
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	August 1995 through May 1999
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- Analyses are suitable for aqueous, soil or sediment samples at low to medium concentration levels.
- Metals except mercury are analyzed by furnace AA, flame AA, and/or ICP-AES; mercury by cold vapor AA; and cyanide by spe distillation.

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent of p during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Remedial Design/Rem activities comply with pre-determined clean-up standards. This method is suitable when a 14 day or 35 day turnaround for results is a for samples from known or suspected hazardous waste sites where potential contamination may be present at significant risk levels.

* This method is not applicable to soil/sediment samples with high moisture content.

REVISIONS/MODIFICATIONS

The following is a list of significant changes from the SOW ILM03.0 that are incorporated in the SOW ILM04.0:

- New procedure: The lab must measure the sample shipping cooler temperature at time of sample receipt using the cooler temperat present, located in the cooler. The lab must contact CLASS if the cooler's temperature exceeds 10°C.
- If dissolved metals are required by the EPA Regional offices and there are no instructions on the Traffic Report, the lab must dige dissolved metals.
- If elements (e.g., As, Pb, Se, Tl) traditionally analyzed by GFAA are analyzed by ICP, the spiking levels for Furnace AA must be are ≤CRDL).
- Additional analysis frequency requirement for ICP CRDL and ICS standards: also analyzed at a frequency of not greater than 20 a analysis run.
- For analytes with CRDLs ≤10 µg/L, ICP ICSA results must be within ±2x CRDL of the true value; otherwise, these analytes must alternate method (e.g., GFAA) for samples analyzed since the last compliant ICSA.
- For analytes with CRDLs ≤10 µg/L, the ICP ICSA results must be reported from an undiluted sample analysis.
- Independent ICP Interference Check Samples: Elements As, Sb, Se, Tl were added (0.1, 0.6, 0.05, and 0.1 mg/L, respectively); Ag lowered (0.2 and 0.05 mg/L, respectively).
- Alternate methods for catastrophic ICP failure are no longer included.
- Cyanide spiking concentration for aqueous and soil matrix spikes: 100 µg/L in the distillate (i.e., the final sample solution prepare the amount of sample used.
- Mercury - clarification of the CRDL standard requirement in the calibration curve for manual cold vapor AA; a linear regression e automated cold vapor AA analysis.

ANALYTES/CRDLS

The parameters included in the analysis and the Contract Required Detection Limits (CRDLs) are listed in Attachment I.

ATTACHMENT I

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR INORGANICS ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
ILM04.0

TARGET ANALYTE LIST

Analyte	Aqueous CRDL (ug/L, ppb)	Soil CRDL (mg/kg, ppm)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6

Analyte	Aqueous CRDL (ug/L, ppb)	Soil CRDL (mg/kg, ppm)
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2.5

NOTE:

! The CRDLs for soils are based on the following: for all metals (except mercury) - 1 gram sample, 200 mL final digestate volume mercury - 0.2 gram sample, 100 mL final sample solution volume, 100% solids; and for cyanide - 1 gram sample, 250 mL final d distillate taken for manual spectrophotometric determination, and 100% solids.

! The sample-specific CRDLs for soil/sediment samples will be adjusted for percent solids and will be higher than those listed abov

TITLE: **USEPA CONTRACT LABORATORY PROGRAM**
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION

DOCUMENT NUMBER:	OLM03.2
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	July 1994 through February 1999
CONCENTRATION:	Low to Medium
DATA TURNAROUND:	14 Days or 35 Days
MATRICES:	Aqueous/Soil/Sediment*

SIGNIFICANT FEATURES

- **!** The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- **!** Volatiles and semivolatiles are analyzed by GC/MS; pesticides/PCBs are analyzed by GC/ECD.
- **!** Major Tentatively Identified Compounds (TICs) are reported for GC/MS analyses.
- **!** All pesticide/PCB analyses require second column confirmation by GC/ECD. Pesticide/PCB compounds are confirmed by GC/ concentration to be detected by GC/MS. (Concentrations in the sample extract at or above 10 ng/µL for pesticides, 50 ng/µL fo Toxaphene should enable the lab to confirm by GC/MS analysis.)

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent o contamination during Site Investigation (SI) and Remedial Investigation/Feasibility Study (RI/FS) activities and to verify that Reme (RD/RA) activities comply with the pre-determined clean-up standards. This method is suitable when a 14 day or 35 day turnaroun recommended for samples from known or suspected hazardous waste sites where potential contamination may be present at signific

* This method is not applicable to soil/sediment samples with high moisture content.

REVISIONS/MODIFICATIONS

The following is a list of significant changes from the SOW OLM01.9 SOW that are incorporated in the SOW OLM03.1 (inclu

Volatiles

- **!** Medium level soil/sediment extract and purge solution preparation procedures were revised for the method blank, MS/MSD, an
- **!** Minimum sample amount for low level soil/sediment analysis was lowered to 0.5 g.
- **!** Minimum initial and continuing calibration Relative Response Factor (RRF) for 1,1,2,2-Tetrachloroethane was lowered to 0.30
- **!** In addition to % recovery criteria, System Monitoring Compounds are evaluated based on Relative Retention Time (RRT) crite
- **!** The concentration of Methylene Chloride in method, storage, and instrument blanks must be less than 2.5 times the CRQL.

Semivolatiles

- **!** Low level soil CRQLs were changed from 800 ug/kg to 830 ug/kg for the following compounds: 2,4,5-Trichlorophenol, 2-Nitro Dinitrophenol, 4-Nitrophenol, 4-Nitroaniline, 4,6-Dinitro-2-methylphenol, and Pentachlorophenol.
- **!** The minimum RRF for initial and continuing calibrations for Acenaphthylene and Acenaphthene was changed to 0.900.
- **!** The RRT of each surrogate must be within ± 0.06 RRT units of its RRT in the continuing calibration standard. The sample(s) r RRT criteria are not met.
- **!** The GPC blank must contain target compounds at less than the CRQL, except phthalate esters (less than 5 times the CRQL).

Volatiles and Semivolatiles

- **!** GC/MS Performance Check: Background subtraction must be performed using a single scan acquired no more than 20 scans pr (VOA) or DFTPP (SVOA).
- **!** The number of volatile organic TICs was raised from 10 to 30; the number of semivolatile organic TICs was raised from 20 to and summed separately (not included in the 30). For both fractions, the data collection window is specified as 30 sec before the min after the last target analyte.
- **!** The non-TCL library search is performed using NIST/EPA/NIH (1992 or later) and/or Wiley (1991 or later) or equivalent.

Pesticides/PCBs

- **!** Packed columns must not be used for analysis.
- **!** Initial and continuing calibration PEM Resolution Criteria are ≥ 90%; %D criteria are ≥ -25% and ≤ 25%.
- **!** Initial calibration %RSD criteria are ≤ 20% except for alpha-BHC and delta-BHC which are ≤ 25%.
- **!** PEMs, INDAs, INDBs and instrument blanks must meet initial calibration criteria.
- **!** Surrogate advisory limits are 30-150%. Method blank surrogates must meet criteria.
- **!** Detailed Sulfur Cleanup Blank preparation and QC acceptance/corrective action procedures were added.

Semivolatiles and Pesticides/PCBs

- **!** Continuous L/L extraction procedures with and without hydrophobic membranes are provided for sample preparation.
- **!** GPC calibration solution concentrations changed for bis(2-Ethylhexyl)phthalate (0.5 mg/mL) and Methoxychlor (0.1 mg/mL).

All Fractions

- **!** The lab must measure the sample shipping cooler temperature at time of sample receipt using the cooler temperature indicator l the cooler. The lab must contact CLASS if the cooler's temperature exceeds 10°C.
- **!** Specifications for analysis of multiphase samples are included.
- **!** Detailed requirements were added for sample collection and preservation, standard analysis and documentation, and corrective standards, mixture standards, and blanks.
- **!** The MS/MSD are analyzed and reported at the same dilution as the least dilute sample for which the original sample results are

The following are significant changes from the OLM03.0 or OLM03.1 revision that are incorporated in the OLM03.2 revision:

- **!** Semivolatiles: Limits number of searched alkanes to 20 suspected alkane peaks of greatest apparent concentration. Alkanes ar 30 TICs.
- **!** Semivolatiles: Revised for clarification - if Internal Standard (IS) recoveries were outside criteria for a sample used for the MS sample extract is required only if IS recovery criteria were met in both the MS and MSD.
- **!** Pesticide/PCB: The pesticide GPC calibration check solution contains Endrin and Dieldrin at 0.5 ug/mL for a 2 mL GPC loop; contains Aroclor 1016 and Aroclor 1260 in methylene chloride at 0.5 ug/mL for a 2 mL GPC loop.
- **!** All fractions: Ampulated standard solution extracts in glass vials may be used until the manufacturer's expiration date, or 2 yrs manufacturer's expiration date is provided.

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Attachment I.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM03.2

TARGET COMPOUND LIST - VOLATILES

Compound	Aqueous CRQL ug/L, ppb	Low Soil CRQL ug/kg, ppb	Medium Soil CRQL ug/kg, ppb
Chloromethane	10	10	1200
Bromomethane	10	10	1200
Vinyl Chloride	10	10	1200
Chloroethane	10	10	1200
Methylene Chloride	10	10	1200
Acetone	10	10	1200
Carbon Disulfide	10	10	1200
1,1-Dichloroethene	10	10	1200
1,1-Dichloroethane	10	10	1200
1,2-Dichloroethene (total)	10	10	1200
Chloroform	10	10	1200
1,2-Dichloroethane	10	10	1200
2-Butanone	10	10	1200
1,1,1-Trichloroethane	10	10	1200
Carbon Tetrachloride	10	10	1200
Bromodichloromethane	10	10	1200
1,2-Dichloropropane	10	10	1200
cis-1,3-Dichloropropene	10	10	1200
Trichloroethene	10	10	1200
Dibromochloromethane	10	10	1200
1,1,2-Trichloroethane	10	10	1200
Benzene	10	10	1200
trans-1,3-Dichloropropene	10	10	1200
Bromoform	10	10	1200
4-Methyl-2-Pentanone	10	10	1200
2-Hexanone	10	10	1200
Tetrachloroethene	10	10	1200
1,1,2,2-Tetrachloroethane	10	10	1200
Toluene	10	10	1200
Chlorobenzene	10	10	1200
Ethylbenzene	10	10	1200
Styrene	10	10	1200

Xylenes (Total)	10	10	1200

NOTE:

! The sample-specific CRQLs for soil/sediment samples will be adjusted for percent moisture and will be higher than those listed above.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM03.2

TARGET COMPOUND LIST - SEMIVOLATILES

Compound	Aqueous CRQL ug/L, ppb	Low Soil CRQL ug/kg, ppb	Medium Soil CRQL ug/kg, ppb
Phenol	10	330	10000
bis(2-Chloroethyl)ether	10	330	10000
2-Chlorophenol	10	330	10000
1,3-Dichlorobenzene	10	330	10000
1,4-Dichlorobenzene	10	330	10000
1,2-Dichlorobenzene	10	330	10000
2-Methylphenol	10	330	10000
2,2'-oxybis(1-Chloropropane)	10	330	10000
4-Methylphenol	10	330	10000
N-Nitroso-di-n-propylamine	10	330	10000
Hexachloroethane	10	330	10000
Nitrobenzene	10	330	10000
Isophorone	10	330	10000
2-Nitrophenol	10	330	10000
2,4-Dimethylphenol	10	330	10000
bis(2-Chloroethoxy)methane	10	330	10000
2,4-Dichlorophenol	10	330	10000
1,2,4-Trichlorobenzene	10	330	10000
Naphthalene	10	330	10000
4-Chloroaniline	10	330	10000
Hexachlorobutadiene	10	330	10000
4-Chloro-3-methylphenol	10	330	10000
2-Methylnaphthalene	10	330	10000
Hexachlorocyclopentadiene	10	330	10000
2,4,6-Trichlorophenol	10	330	10000
2,4,5-Trichlorophenol	25	830*	25000
2-Chloronaphthalene	10	330	10000
2-Nitroaniline	25	830*	25000
Dimethylphthalate	10	330	10000
Acenaphthylene	10	330	10000
2,6-Dinitrotoluene	10	330	10000
3-Nitroaniline	25	830*	25000

NOTE:

! The sample-specific CRQLs for soil/sediment samples will be adjusted for percent moisture and will be higher than those listed above.
* Low level soil CRQLs were changed from 800 µg/kg in SOW OLM01.9 to 830 µg/kg in SOW OLM03.2.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM03.2

TARGET COMPOUND LIST - SEMIVOLATILES

Compound	Aqueous CRQL ug/L, ppb	Low Soil CRQL ug/kg, ppb	Medium Soil CRQL ug/kg, ppb
Acenaphthene	10	330	10000
2,4-Dinitrophenol	25	830*	25000
4-Nitrophenol	25	830*	25000
Dibenzofuran	10	330	10000
2,4-Dinitrotoluene	10	330	10000
Diethylphthalate	10	330	10000
4-Chlorophenyl-phenylether	10	330	10000
Fluorene	10	330	10000
4-Nitroaniline	25	830*	25000
4,6-Dinitro-2-methylphenol	25	830*	25000
N-Nitrosodiphenylamine	10	330	10000
4-Bromophenyl-phenylether	10	330	10000
Hexachlorobenzene	10	330	10000
Pentachlorophenol	25	830*	25000
Phenanthrene	10	330	10000
Anthracene	10	330	10000
Carbazole	10	330	10000
Di-n-butylphthalate	10	330	10000
Fluoranthene	10	330	10000
Pyrene	10	330	10000
Butylbenzylphthalate	10	330	10000
3,3'-Dichlorobenzidine	10	330	10000
Benzo(a)anthracene	10	330	10000
Chrysene	10	330	10000
bis(2-Ethylhexyl)phthalate	10	330	10000
Di-n-octylphthalate	10	330	10000
Benzo(b)fluoranthene	10	330	10000
Benzo(k)fluoranthene	10	330	10000
Benzo(a)pyrene	10	330	10000
Indeno(1,2,3-cd)pyrene	10	330	10000
Dibenzo(a,h)anthracene	10	330	10000

Benzo(g,h,i)perylene	10	330	10000

NOTE:

! The sample-specific CRQLs for soil/sediment samples will be adjusted for percent moisture and will be higher than those listed above.
* Low level soil CRQLs were changed from 800 µg/kg in SOW OLM01.9 to 830 µg/kg in SOW OLM03.2.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANIC ANALYSIS
MULTI-MEDIA, MULTI-CONCENTRATION
OLM03.2

TARGET COMPOUND LIST - PESTICIDES/PCBS

Compound	Aqueous CRQL ug/L, ppb	Soil CRQL ug/kg, ppb
alpha-BHC	0.050	1.7
beta-BHC	0.050	1.7
delta-BHC	0.050	1.7
gamma-BHC (Lindane)	0.050	1.7
Heptachlor	0.050	1.7
Aldrin	0.050	1.7
Heptachlor epoxide	0.050	1.7
Endosulfan I	0.050	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3
4,4'-DDT	0.10	3.3
Methoxychlor	0.50	17
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.050	1.7
gamma-Chlordane	0.050	1.7
Toxaphene	5.0	170
Aroclor-1016	1.0	33
Aroclor-1221	2.0	67
Aroclor-1232	1.0	33
Aroclor-1242	1.0	33
Aroclor-1248	1.0	33
Aroclor-1254	1.0	33
Aroclor-1260	1.0	33

NOTE:

! The sample-specific CRQLs for soil/sediment samples will be adjusted for percent moisture and will be higher than those listed above.

TITLE: USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANICS ANALYSIS
LOW CONCENTRATION WATER

DOCUMENT NUMBER:	OLC02.1
DOCUMENT DATE:	Not Applicable
EFFECTIVE DATES:	September 1996 through September 2000
CONCENTRATION:	Low
DATA TURNAROUND:	14 Days
MATRICES:	Aqueous*

SIGNIFICANT FEATURES

- The parameters include volatile, semivolatile, and pesticide/PCB compounds.
- Volatiles and semivolatiles are analyzed by GC/MS; pesticides/PCBs are analyzed by GC/ECD.
- All parameters have significantly reduced CRQLs as compared to SOW OLM03.2.
- A 25 ml aliquot of sample is purged for volatiles analysis.
- Semivolatile samples are extracted by continuous liquid-liquid extraction procedures; pesticide/PCB samples are extracted by separatory funnel or continuous liquid-liquid extraction procedures.
- Analysis of a Laboratory Control Sample (LCS) for each parameter is required.
- Analysis of an MS/MSD duplicate pair is <u>not</u> required.

REVISIONS/MODIFICATIONS

The following is a list of the significant changes from the 10/92 Low Concentration SOW that are incorporated in SOW OLC02.1:

- The lab must measure the sample shipping cooler temperature at time of sample receipt using the cooler temperature indicator blank, if present, located in the cooler. The lab must contact CLASS if the cooler's temperature indicator exceeds 10^0C.
- The term Laboratory Evaluation Sample (LES) replaced Performance Evaluation Sample.
- Compound 1,2,4-Trichlorobenzene was removed from the semivolatile Target Compound List (TCL) and added to the volatile TC
- The number of volatile TICs was raised from 10 to 30; the number of semivolatile TICs was raised from 20 to 30. Up to 10 alkanes, which are not part of the 30 semivolatile TICs, are searched.
- The separatory funnel extraction method for pesticide/PCB samples was added.
- Control limits for recovery of volatile, semivolatile, and pesticide/PCB Laboratory Control Sample (LCS) compounds were modifi

RECOMMENDED USES

This Contract Laboratory Program (CLP) method is recommended for broad spectrum analysis to define the nature and extent of potential low level organic contamination in water supplies during Site Investigation (SI) and Remedial Investigation/ Feasibility Study (RI/FS) activities and to verify that Remedial Design/Remedial Action (RD/RA) activities comply with pre-determined clean-up standards. This method attains lower detection limits than SOW OLM03.2 and can aid in the determination of low level contamination in public drinking water supplies. The majority of samples are expected to be from drinking water and well/ground water sources around Superfund sites. This method is suitable when a 14 day turnaround for results is adequate.

* This method may not be applicable for analysis of aqueous low concentration volatile organic samples when project DQOs require comparison of sample results to the drinking water Maximum Contaminant Levels (MCLs).

ANALYTES/CRQLs

The parameters included in the analysis and the Contract Required Quantitation Limits (CRQLs) are listed in Attachment 1.

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANICS ANALYSIS
LOW CONCENTRATION WATER
OLC02.1

TARGET COMPOUND LIST - VOLATILES

COMPOUND	CRQL (ug/L, ppb)
Chloromethane	1
Bromomethane	1
Vinyl chloride	1
Chloroethane	1
Methylene chloride	2
Acetone	5
Carbon disulfide	1
1,1-Dichloroethene	1
1,1-Dichloroethane	1
cis-1,2-Dichloroethene	1
trans-1,2-Dichloroethene	1
Chloroform	1
1,2-Dichloroethane	1
2-Butanone	5
Bromochloromethane	1
1,1,1-Trichloroethane	1
Carbon Tetrachloride	1
Bromodichloromethane	1
1,2-Dichloropropane	1
cis-1,3-Dichloropropene	1
Trichloroethene	1

COMPOUND	CRQL (ug/L, ppb)
Dibromochloromethane	1
1,1,2-Trichloroethane	1
Benzene	1
trans-1,3-Dichloropropene	1
Bromoform	1
4-Methyl-2-pentanone	5
2-Hexanone	5
Tetrachloroethene	1
1,1,2,2-Tetrachloroethane	1
1,2-Dibromoethane	1
Toluene	1
Chlorobenzene	1
Ethylbenzene	1
Styrene	1
Xylenes (Total)	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
1,2-Dichlorobenzene	1
1,2-Dibromo-3-chloropropane	1
1,2,4-Trichlorobenzene	1

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANICS ANALYSIS
LOW CONCENTRATION WATER
OLC02.1

TARGET COMPOUND LIST - SEMIVOLATILES

COMPOUND	CRQL (ug/L, ppb)
Phenol	5
bis(2-Chloroethyl)ether	5
2-Chlorophenol	5
2-Methylphenol	5
2,2'-oxybis(1-Chloropropane)	5
4-Methylphenol	5
N-Nitroso-di-n-propylamine	5
Hexachloroethane	5
Nitrobenzene	5
Isophorone	5
2-Nitrophenol	5
2,4-Dimethylphenol	5
bis(2-Chloroethoxy)methane	5
2,4-Dichlorophenol	5
Naphthalene	5
4-Chloroaniline	5
Hexachlorobutadiene	5
4-Chloro-3-methylphenol	5
2-Methylnaphthalene	5
Hexachlorocyclopentadiene	5
2,4,6-Trichlorophenol	5
2,4,5-Trichlorophenol	20
2-Chloronaphthalene	5
2-Nitroaniline	20
Dimethylphthalate	5
Acenaphthylene	5
2,6-Dinitrotoluene	5
3-Nitroaniline	20

COMPOUND	CRQL (ug/L, ppb)
4-Nitrophenol	20
Dibenzofuran	5
2,4-Dinitrotoluene	5
Diethylphthalate	5
4-Chlorophenyl-phenylether	5
Fluorene	5
4-Nitroaniline	20
4,6-Dinitro-2-methylphenol	20
N-Nitrosodiphenylamine	5
4-Bromophenyl-phenylether	5
Hexachlorobenzene	5
Pentachlorophenol	20
Phenanthrene	5
Anthracene	5
Di-n-butylphthalate	5
Fluoranthene	5
Pyrene	5
Butylbenzylphthalate	5
3,3'-Dichlorobenzidine	5
Benzo(a)anthracene	5
Chrysene	5
bis(2-Ethylhexyl)phthalate	5
Di-n-octylphthalate	5
Benzo(b)fluoranthene	5
Benzo(k)fluoranthene	5
Benzo(a)pyrene	5
Indeno(1,2,3-cd)pyrene	5
Dibenzo(a,h)anthracene	5

Acenaphthene	5
2,4-Dinitrophenol	20

Benzo(g,h,i)perylene	5

ATTACHMENT 1 (Page 3 of 3)

USEPA CONTRACT LABORATORY PROGRAM
STATEMENT OF WORK FOR ORGANICS ANALYSIS
LOW CONCENTRATION WATER
OLC02.1

TARGET COMPOUND LIST - PESTICIDES/PCBS

COMPOUND	CRQL (ug/L, ppb)
alpha-BHC	0.01
beta-BHC	0.01
delta-BHC	0.01
gamma-BHC (Lindane)	0.01
Heptachlor	0.01
Aldrin	0.01
Heptachlor epoxide	0.01
Endosulfan I	0.01
Dieldrin	0.02
4,4'-DDE	0.02
Endrin	0.02
Endosulfan II	0.02
4,4'-DDD	0.02
Endosulfan sulfate	0.02

COMPOUND	CRQL (ug/L,ppb)
4,4'-DDT	0.02
Methoxychlor	0.10
Endrin ketone	0.02
Endrin aldehyde	0.02
alpha-Chlordane	0.01
gamma-Chlordane	0.01
Toxaphene	1.0
Aroclor-1016	0.20
Aroclor-1221	0.40
Aroclor-1232	0.20
Aroclor-1242	0.20
Aroclor-1248	0.20
Aroclor-1254	0.20
Aroclor-1260	0.20

Analytical Services Available Through Superfund's Analytical Operations Branch/Contract Laboratory Program (CLP)

e Analytical Operations Branch of the Hazardous Site Evaluation Division has 18 analytical services available tc eet a variety of needs for Superfund decision-making. The following table shows types of analyses available, atement of work and fact sheet numbers, dates, turnaround times, lower limit of required quantitation, and uses, nitations, average costs, and monthly laboratory capacity. For additional information about a specific service, cluding target analytes, consult the method-specific fact sheet or call your Regional Sample Control Coordinator or LP Technical Project Officer, or call the Analytical Operations Branch on (703) 603-8870.

nalytical Services for Superfund

August 1993

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation[1]	Uses/Limitations/Costs/Capacity
Full Organics. Volatiles, Semivolatiles, Pesticides in Water and Soil (OLM0.8, 9240.0-08FS)	35 days 14 days	WATER: *Volatiles*: 10 µg/L *Semivolatiles*: 10-25 µg/L *Pesticides*: 0.05-0.10 µg/L, except methoxychlor 0.5 µg/L; Aroclors 1-2 µg/L, toxaphene 5 µg/L SOILS: *Volatiles*: Low conc. 10 µg/Kg; medium conc. 1,200 µg/Kg *Semivolatiles*: Low conc. 330-800 µg/Kg; medium conc. 10,000-25,000 µg/Kg *Pesticides*: 1.7-3.3 µg/Kg, except methoxychlor 17 µg/Kg; Aroclors 33-67 µg/Kg, toxaphene 170 µg/Kg	USES: To determine extent of organics contamination; assess potential for risk to human health and environment; determine appropriate clean-up action; determine when remedial actions are complete; determine absence of organic contaminants. LIMITATIONS: Main survey method to determine general organics concentration. Use for other than low concentration ground and drinking water samples. For suspected high hazard samples, use high concentration organic method. AVE COST: $ 790 per sample (35-day; all fractions) $1,355 per sample (14-day; all fractions) CAPACITY: 4,300 samples per month (35-day) 700 samples per month (14-day)
Low concentration Volatiles, Semivolatiles, Pesticides in Water (SOW Rev. 10/92, 9240.0-10FS) (available in SAS)	14 days	WATER: *Volatiles*: 1 µg/L (ketones 5 µg/L) *Semivolatiles*: 5-20 µg/L *Pesticides*: 0.01-0.02 µg/L, except methoxychlor 0.1 µg/L; Aroclors 0.2-0.4 µg/L, toxaphene 1.0 µg/L	USES: To determine extent of well/ground water contamination; assess potential for risk to human health; determine appropriate clean-up actions; determine when remedial actions are completed; determine absence of organic contaminants. LIMITATIONS: Use only for drinking/ground water type samples when concentrations for undiluted samples are not expected to exceed the upper limit of the calibration curves as follows: 25 µg/L for volatiles, 80 µg/L for semivolatiles, and 0.32 µg/L for pesticides except for Aroclors 6.4 µg/L and toxaphene 16 µg/L. AVE COST: $875 per sample (all fractions) $135 per sample (VOA only) CAPACITY: 50 samples per month (all fractions) 100 samples per month (VOA only)
Dioxins/Furans in Water, Fly Ash, Soil, and Waste (OLM01.1, 9240.0-17FS)	45 days	WATER: 0.01-0.05 µg/L FLY ASH: 1-5 µg/Kg SOIL: 1-5 µg/Kg WASTE: 10-50 µg/Kg	USES: To determine extent of dioxin/furan contamination; assess potential for risk to human health and environment; determine appropriate clean-up actions; determine when remedial actions are complete; determine absence of dioxin/furan contaminants. LIMITATIONS: Use only for specified matrices, analytes, and concentrations. AVE COST: $635 per sample CAPACITY: 50 samples per month

[1] The required quantitation level is a range of quantitation levels for analytes in a service. See the fact sheet for analyte- and technique-specific information.

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation	Uses/Limitations/Costs/Capacity
Rapid Turnaround Dioxins in Solids, Asphalt, Water, Air and Wipe (SOW Rev. 11/92)	16 hours (electronic) 40 hours (electronic, air) 7 days (hardcopy)	SOLIDS: 0.3 µg/Kg ASPHALT: 0.7 µg/Kg WIPE, WATER, AIR: 1.0 ng/sample	USES: To address dioxin situations requiring a quick answer or on-site feedback; identify critical samples for confirmatory analyses; optimize analysis conditions for confirmatory analyses. LIMITATIONS: Use only for samples likely to contain low level dioxin and when data are needed quickly. AVE COST: $166 per sample CAPACITY: 3,600 samples per month
Quick Turnaround Organics in Water, Soil/Solid, Wipe (QTM SOW Rev. 2/93, Draft Fact Sheet 7/93) (available in SAS)	48 hours* (electronic) 7 days (hardcopy) * validated data	WATER: *PAHs*: 20 µg/L *Phenols*: 50 µg/L *Aroclors*: 1-2 µg/L (toxaphene 5µg/L) *Pesticides*: 0.1 µg/L *Volatiles*: 20 µg/L SOIL/SOLID: *PAHs*: 330 µg/Kg *Phenols*: 830 µg/Kg *Aroclors*: 17-33 µg/Kg (toxaphene 83 µg/Kg) *Pesticides*: 1.7 µg/Kg *Volatiles*: 40 µg/Kg	USES: To address organics situations requiring a quick answer or on-site feedback; to direct sampling efforts, monitoring well placement; selection of screen intervals; to monitor cleanups and treatments for effectiveness. LIMITATION: Service is most effective when contaminants of concern are known or suspected and can be focused on analyte(s) or fraction(s) of concern. Use with the full organics service. AVE COST: $250 per fractional analysis CAPACITY: 90 fractional analyses per day 2,160 fractional analyses per month
Inorganics, Total Metals, Dissolved Metals, Cyanide in Water and Soil (ILM03.0,9240.0-09F5)	35 days 14 days	WATER: *Total metals, Dissolved metals*: 3-60 µg/L (Hg 0.2 µg/L) (Al 200 µg/L); (Ca, K, Mg, Na 5,000 µg/L) *Cyanide*: 10 µg/L SOIL: *Total metals*: 600-10[illegible] µg/Kg (Hg 100 µg/Kg) *Cyanide*: 1,000 µg/Kg	USES: To determine extent of inorganics contamination; assess potential for risk in human health and environment; determine appropriate clean-up action, determine when remedial actions are complete; determine absence of inorganic contaminants. LIMITATIONS: Main survey method to determine general inorganics concentration. Use for other than low concentration ground and drinking water samples. For suspected high hazard samples, use high concentration inorganic method. AVE COST: $109 per sample (35-day) $165 per sample (14-day) CAPACITY: 4,600 samples per month (35-day) 900 samples per month (14-day)
Toxicity Characteristic Leaching Procedure (TCLP SOW Rev. 6/93) (available in SAS)	35 days	ORGANICS *Volatiles, Semivolatiles*: 50 µg/L (pentachlorophenol and 2,4,5-trichlorophenol 125 µg/L) *Pesticides*: 1.0 µg/L (g-BHC 0.5 µg/L, toxaphene 50 µg/L) METALS 100-200 µg/L (Pd, Hg 40-60 µg/L, Ba 4,000 µg/L)	USES: Determination of toxicity characteristic for disposal of waste. LIMITATION: Quantitation limits are highly matrix-dependent. AVE COST: Organics: $675 per sample (all fractions) Inorganics: $135 per sample CAPACITY: Organics: 50 samples per month Inorganics: 50 samples per month

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation	Uses Limitations/Costs/Capacity
Multi-Concentration Water Quality Parameters (SOW Rev. 7/93, Draft Fact Sheet 6/93) (available in SAS)	14 days	PARAMETER *ALK, Cl, SO4* 2,000 µg/L *TOC, Nit, TP* 100 µg/L *NH3* 1,000 µg/L; *COD* 3,000 µg/L *TSS* 4,000 µg/L; *TDS* 10,000 µg/L	USES: To determine water quality; determine appropriate clean-up actions; determine when remedial actions are complete; determine presence or absence of water quality parameters. LIMITATIONS: Use when water samples contain low or medium levels of up to 10 water quality parameters. Quantitation limits are highly matrix-dependent. AVE COST: $215 per sample (all parameters) CAPACITY: 250 samples per month
High Concentration Total Metals and Cyanide in Liquid, Solid, and Multiphase Samples (HC01.2, 9240.0-18FS)	35 days	LIQUID, SOLID, AND MULTIPHASE: *Total metals*: 5,000-80,000 µg/Kg (for Hg 300 µg/Kg) *Cyanide*: 1,500 µg/Kg	USES: To determine extent of high level contamination; determine drum contamination; assess potential for risk to human health and environment; determine appropriate clean-up actions; determine presence or absence of high levels of inorganic contaminants. LIMITATION: Use for determination of other than low or medium concentration inorganic contaminants. AVE COST: $95 per sample CAPACITY: 100 samples per month
High Concentration Organics in Water - Immiscible Liquids and Solids (SOW Rev. 9/88 and 4/89) (available in SAS)	35 days	LIQUIDS and SOLIDS: *Volatiles*: 2.5-5.0 mg/Kg *Extractables*: 20-200 mg/Kg *Aroclors*: 10 mg/Kg *Toxaphene*: 50 mg/Kg	USES: To determine extent of high level contamination; determine drum contamination; assess potential for risk to human health and environment; determine appropriate clean-up actions; determine presence or absence of high levels of organic contaminants. LIMITATIONS: Use for determination of other than low or medium concentration organic contaminants. AVE COST: $650 (all fractions) CAPACITY: Project-specific requests
Low Concentration Total Metals, Cyanide, Total Nitrogen, Fluoride in Water (7/92 Draft, 9240.0-11FS) (available in SAS)	14 days	WATER: *Total metals*: 1-10 µg/L (Hg 0.2 µg/L) (As, Ni, Zn 20µg/L; Fe 100 µg/L) (Ca, Mg, Na 500 µg/L, K 750 µg/L) *Cyanide*: 10 µg/L *Tot Nit*: 100 µg/L *Fluoride*: 200 µg/L	USES: To determine extent of well/ground water contamination; assess potential for risk in human health; determine appropriate clean-up actions; determine when remedial actions are complete; determine absence or inorganic contaminants. LIMITATIONS: Use only for drinking/ground water type samples when concentrations for undiluted samples are not expected to exceed the upper limit of the calibration curves: 200 µg/L for Hg, 200-400 µg/L for cyanide, 1 mg/L for total nitrogen, 1000 mg/L for fluoride, and for the following analytes using flame AA - 7 mg/L for Ca, 0.5 mg/L for Mg, 2 mg/L for K, and 1 mg/L for Na. For analytes not described above, no upper calibration limit is specified. AVE COST: $215 per sample CAPACITY: Project-specific requests

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation	Uses/Limitations/Costs/Capacity
Analysis of Ambient Air (SOW Rev. VCAA01.0, VTAA01.0,SVAA01.0 and MAA01.0, 9240.0-15FSI (available in SAS)	35 days	ORGANICS. *Volatiles*: canister 2-5 ppbv; Tenax 2-48 ng on column *Semivolatiles, Pesticides*: 3.1-183 ng/m³ INORGANICS: *Total metals*: 1-109 ng/m³	USES: To determine extent of contamination; assess potential for risk in human health and environment; determine appropriate clean-up action; determine when remedial actions are complete; determine presence or absence of air contamination. LIMITATIONS: (Volatiles) For canister, insufficient flow rates and sampling times may prevent collection of sufficient quantity of sample; for Tenax, certain analytes are not captured. AVE COST: $700 per sample (organics) $375 per sample (metals) CAPACITY: Project-specific requests
General Radiochemical Analytical Services Protocol in Water and Soil (GRASP SOW Rev. 6/1/92) (available in SAS)	45 days	WATER *Gross Alpha*: 3 pCi/L *Gross Beta*: 2 pCi/L *Metals*: 0.05 pCi/L (for Ra, Sr-90 1 pCi/L; Cs-137 10 pCi/L; T 1,000 pCi/L) SOIL/SEDIMENT *Metals*: 0.05 pCi/g (for Ra, Sr-90 0.1 pCi/g)	USES: To determine presence or absence of radioactivity; assess potential for risk in human health and environment; determine appropriate clean-up action; determine when remedial actions are complete LIMITATION: Main survey method to determine general radioactivity AVE COST: $1,300 per sample (all analytes) CAPACITY: Project-specific requests

EPA

Analytical Services Available Through Superfund's Analytical Operations Branch/Contract Laboratory Program (CLP)

T nalytical Operations Branch of the Hazardous Site Evaluation Division has 18 analytical services available to -eet a variety of needs for Superfund decision-making. The following table shows types of analyses available, -tatement of work and fact sheet numbers, dates, turnaround times, lower limit of required quantitation, and uses, -nitations, average costs, and monthly laboratory capacity. For additional information about a specific service, -cluding target analytes, consult the method-specific fact sheet or call your Regional Sample Control Coordinator or P Technical Project Officer, or call the Analytical Operations Branch on (703) 603-8870.

-lytical Services for Superfund — October 1993

-pe of Analysis (State-ent of Work No., Fact -eet Number or Date)	Date Tumaround Time(s)	Lower Limit of Required Quantitation[1]	Uses/Limitations/Costs/Capacity
-l Organics, Volatiles, -emivolatiles, Pesticides Water and Soil -LM0.8, 9240.0-08FS) -vailable in RAS	35 days 14 days	WATER: *Volatiles*: 10 µg/L *Semivolatiles*: 10-25 µg/L *Pesticides*: 0.05-0.10 µg/L, except methoxychlor 0.5 µg/L; Aroclors 1-2 µg/L, toxaphene 5 µg/L SOILS: *Volatiles*: Low conc. 10 µg/Kg; medium conc. 1,200 µg/Kg *Semivolatiles*: Low conc. 330-800 µg/Kg; medium conc. 10,000-25,000 µg/Kg *Pesticides*: 1.7-3.3 µg/Kg, except methoxychlor 17 µg/Kg; Aroclors 33-67 µg/Kg, toxaphene 170 µg/Kg	USES: To determine extent of organics contamination; assess potential for risk to human health and environment; determine appropriate clean-up action; determine when remedial actions are complete; determine absence of organic contaminants. LIMITATIONS: Main survey method to determine general organics concentration. Use for other than low concentration ground and drinking water samples. For suspected high hazard samples, use high concentration organic method. AVE COST: $ 790 per sample (35-day; all fractions) $1,355 per sample (14-day; all fractions) CAPACITY: 4,300 samples per month (35-day) 700 samples per month (14-day)
-w concentration -latiles, Semivolatiles, -ticides in Water (SOW -v, 10/92, 9240.0-FS) -ailable as HQ Multi-ant SAS	14 days	WATER: *Volatiles*: 1 µg/L (ketones 5 µg/L) *Semivolatiles*: 5-20 µg/L *Pesticides*: 0.01-0.02 µg/L, except methoxychlor 0.1 µg/L; Aroclors 0.2-0.4 µg/L, toxaphene 1.0 µg/L	USES: To determine extent of well/ground water contamination; assess potential for risk to human health; determine appropriate clean-up actions; determine when remedial actions are completed; determine absence of organic contaminants. LIMITATIONS: Use only for drinking/ground water type samples when concentrations for undiluted samples are not expected to exceed the upper limit of the calibration curves as follows: 25 µg/L for volatiles, 80 µg/L for semivolatiles, and 0.32 µg/L for pesticides except for Aroclors 6.4 µg/L and toxaphene 16 µg/L. AVE COST: $675 per sample (all fractions) $135 per sample (VOA only) CAPACITY: 50 samples per month (all fractions) 100 samples per month (VOA only)
-oxins/Furans in Water, -Ash, Soil, and Waste -LM01.1, 9240.0-FS) -ail n RAS	45 days	WATER: 0.01-0.05 µg/L FLY ASH: 1-5 µg/Kg SOIL: 1-5 µg/Kg WASTE: 10-50 µg/Kg	USES: To determine extent of dioxin/furan contamination; assess potential for risk to human health and environment; determine appropriate clean-up actions; determine when remedial actions are complete; determine absence of dioxin/furan contaminants. LIMITATIONS: Use only for specified matrices, analytes, and concentrations. AVE COST: $635 per sample CAPACITY: 50 samples per month

[1] The required quantitation level is a range of quantitation levels for analytes in a service. See the fact sheet for analyte- and technique-specific information.

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation	Uses/Limitations/Costs/Capacity
Rapid Turnaround Dioxins in Solids, Asphalt, Water, Air and Wipe (SOW Rev. 11/92) Available in RAS	16 hours (electronic) 40 hours (electronic, air) 7 days (hardcopy)	SOLIDS: 0.3 µg/Kg ASPHALT: 0.7 µg/Kg WIPE, WATER, AIR: 1.0 ng/sample	USES: To address dioxin situations requiring a quick answer or on-site feedback; identify critical samples for confirmatory analyses; optimize analysis conditions for confirmatory analyses. LIMITATIONS: Use only for samples likely to contain low level dioxin and when data are needed quickly. AVE COST: $166 per sample CAPACITY: 3,600 samples per month
Quick Turnaround Organics in Water, Soil/Solid, Wipe (QTM SOW Rev. 2/93, Draft Fact Sheet 7/93) Available as HQ Multi-Client SAS	48 hours* (electronic) 7 days (hardcopy) * validated data	WATER: *PAHs*: 20 µg/L *Phenols*: 50 µg/L *Aroclors*: 1-2 µg/L (toxaphene 5 µg/L) *Pesticides*: 0.1 µg/L *Volatiles*: 20 µg/L SOIL/SOLID: *PAHs*: 330 µg/Kg *Phenols*: 830 µg/Kg *Aroclors*: 17-33 µg/Kg (toxaphene 83 µg/Kg) *Pesticides*: 1.7 µg/Kg *Volatiles*: 40 µg/Kg	USES: To address organics situations requiring a quick answer or on-site feedback; direct sampling efforts; locate sampling areas; monitor the placement of wells; select screen intervals; monitor cleanups and treatment for effectiveness. Can be used to monitor PRP activities and provide feedback at public meetings. LIMITATION: Service is most effective when contaminants of concern are known or suspected and can be focused on analyte(s) or fraction(s) of concern. Use with the full organics service AVE COST: $250 per fractional analysis CAPACITY: 90 fractional analyses per day 2,160 fractional analyses per month
Inorganics, Total Metals, Dissolved Metals, Cyanide in Water and Soil (ILM03.0/S240.0-CLP) Available in RAS	35 days 14 days	WATER: *Total metals, Dissolved metals*: 3-60 µg/L (Hg 0.2 µg/L) (Al 200 µg/L); (Ca, K, Mg, Na 5,000 µg/L) *Cyanide*: 10 µg/L SOIL: *Total metals*: 600-10^4 µg/Kg (Hg 100 µg/Kg) *Cyanide*: 1,000 µg/Kg	USES: To determine extent of inorganics contamination; assess potential for risk in human health and environment; determine appropriate clean-up action; determine when remedial actions are complete; determine absence of inorganic contaminants. LIMITATIONS: Main survey method to determine general inorganics concentration. Use for other than low concentration ground and drinking water samples. For suspected high hazard samples, use high concentration inorganic method. AVE COST: $109 per sample (35-day) $165 per sample (14-day) CAPACITY: 4,600 samples per month (35-day) 900 samples per month (14-day)
Toxicity Characteristic Leaching Procedure (TCLP SOW Rev. 6/93) Available as HQ Multi-Client SAS	35 days	ORGANICS *Volatiles, Semivolatiles*: 50 µg/L (pentachlorophenol and 2,4,5-trichlorophenol 125 µg/L) *Pesticides*: 1.0 µg/L (g-BHC 0.5 µg/L), (toxaphene 50 µg/L) METALS 100-200 µg/L (Pb, Hg 40-60 µg/L, Ba 4,000 µg/L)	USES: Determination of toxicity characteristic for disposal of waste LIMITATION: Quantitation limits are highly matrix dependent. AVE COST: Organics: $675 per sample (all fractions) Inorganics: $135 per sample CAPACITY: Organics: 50 samples per month Inorganics: 50 samples per month

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation	Uses Limitations/Costs/Capacity
Multi-Concentration Water Quality Parameters (SOW Rev. 7/93, Draft Fact Sheet 8/93) Available as HQ Multi-Client SAS	14 days	PARAMETER *ALK, Cl, SO4* 2,000 µg/L *TOC, Nit, TP* 100 µg/L *NH3* 1,000 µg/L; *COD* 3,000 µg/L *TSS* 4,000 µg/L; *TDS* 10,000 µg/L	USES: To determine water quality; determine appropriate clean-up actions; determine when remedial actions are complete; determine presence or absence of water quality parameters. LIMITATIONS: Use when water samples contain low or medium levels of up to 10 water quality parameters. Quantitation limits are highly matrix-dependent. AVE COST: $215 per sample (all parameters) CAPACITY: 250 samples per month
High Concentration Total Metals and Cyanide in Liquid, Solid, and Multiphase Samples (HC01.2, 9240.0-16FS) Available in RAS	35 days	LIQUID, SOLID, AND MULTIPHASE: *Total metals*: 5,000-80,000 µg/Kg (for Hg 300 µg/Kg) *Cyanide*: 1,500 µg/Kg	USES: To determine extent of high level contamination; determine drum contamination; assess potential for risk to human health and environment; determine appropriate clean-up actions; determine presence or absence of high levels of inorganic contaminants. LIMITATION: Use for determination of other than low or medium concentration inorganic contaminants. AVE COST: $95 per sample CAPACITY: 100 samples per month
High Concentration Organics in Water-Immiscible Liquids and Solids (SOW Rev. 9/88 and 4/89) Available in SAS	35 days	LIQUIDS and SOLIDS: *Volatiles*: 2.5-5.0 mg/Kg *Extractables*: 20-200 mg/Kg *Aroclors*: 10 mg/Kg *Toxaphene*: 50 mg/Kg	USES: To determine extent of high level contamination; determine drum contamination; assess potential for risk to human health and environment; determine appropriate clean-up actions; determine presence or absence of high levels of organic contaminants. LIMITATIONS: Use for determination of other than low or medium concentration organic contaminants. AVE COST: $650 (all fractions) CAPACITY: Project specific requests
Low Concentration Total Metals, Cyanide, Total Nitrogen, Fluoride in Water (7/92 Draft, 9240.0-11FS) Available as HQ Multi-Client SAS	14 days	WATER: *Total metals*: 1-10 µg/L (Hg 0.2 µg/L) (As, Ni, Zn 20µg/L; Fe 100 µg/L) (Ca, Mg, Na 500 µg/L; K 750 µg/L) *Cyanide*: 10 µg/L *Tot Nit*: 100 µg/L *Fluoride*: 200 µg/L	USES: To determine extent of well/ground water contamination; assess potential for risk in human health; determine appropriate clean-up actions; determine when remedial actions are complete; determine absence of inorganic contaminants. LIMITATIONS: Use only for drinking/ground water type samples when concentrations for undiluted samples are not expected to exceed the upper limit of the calibration curves: 200 µg/L for Hg, 200-400 µg/L for cyanide. 1 mg/L for total nitrogen, 1000 mg/L for fluoride, and for the following analytes using flame AA: 7 mg/L for Ca, 0.5 mg/L for Mg, 2 mg/L for K, and 1 mg/L for Na. For analytes not described above, no upper calibration limit is specified. AVE COST: $215 per sample CAPACITY: Project specific requests

Type of Analysis (Statement of Work No., Fact Sheet Number or Date)	Data Turnaround Time(s)	Lower Limit of Required Quantitation	Uses/Limitations/Costs/Capacity
Analysis of Ambient Air SOW Rev. VCAA01.0, VTAA01.0,SVAA01.0 and MAA01.0, 9240.0-15FS) Available in SAS	35 days	ORGANICS: *Volatiles*: canister 2-5 ppbv; Tenax 2-46 ng on column *Semivolatiles, Pesticides*: 37-183 ng/m³ INORGANICS: *Total metals*: 1-109 ng/m³	USES: To determine extent of contamination; assess potential for risk in human health and environment; determine appropriate clean-up action; determine when remedial actions are complete; determine presence or absence of air contamination. LIMITATIONS: (Volatiles) For canister, insufficient flow rates and sampling times may prevent collection of sufficient quantity of sample; for Tenax, certain analytes are not captured. AVE COST: $700 per sample (organics) $375 per sample (metals) CAPACITY: Project-specific requests
General Radiochemical Analytical Services Protocol in Water and Soil GRASP SOW Rev. 9/1/92) Available in SAS	45 days	WATER *Gross Alpha*: 3 pCi/L *Gross Beta*: 2 pCi/L *Metals*: 0.05 pCi/L (for Ra, Sr-90 1 pCi/L, Cs-137 10 pCi/L; T 1,000 pCi/L) SOIL/SEDIMENT *Metals*: 0.05 pCi/g (for Ra, Sr-90 0.1 pCi/g)	USES: To determine presence or absence of radioactivity; assess potential for risk in human health and environment; determine appropriate clean-up action; determine when remedial actions are complete LIMITATION: Main survey method to determine general radioactivity AVE COST: $1,300 per sample (all analytes) CAPACITY: Project-specific requests

TRAINING MANUAL FOR REVIEWING LABORATORY DATA PACKAGE COMPLETENESS

June 1994

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1.0 INTRODUCTION

When an analytical laboratory is requested/contracted by the Client to analyze field samples, the laboratory is required to provide adequate documentation supporting all current and future uses of the data. Potential uses of the data may include monitoring, modelling, risk assessment, site characterization, support of a remedy decision, and/or confirmation of treatment. Laboratory documentation and data may also be utilized in potential litigation as evidence.

Data packages produced by an analytical laboratory must contain all of the documents which were produced or used by the laboratory for that particular analysis. Although the specific documents required by the laboratory depends on the particular CLP RAS SOW or Client Request/Contract, in general, the laboratory data package must resemble as closely as possible the data packages required by the current CLP RAS SOWs for organics and inorganics. The tabulated summary forms provided in the SOWs must be utilized and modified appropriately, and qualifier flags such as those in the SOWs must be applied to the data as appropriate. For projects other than CLP RAS SOW projects, the data package must contain all modifications from the CLP RAS SOWs as specified in the Client Request/Contract.

This training manual provides procedures for reviewing laboratory data package completeness. Section 2.0 contains the specific laboratory documentation required in the data package, and Section 3.0 contains the specific information which must be provided on each document for both organic and inorganic analyses.

The required laboratory documentation and contents described in this manual closely resemble those required in the current CLP RAS SOWs for organics and inorganics. The exact format of the tabulated summary forms and specific documents required will depend on the particular analysis method and format requested/ contracted by the Client. Sections 2.0 and 3.0 contain comprehensive laboratory data package requirements which can be modified according to the Client Request/Contract. A checklist of the required laboratory documentation and contents for organic and inorganic data are provided in Attachment V. Attachment VI contains the Region I CSF Completeness Evidence Audit Program document, dated July 3, 1991.

2.0 LABORATORY DOCUMENTATION

The laboratory data package must adhere to the following general requirements:

- The data package must contain all original documents where possible
- The data package must be legible
- The data package must be clearly labeled and completed in accordance with Client instructions
- The data package must be arranged in increasing alphanumeric Client sample number order, or organized in a logical manner as specified by the Client Request/Contract
- The data package must be paginated consecutively in ascending order.

The laboratory data package documentation for both organic and inorganic analyses consists of the following comprehensive list:

1. Original sample data package including tabulated summary forms and raw data for field samples, standards, QC samples, and blanks (see below - sample data package)
2. A completed and signed Document Inventory Sheet used to record the inventory of the complete laboratory data package (see Attachments I and II for a comprehensive list of required documents for organics and inorganics, respectively)
3. All original shipping documents including, but not limited to, the following documents:
   a. Client Chain-of-Custody Records/Traffic Reports
   b. Airbills
   c. Custody Seals
   d. Sample tags (if present)
4. All original receiving documents including, but not limited to, the following documents:
   a. Sample Log-In Sheet used to document the receipt and inspection of samples and containers
   b. Other receiving forms or copies of receiving logbooks
   c. Sample Delivery Group cover sheet identifying the samples received for the group of samples in the data package
5. All original laboratory records of sample transfer, preparation, and analysis including, but not limited to, the following documents:
   a. Original preparation and analysis forms and/or copies of preparation and analysis logbook pages
   b. Internal sample and sample extract (organics) or sample digestate/distillate (inorganics) transfer chain-of-custody records

6. All other original project-specific documents in the possession of the laboratory including, but not limited to, the following documents:
   a. Telephone contact logs
   b. Copies of personal logbook pages
   c. All handwritten project-specific notes
   d. All other project-specific documents not covered by the above.

The sample data package must include data for analysis of all samples in that Sample Delivery Group including the following:

1. Narrative
2. Tabulated summary forms for:
   a. Field sample data (in increasing Client sample identification number)
   b. Laboratory standards (in chronological order by instrument)
   c. QC samples (in chronological order by type of QC sample)
   d. Blanks (in chronological order by instrument)
3. Raw data for field samples, laboratory standards, QC samples, and blanks (in chronological order by instrument)
4. Laboratory logbook pages for preparation and analysis of field samples, standards, QC samples, and blanks
5. Chain-of-Custody Records
6. Other project-specific documents in the laboratory's possession.

In addition, for organic data each type of tabulated summary form must be grouped by fraction (volatile, semivolatile, pesticide/PCB). Depending on whether the data package contains organic or inorganic analytical data, the required tabulated forms and format for field samples, standards, QC samples, and blanks will vary. Section 3.0 describes the specific information required for documentation of these analyses.

3.0 LABORATORY DATA PACKAGE CONTENTS

The following sections list the information which must be provided on each laboratory document (listed below) in the sample data package for organic and inorganic analytical data:

1. Narrative
2. Tabulated summary forms
3. Raw data
4. Logbook pages
5. Chain-of-Custody Records.

Because of the differences in the required information between organic and inorganic analyses on the tabulated summary forms and raw data, these documents are discussed separately for organic and inorganic data (Sections 3.4.1 and 3.4.2, respectively). The narrative, logbook pages, and Chain-of-Custody Record documentation for both organic and inorganic analyses require similar information and are discussed together below (Sections 3.1, 3.2, and 3.3). The specific requirements resemble those of the current CLP RAS SOWs and must be modified as appropriate to the Client Request/Contract.

3.1 Narrative

The narrative must describe the analytical methods and exact procedures performed by the laboratory as well as any deviations from the methods. The laboratory must document in detail all problems encountered with quality control, samples, shipment, and all analytical problems encountered in processing the samples. Problem resolution must be documented as well as any other factors which may affect the validity of the data. The laboratory must also discuss all unusual occurrences encountered during the analysis of the sample set. The laboratory must explain all data flags if not specified in the analytical method.

Additionally, for CLP RAS specific organic data the laboratory must list the pH of each water sample submitted for volatiles analysis. The laboratory must also list all instances of manual integrations performed by analysts. For CLP RAS specific inorganic data, the laboratory must indicate whether ICP interelement corrections and background corrections were applied.

The laboratory must provide examples of calculations of both a detected positive result and a detection/quantitation limit reported for each type of sample analysis. All equations, sample volumes, sample weights, dilution factors, percent solids/percent moisture, and other information required to reproduce the laboratory results must be indicated.

The narrative must also include the following additional items of information:

- Laboratory name
- Client Request/Contract project number
- CLP RAS or other Client sample identification numbers cross-referenced to the laboratory sample identification numbers.

In addition, the narrative must be signed and dated by the laboratory manager or designee.

Communication Logs

All telephone communications between the laboratory and sampling personnel or other parties outside the laboratory, which took place to resolve sampling discrepancies or analytical problems must be documented in detail on telephone communication logs. Those telephone logs must explicitly detail the problems discussed, the resolution agreed upon, the names and affiliations of the communicating parties, and the date the communication took place. All telephone logs must be appended to the narrative.

3.2 Laboratory Logbook Pages

The data package must contain the following laboratory logbook pages. Where possible, the originals must be submitted.

- Standards preparation logs
- QC sample preparation logs
- Sample preparation/extraction/digestion logs
- Sample analysis run logs
- Personal logs
- Hand-written project-specific notes.

The logbook pages must contain the following information where applicable:

- Laboratory name
- Client sample identification number
- Laboratory sample identification number
- Dates of preparation and analysis, and initials of preparer/analyst
- Source of standards and QC samples
- Weights and volumes of samples and standards
- Initial and final volumes of sample prepared/purged/extracted/digested
- Percent moisture/percent solids
- Injection/analysis volumes
- Date and time of sample injection
- Dilution factors.

3.3 Chain-of-Custody Records

Documentation must be provided of the traceability of the Client samples from the time the samples are released to the laboratory and while in the laboratory's possession. Two types of custody records must be provided in the data package: records of external sample transfer and custody from parties outside the laboratory, and records of internal sample transfer and custody within the laboratory.

On the external Chain-of-Custody Record, usually initiated by the Client/sampler, the laboratory is responsible for providing the following information:

- Date of sample receipt
- Signature of receiving personnel
- Condition of shipping containers and sample bottles upon receipt
- Condition of custody seals
- Presence/absence of airbills, custody seals, Client custody records, traffic reports, sample tags
- Problems or discrepancies with samples received or the documentation on the Chain-of-Custody Record.

The Chain-of-Custody Record also contains other vital information (e.g. sampling date/time, etc.) but documentation of this information is the responsibility of the Client/sampler.

The laboratory's internal Chain-of-Custody Records must contain the following information:

- Laboratory name
- Client sample identification number
- Laboratory sample identification number
- Date of sample transfer and receipt
- Signature of personnel transferring/receiving the sample
- Purpose of transfer/receipt, location of sample transferred.

3.4 Tabulated Summary Forms and Raw Data

The exact format of the tabulated summary form for each field sample, QC sample, standard, and blank will depend on the particular analysis method requested/contracted by the Client. The laboratory must provide certain information on all the tabulated summary forms and raw data: laboratory name, project/contract number, concentration units wherever numerical values are reported, and indication of wet or dry weight for solid matrices. The following sections list the specific requirements for organic data and inorganic data.

3.4.1 ORGANIC FORMS AND RAW DATA

Information required on tabulated summary forms are presented in the following sections for the organic data listed below:

1. Field sample results
2. Surrogate results (system monitoring compound results)
3. Matrix spike/matrix spike duplicate results
4. Method/laboratory blank results
5. Tuning results (GC/MS instrument performance check)
6. Initial calibration results (GC/MS)
7. Initial calibration results (GC)
8. Continuing calibration results (GC/MS)
9. Continuing calibration results (GC)
10. Internal standard results (GC/MS)
11. GC analytical sequence
12. Pesticide cleanup results
13. Pesticide/PCB identification summary
14. Method detection limit study results

Tabulated summary forms for organic data required by the CLP RAS SOWs are provided in Attachment III as examples.

Field Sample Results (Form 1)

Comprehensive tabulated summary forms must be prepared for each field sample analyzed by the laboratory. At a minimum, the tabulated summary forms must contain the following information:

- Client sample identification (ID) number
- Laboratory sample ID number
- Target compound names
- Tabulated analytical results for identification (numerical quantitation limits) and quantitation (positive hits) with concentration units
- Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags).

For each field sample, the tabulated summary forms must also contain the following information as appropriate to the analysis method:

- Laboratory file ID
- Sample matrix type
- Level of analysis (low, medium)
- Percent moisture or percent solids
- GC column
- Sample weights and/or sample volumes prepared/purged/ extracted/analyzed

- Initial and final extract and extract clean-up volumes, injection volume
- Clean-ups performed
- Dilution factor
- Measured pH
- Dates of sample receipt, extraction, and analysis.

Surrogate Results (System Monitoring Compound Results) (Form 2)

Surrogate recovery data help to evaluate the efficiency of the sample preparation and analysis procedures and analytical system. The tabulated surrogate results summary form must contain the following information:

- Sample matrix
- Level of analysis (low, med)
- GC column
- Client sample ID numbers
- Surrogate compounds added
- Percent recoveries of surrogates
- QC limits for all surrogate standards in field samples, QC samples, and blanks
- All outliers flagged
- Total number of surrogates outside QC limits
- Indication of surrogates diluted out.

Matrix Spike/Matrix Spike Duplicate Results (Form 3)

Matrix spike and matrix spike duplicate samples are analyzed to evaluate the effects of the sample matrix on the methods used for analysis. The tabulated MS/MSD results summary form must contain the following information:

- Sample matrix
- Level of analysis (low, med)
- Client sample ID number
- Matrix spike compounds added
- True concentrations of the spikes added
- Concentrations of the spike compounds observed in the spiked sample
- Sample concentration of each spike compound detected in the original unspiked sample for the MS and MSD
- Percent recoveries of the spiked compounds in the MS/MSD samples
- Relative percent differences of the spiked compounds between the MS and MSD samples
- QC limits for all spike compounds - percent recovery and relative percent difference
- All outliers flagged.

In addition to the above, the results for all target compounds in the MS and MSD samples must be tabulated on the summary forms used to tabulate the field sample results.

Method/Laboratory Blank Results (Form 4)

The laboratory must provide blank information to determine the levels of contamination associated with the processing and analysis of the samples for method blanks and, depending on the analysis method, laboratory (instrument) blanks. The tabulated method/laboratory blanks results form must contain the following information:

- GC column, instrument ID
- Date and time of analysis for the blank itself
- Date of extraction
- Matrix with which the blank is associated
- Level of analysis (low, med)
- Laboratory sample ID number
- List of Client field sample ID numbers and MS/MSD samples associated with each blank (separate forms are used for each blank)
- Laboratory file IDs of the samples and associated blank
- Dates/times of analysis for field samples and MS/MSD samples which are associated with each blank.

In addition, results for each method and laboratory instrument blank must be included on the tabulated summary forms that are used for field sample results (Form 1).

Tuning Results (GC/MS Instrument Performance Check) (Form 5)

For GC/MS analyses, the laboratory must perform instrument performance checks to assure correct mass calibration, mass resolution, and mass transmission. The tabulated GC/MS tuning results summary form must contain the following information:

- Instrument ID, laboratory file ID
- Date and time of injection for each tune compound analysis (each tune on a separate form)
- Tune compound name
- Mass-to-charge ratio (m/e) for each ion
- Ion abundance criteria
- Percent relative abundances.

The form must also contain the following tabulated information associated with each tune and in chronological order:

- Client sample ID numbers associated with that tune
- Laboratory sample ID numbers

- Laboratory file IDs
- Date and time of analysis for all field samples, MS/MSD samples, blanks, and standards associated with that tune
- All outliers flagged.

Initial Calibration Results (GC/MS) (Forms 6A-6C)

Prior to any analysis, the laboratory must initially calibrate the GC/MS system to determine the linearity of the response. The tabulated GC/MS initial calibration results summary form must contain the following information:

- Instrument ID, laboratory file IDs
- Purge method
- Dates and times of standard analyses for that initial calibration
- Target compound names
- Concentrations of the calibration standards
- Relative response factors for each target and surrogate compound at each standard concentration
- Mean relative response factors for each target and surrogate compound
- Percent relative standard deviations for each target and surrogate compound
- QC limits for each initial calibration (each initial calibration on a separate form) - minimum RRF, maximum % RSD values
- All outliers flagged.

Initial Calibration Results (GC) (Forms 6D-6G)

Because the identification of compounds using GC is based primarily on retention time data or pattern recognition, the retention times and retention time windows are crucial to the provision of valid data. Generally, the tabulated initial calibration results summary forms for GC systems consist of retention time and calibration factor information. The data for pesticides, generally multi-point calibrations, and PCBs, generally single-point calibrations, are usually provided on two separate forms.

The following retention time information must be documented on the initial calibration results summary form:

- Instrument ID, GC column
- Dates of analysis
- Concentration of the calibration standards
- Target compound and surrogate compound names
- Retention times for each target and surrogate compound at each standard concentration

- Mean retention times for each target and surrogate compound (if multi-point calibration)
- Retention time windows for each target and surrogate compound (QC limits).

The following calibration factor information must be documented as well:

- Instrument ID, GC column
- Dates of analysis
- Concentrations of the calibration standards
- Target compound and surrogate compound names
- Calibration factors for each target and surrogate compound at each standard concentration
- Mean calibration factor (for multi-point calibration) for each target and surrogate compound
- Percent Relative Standard Deviation for each target and surrogate compound
- QC limits - % RSD
- All outliers flagged.

Resolution between compounds is documented with the following information:

- Instrument ID, GC column
- Dates and times of analysis
- Laboratory sample ID
- Names of compounds for which resolution is measured
- Retention times for each of those compounds
- Percent resolution between each pair of compounds
- QC limits - % resolution
- All outliers flagged.

Continuing Calibration Results (GC/MS) (Form 7A-7C)

The continuing calibration standards are analyzed to verify the accuracy of the initial calibration. The tabulated continuing calibration results form must contain the following information:

- Instrument ID, laboratory file ID
- Purge method
- Date and time of continuing calibration analysis
- Date and time of initial calibration analysis associated with that continuing calibration
- Target compound and surrogate compound names
- Mean relative response factors from initial calibration for each target and surrogate compound
- Relative response factors from continuing calibration for each target and surrogate compound (each continuing calibration on a separate form)

- Percent differences for each compound
- QC limits for each target and surrogate compound (each continuing calibration on a separate form) - minimum RRF, maximum % D
- Concentrations of the continuing calibration standards
- All outliers flagged.

Continuing Calibration Results (GC) (Forms 7D, 7E)

The tabulated continuing calibration results form must contain the following information:

- Instrument ID, GC column
- Laboratory sample ID
- Dates and times of continuing calibration standards analysis
- Date of associated initial calibration analysis
- Target compound and surrogate compound names
- Retention time for each target and surrogate compound
- Calculated amount of standard
- Nominal amount of standard
- Relative Percent Difference for each compound
- QC control limits - RPD
- Percent breakdowns for compounds used to measure extent of breakdown (endrin and 4,4'-DDT) and combined breakdown
- QC limits - percent breakdown.

Internal Standard Results (GC/MS) (Forms 8A-8C)

Internal standard responses in all calibration standards, field samples, QC samples, and blanks are crucial to the provision of reliable analytical results because the internal standards are used to quantitate the compounds. The tabulated internal standard results summary forms must contain the following information.

- Instrument ID, laboratory file ID
- GC column, purge method
- Date and time of continuing calibration standard analysis
- Client sample identification numbers
- Internal standard compound names
- Retention times and area counts of the quantitation for each internal standard compound in the continuing calibration standard, field samples, MS/MSD samples, and blanks associated with that continuing calibration (separate form for each continuing or initial calibration)
- QC limits - area counts and retention times
- All outliers flagged.

GC Analytical Sequence (Form 8D)

The Client Request/Contract may require standards and samples to be analyzed according to a special sequence. In this case, the following information must be provided:

- Instrument ID, GC column
- Initial calibration dates
- List of Client sample ID numbers in that analytical sequence (in chronological order) for all standards, field samples, QC samples, and blanks
- Laboratory sample ID numbers
- Dates and times of analyses
- Mean surrogate retention times - from initial calibration
- Retention times of the surrogate compounds
- QC limits of the surrogates - retention times
- All outliers flagged.

Pesticide Cleanup Results (Form 9)

Tabulated summary forms may be required when cleanup procedures are employed during the preparation of pesticide extracts for analysis. The following information are documented for reporting the results of the check of the Florisil cartridges used to process samples and extracts, and to summarize the results of the calibration of the Gel Permeation Chromatography (GPC) used to process soil sample extracts for Pesticide/PCB analyses.

Florisil Cartridge Check Results (Form 9A):

- GC column
- Florisil cartridge lot number (each lot on a separate form)
- Date of check solution analysis
- Names of compounds in the Florisil cartridge check solution
- Amount of spike in the check solution
- Amount of spike recovered in the check solution
- Percent recoveries
- QC limits - percent recoveries
- All outliers flagged
- Client sample ID numbers associated with that Florisil cartridge
- Laboratory sample ID numbers associated with that Florisil cartridge
- Dates of sample analysis.

GPC Calibration Results (Form 9B):

- GPC column
- Calibration date of GPC column
- GC columns

- Name of spike compounds added to GPC column
- Amount of spike added
- Amount of spike recovered
- Percent recoveries
- QC limits - % recoveries
- All outliers flagged
- Client sample ID numbers associated with the GPC column calibration
- Laboratory sample ID numbers associated with the GPC column calibration
- Dates of sample analyses.

Pesticide/PCB Identification Summary (Form 10)

This form summarizes the quantitations of all target Pesticide/PCB compounds detected in each field sample, QC sample, and blanks. If no compounds are detected in a given sample, this form is not required.

- Instrument ID, GC columns
- Dates of analysis
- Client sample ID number (on a separate form for each sample)
- Laboratory sample ID number
- Target compound name detected
- Retention time of compound on each column
- Retention time windows
- Concentration (mean concentration for multicomponent compounds)
- Percent difference.

Method Detection Limit Study Results

The tabulated MDL study results must contain the following information:

- Target compound names
- Concentrations of spikes added
- Concentration detected for each MDL spike
- Standard deviation and calculated MDL for each target compound.

The exact procedure utilized to generate the MDLs must be documented in detail in the narrative. The equation and associated constant values utilized to calculate the MDL for each analysis must be documented. The column, instrument ID, trap composition, and operating conditions must be clearly documented in the raw data.

Raw Data

The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and matrix spike duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, all instrument printouts, strip chart recordings, chromatograms, quantitation reports, mass spectra, and other types of raw data generated by the laboratory for a particular project must be provided in the data package.

Typical raw data for organic GC/MS analyses includes, but is not limited to the following:

- Reconstructed total ion chromatogram for each sample or sample extract, standards, QC samples, and blanks
- Instrument quantitation reports containing the following information: laboratory sample identification number, Client sample identification number, date and time of analysis, retention time and/or scan number of quantitation ion with measured area, analyte concentration, copy of area table from data system, GC/MS instrument ID, laboratory file ID, column, trap composition, and operating conditions
- Raw and enhanced mass spectra for all positive target compound results in field samples; daily continuing calibration standard reference spectra for all positive field sample results
- Mass spectra and three library searched best-match mass spectra for all tentatively identified compounds reported
- Instrument normalized mass listing and the mass spectrum for each tune.

Typical raw data for organic GC analyses includes, but is not limited to the following:

- Chromatograms for field samples, calibration standards, QC samples, and blanks containing the following information: Client sample identification number, laboratory sample identification number, volume injected, date and time of injection, GC column identification, GC instrument identification, laboratory file ID, operating conditions, positively identified compounds must be labeled with the compound names either directly from the peak or on a printout of the retention times
- Chromatograms for both GC columns
- GC integration report or data system printout
- Manual worksheets.

3.4.2 INORGANIC FORMS AND RAW DATA

Tabulated summary form requirements are presented in the following sections for the inorganic data listed below:

1. Field sample results
2. Initial and continuing calibration verification results
3. Contract required detection limit standard results
4. Blank results
5. ICP interference check sample results
6. Matrix spike and post-digestion spike sample results
7. Duplicate sample results
8. Laboratory control sample results
9. Method of standard additions results
10. ICP serial dilution results
11. Instrument detection limits
12. ICP interelement correction factors
13. ICP linear ranges
14. Preparation log
15. Analysis run log and Furnace AA QC results.

Tabulated data reporting forms for inorganic data required by the CLP RAS SOWs are provided in Attachment IV as examples.

Field Sample Results (Form 1)

Comprehensive tabulated summary forms must be prepared for each field sample analyzed by the laboratory. At a minimum, the tabulated summary forms must contain the following information as appropriate to the analysis method:

- Sample matrix type
- Level of analysis (low, medium)
- Percent moisture or percent solids
- Date of sample receipt
- Client sample identification number
- Laboratory sample identification number
- Target analyte names
- Tabulated analytical results for identification (numerical detection/quantitation limits) and quantitation (positive hits) with concentration units
- Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags)
- Concentration qualifier - indication of results less than the contract required detection limits
- Analytical method used for each analyte (usually indicated with a symbol).

Information regarding sample weights, volumes, dilution factors, and dates of digestion and analysis which are provided on the field sample results summary forms for organic data are not typically provided on those forms for inorganic data. Instead, this information is provided on other tabulated forms which follow.

Initial and Continuing Calibration Verification Results (Form 2A)

Initial and continuing calibration verification standards are analyzed to verify and ensure the accuracy of the initial and continuing calibrations. The tabulated initial and continuing calibration verification results summary form must contain the following information:

- Sources of the initial and continuing calibration verification standards
- Target analyte names
- True values of the calibration verification standards
- Concentrations found for the calibration verification standards
- Percent recoveries
- QC limits - percent recoveries
- Analytical method used for each analyte (usually indicated with a symbol).

The order of reporting the initial and continuing calibration verification standards for each analyte must follow the order in which the standards were analyzed.

Contract Required Detection Limit Standard Results (Form 2B)

Contract required detection limit (CRDL) standards are analyzed to verify the linearity of the instrument near the contract required detection limit. The tabulated CRDL results form must contain the following information:

- Source of the CRDL standards
- Target analytes
- True values of the CRDL standard for each analyte
- Concentrations found for each analyte
- Percent recoveries for each analyte
- QC limits (if known).

The order of reporting the CRDL standard results for each analyte must follow the order in which they were analyzed.

Blank Results (Form 3)

The tabulated blank results summary form must, in general,

contain information for two types of blanks: initial and continuing calibration blanks, or instrument blanks, and preparation blanks. The tabulated blank results summary form must contain the following information:

- Matrix for which the preparation blank is associated
- Concentration units of each blank type
- Target analyte names
- Initial and continuing calibration blank results
- Preparation blank results
- Concentration qualifiers
- Analytical method used for each analyte (usually indicated with a symbol).

The order of reporting the initial and continuing calibration blanks and preparation blanks for each analyte must follow the order in which they were analyzed.

ICP Interference Check Sample Results (Form 4)

ICP interference check samples are analyzed to verify interelement and background correction factors by analyzing target analytes in the presence of interferents. The tabulated ICP interference check sample results forms must contain the following information:

- ICP instrument ID number
- Source of the ICS solutions
- Target ICP analytes
- True values of each target ICP analyte in the solution containing interferents only
- True values of each target ICP analyte in the solution containing interferents and analytes
- Concentrations of target ICP analytes detected in the solution containing interferents only
- Concentrations of target ICP analytes detected in the solution containing interferents and analytes
- Percent recoveries.

The order of reporting the interference check sample results for each analyte must follow the order in which they were analyzed.

Matrix Spike and Post-Digestion Spike Sample Results (Form 5A, 5B)

The matrix spike sample analysis provides information about the effect of the sample matrix on the digestion and measurement methodology. The post-digestion spike recovery is based on the addition of a known quantity of analyte to an aliquot of digested sample. The tabulated matrix spike and post-digestion spike

sample results (Forms VA and VB, respectively) must contain the following results:

- Sample matrix
- Level of analysis (low, medium)
- Percent solids of the sample
- Client sample identification number
- Target analyte names
- Concentrations of the spikes added to the sample
- Concentrations found in the spiked sample
- Concentration found in the unspiked sample
- Percent recoveries
- QC limits - percent recovery
- All outliers flagged
- Concentration qualifiers
- Analytical method used for each analyte (usually indicated with a symbol).

Duplicate Sample Results (Form 6)

Duplicate sample analysis provide information about the laboratory precision. The tabulated duplicate sample results form must contain the following information:

- Sample matrix
- Level of analysis (low, medium)
- Percent solids of the original sample and duplicate sample
- Client sample identification number
- Target analyte names
- Concentration of the original sample result
- Concentration of duplicate sample result
- Relative percent difference
- QC limits
- All outliers flagged
- Concentration qualifiers
- Analytical method used for each analyte (usually indicated with a symbol).

Laboratory Control Sample Results (Form 7)

Laboratory control sample results provide information about the efficiency of the digestion method and accuracy of the results. The tabulated laboratory control sample results form must contain the following information:

- Source of the laboratory control sample
- Matrix of the LCS
- Target analyte names
- True concentrations
- Concentrations found

- Percent recoveries
- QC limits
- Concentration qualifiers.

Because a laboratory control sample should be digested for each matrix and digestion batch, additional forms must be present as appropriate if more than one LCS for a matrix was analyzed.

Method of Standard Additions Results (Form 8)

The method of standard additions analysis may be performed by the laboratory to quantitate the analyte in the sample when matrix interferences are present. The tabulated method of standard additions results form must contain the following information:

- Client sample identification number
- Concentrations of each MSA spike added
- Absorbance detected in each MSA spike as well as the sample itself
- Final concentration
- Correlation coefficient
- All outliers flagged.

Results for different samples for each analyte must be reported sequentially.

ICP Serial Dilution Results (Form 9)

ICP serial dilution analyses provide information as to the extent of the matrix effects in the sample. The tabulated ICP serial dilution results form must contain the following information:

- Sample matrix
- Level of analysis (low, medium)
- ICP instrument ID
- Client sample identification number
- Target analyte names
- Concentrations of the undiluted sample result
- Concentrations of the diluted sample result
- Percent difference
- QC limits - percent difference
- All outliers flagged
- Concentration qualifiers
- Analytical method used for each analyte (usually indicated with a symbol).

Instrument Detection Limits (Form 10)

The tabulated instrument detection limit results form must contain the following information:

- Instrument ID numbers used for the IDL determination
- Date the IDLs were determined
- Wavelength and background used for each analyte
- Target analyte names
- Type of background correction used (where applicable)
- Instrument detection limits
- Contract required detection limits
- Concentration units
- Analytical method used for each analyte (usually indicated with a symbol.

ICP Interelement Correction Factors (Form 11)

This form must document for each ICP instrument used for analysis, the interelement correction factors applied by the laboratory to obtain the reported data. The tabulated ICP interelement correction factors form must contain the following information:

- ICP instrument ID number
- Wavelength for each analyte used for the determination
- Date of interelement correction factor determination
- Target ICP analyte names
- Interfering analytes with which the interelement correction factors were determined
- Interelement correction factors for each analyte.

ICP Linear Ranges (Form 12)

A linear range verification check standard must be analyzed and reported for each target analyte as this concentration is the upper limit of the ICP linear range beyond which results should not be reported without dilution of the sample. The tabulated ICP linear ranges must contain the following information:

- ICP instrument ID number
- Date of the linear range determination
- Integration time for each analyte
- Target ICP analytes
- Concentration of the upper limit of the linear range for each analyte.

Preparation Log (Form 13)

This form provides sample preparation information which documented in the laboratory logbook pages. The following information is required:

- Analytical method (each analytical method on a separate form)

- Client sample ID number of all field samples, QC samples, standards, and blanks digested/distilled
- Sample preparation date
- Sample weight
- Sample volume.

Analysis Run Log and Furnace AA QC Results (Form 14)

This form provides information as to the analytical sequence of each analyte and any dilution factors applied. The tabulated analysis run log summary form must provide the following information.

- Instrument ID
- Analytical method
- Start and end dates of the analytical sequence
- Client sample ID numbers in chronological order
- Dilution factors
- Time of analysis for each analytical sample and standard
- Analytes associated with the run sequence.

Furnace AA QC analysis results are also typically provided on this form. Because of the nature of the furnace AA technique, the Client Request/Contract may require special QC sample analyses for quantitation of field samples. The QC samples which may be required are the following: duplicate injections of each analytical and field sample, and post-digestion spikes of each field sample. For furnace AA analyses, the following additional information are required on the Analysis Run Log Form:

- Percent relative standard deviation of duplicate injections (outliers usually indicated with flags on the tabulated field sample results summary form, Form 1)
- Percent recoveries of post-digestion spikes

The above information must be reported on separate forms for each furnace AA analyte.

Raw Data

The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/ contracted. However, for each reported value for a particular project, the laboratory must include all raw data used to obtain that reported value.

Typical raw data for inorganic analyses include, but is not limited to the following:

- Instrument printouts, strip chart recordings, etc., for all field samples, QC samples, standards, and blanks containing the following information: laboratory sample identification number, Client sample ID number, date and time of analysis, absorbance/emission values, analyte concentration, instrument ID, lab file ID, and instrument operating conditions.
- Standard curve raw data, plotted standard curves, linear regression equations, and correlation coefficients.

4.0 ACRONYMS

AAS	Atomic Absorption Spectrometry
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GPC	Gel Permeation Chromatography
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
ICV	Initial Calibration Verification
ID	Identification
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSA	Method of Standard Additions
PB	Preparation Blank
QC	Quality Control
RAS	Routine Analytical Services
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
SOW	Statement of Work

ATTACHMENT I

ORGANIC DOCUMENT INVENTORY CHECKLIST

For hardcopy of Attachment I contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT II

INORGANIC DOCUMENT INVENTORY CHECKLIST

For hardcopy of Attachment II contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT III

EXAMPLE ORGANIC TABULATED SUMMARY FORMS

For hardcopy of Attachment III contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT IV

EXAMPLE INORGANIC TABULATED SUMMARY FORMS

For hardcopy of Attachment IV contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT V

LABORATORY DOCUMENTATION AND CONTENTS CHECKLIST

FOR ORGANIC AND INORGANIC DATA

LABORATORY DATA PACKAGE COMPLETENESS

DOCUMENTATION CHECKLIST

COMPLETE LABORATORY DATA PACKAGE DOCUMENTATION

1. Original **sample data package** including tabulated summary forms and raw data for field samples, standards, QC samples, and blanks (see below - sample data package)
2. A completed and signed Document Inventory Sheet used to record the inventory of the complete laboratory data package
3. All original shipping documents including, but not limited to, the following documents:
   a. Client Chain-of-Custody Records/Traffic Reports
   b. Airbills
   c. Custody Seals
   d. Sample tags (if present)
4. All original receiving documents including, but not limited to, the following documents:
   a. Sample Log-In Sheet used to document the receipt and inspection of samples and containers
   b. Other receiving forms or copies of receiving logbooks
   c. Sample Delivery Group cover sheet identifying the samples received for the group of samples in the data package
5. All original laboratory records of sample transfer, preparation, and analysis including, but not limited to, the following documents:
   a. Original preparation and analysis forms and/or copies of preparation and analysis logbook pages
   b. Internal sample and sample extract (organics) or sample digestate/distillate (inorganics) transfer chain-of-custody records
6. All other original project-specific documents in the possession of the laboratory including, but not limited to, the following documents:
   a. Telephone contact logs
   b. Copies of personal logbook pages
   c. All handwritten project-specific notes
   d. All other project-specific documents not covered by the above

SAMPLE DATA PACKAGE DOCUMENTATION

1. Narrative
2. Tabulated summary forms for
   - Field sample data (in increasing Client sample identification number)
   - Laboratory standards (in chronological order by instrument)
   - QC samples (in chronological order by type of QC sample)
   - Blanks (in chronological order by instrument)
3. Raw data for field samples, laboratory standards, QC samples, and blanks (in chronological order by instrument)
4. Laboratory logbook pages for preparation and analysis of field samples, standards, QC samples, and blanks
5. Chain-of-Custody Records
6. Other project-specific documents in the laboratory's possession

For organic data each type of tabulated summary form must be grouped by fraction (volatile, semivolatile, pesticide/PCB). Depending on whether the data package contains organic or inorganic analytical data, the required tabulated forms and format for field samples, standards, QC samples, and blanks will vary.

LABORATORY DATA PACKAGE COMPLETENESS

DOCUMENTATION CHECKLIST

ORGANIC TABULATED SUMMARY FORMS
1. Field sample results
2. Surrogate results (system monitoring compound results)
3. Matrix spike/matrix spike duplicate results
4. Method/laboratory blank results
5. Tuning results (GC/MS instrument performance check)
6. Initial calibration results (GC/MS)
7. Initial calibration results (GC)
8. Continuing calibration results (GC/MS)
9. Continuing calibration results (GC)
10. Internal standard results (GC/MS)
11. GC analytical sequence
12. Pesticide cleanup results
13. Pesticide/PCB identification summary
14. Method detection limit study results

INORGANIC TABULATED SUMMARY FORMS
1. Field sample results
2. Initial and continuing calibration verification results
3. Contract required detection limit standard results
4. Blank results
5. ICP interference check sample results
6. Matrix spike and post-digestion spike sample results
7. Duplicate sample results
8. Laboratory control sample results
9. Method of standard additions results
10. ICP serial dilution results
11. Instrument detection limits
12. ICP interelement correction factors
13. ICP linear ranges
14. Preparation log
15. Analysis run log and Furnace AA QC results

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - GENERAL CHECKLIST

DOCUMENTATION	CONTENTS
NARRATIVE	• Laboratory name • Client Request/Contract project number • CLP RAS or other Client sample identification numbers cross-referenced to the laboratory sample identification numbers • Analytical methods and exact procedures performed by the laboratory • Any deviations from the methods • All problems encountered with quality control, samples, shipment, and all analytical problems encountered in processing the samples • Problem resolution must be documented as well as any other factors which may affect the validity of the data • All unusual occurrences encountered during the analysis of the sample set • Explanation of all data flags if not specified in the analytical method • For CLP RAS specific organic data - pH of each water sample submitted for volatiles analysis, list of all instances of manual integrations performed • For CLP RAS specific inorganic data - whether ICP interelement corrections and background corrections were applied • Examples of calculations of both a detected positive result and a detection/quantitation limit reported for each type of sample analysis including all equations, sample volumes, sample weights, dilution factors, percent solids/percent moisture, and other information required to reproduce the laboratory results must be indicated • Signed and dated by the laboratory manager • All telephone communications appended to narrative
LABORATORY LOGBOOK PAGES	Logbook documentation: • Standards preparation logs • QC sample preparation logs • Sample preparation/extraction/digestion logs • Sample analysis run logs • Personal logs • Hand-written project-specific notes Logbook pages contents (where applicable): • Laboratory name • Client sample identification number • Laboratory sample identification number • Dates of preparation and analysis, and initials of preparer/analyst • Source of standards and QC samples • Weights and volumes of samples and standards • Initial and final volumes of sample prepared/purged/ extracted/digested • Percent moisture/percent solids • Injection/analysis volumes • Date and time of sample injection • Dilution factors
CHAIN-OF-CUSTODY RECORDS	External Chain-of-Custody Record: • Date of sample receipt • Signature of receiving personnel • Condition of shipping containers and sample bottles upon receipt • Condition of custody seals • Presence/absence of airbills, custody seals, Client custody records, traffic reports, sample tags • Problems or discrepancies with samples received or the documentation on the Chain-of-Custody Record Internal Chain-of-Custody Records: • Laboratory name • Client sample identification number • Laboratory sample identification number • Date of sample transfer and receipt • Signature of personnel transferring/receiving the sample • Purpose of transfer/receipt, location of sample transferred

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
FIELD SAMPLE RESULTS	• Client sample identification (ID) number • Laboratory sample ID number • Target compound names • Tabulated analytical results for identification (numerical quantitation limits) and quantitation (positive hits) with concentration units • Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags). • Laboratory file ID • Sample matrix type • Level of analysis (low, medium) • Percent moisture or percent solids • GC column • Sample weights and/or sample volumes prepared/purged/ extracted/analyzed • Initial and final extract and extract clean-up volumes, injection volume • Clean-ups performed • Dilution factor • Measured pH • Dates of sample receipt, extraction, and analysis
SURROGATE RESULTS	• Sample matrix • Level of analysis • GC column • Client sample ID numbers • Surrogate compounds added • Percent recoveries of surrogates • QC limits for all surrogate standards in field samples, QC samples, and blanks • All outliers flagged • Total number of surrogates outside QC limits • Indication of surrogates diluted out
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RESULTS	• Sample matrix • Level of analysis • Client sample ID number • Matrix spike compounds added • True concentrations of the spikes added • Concentrations of the spike compounds observed in the spiked sample • Sample concentration of each spike compound detected in the original unspiked sample for the MS and MSD • Percent recoveries of the spiked compounds in the MS/MSD samples • Relative percent differences of the spiked compounds between the MS and MSD samples • QC limits for all spike compounds - percent recovery and relative percent difference • All outliers flagged In addition to the above, the results for all target compounds in the MS and MSD samples must be tabulated on the summary forms used to tabulate the field sample results.

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
METHOD/LABORATORY BLANK RESULTS	• GC column, instrument ID • Date and time of analysis for the blank itself • Date of extraction • Matrix with which the blank is associated • Level of analysis • Laboratory sample ID number • List of Client field sample ID numbers and MS/MSD samples associated with each blank (separate forms are used for each blank) • Laboratory file IDs of the samples and associated blank • Dates/times of analysis for field samples and MS/MSD samples which are associated with each blank In addition, results for each method and laboratory instrument blank must be included on the tabulated summary forms that are used for the field sample results.
TUNING RESULTS (GC/MS INSTRUMENT PERFORMANCE CHECK)	• Instrument ID, laboratory file ID • Date and time of injection for each tune compound analysis (each tune on a separate form) • Tune compound name • Mass-to-charge ratio (m/e) for each ion • Ion abundance criteria • Percent relative abundances The form must also contain the following tabulated information associated with each tune and in chronological order: • Client sample ID numbers associated with that tune • Laboratory sample ID numbers • Laboratory file IDs • Date and time of analysis for all field samples, MS/MSD samples, blanks, and standards associated with that tune • All outliers flagged
INITIAL CALIBRATION RESULTS (GC/MS)	• Instrument ID, laboratory file IDs • Purge method • Dates and times of standard analyses for that initial calibration • Target compound names • Concentrations of the calibration standards • Relative response factors for each target and surrogate compound at each standard concentration • Mean relative response factors for each target and surrogate compound • Percent relative standard deviations for each target and surrogate compound • QC limits for each initial calibration (each initial calibration on a separate form) - minimum RRF, maximum %RSD values • All outliers flagged

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
INITIAL CALIBRATION RESULTS (GC)	Retention time information: ● Instrument ID, GC column ● Dates of analysis ● Concentration of the calibration standards ● Target compound and surrogate compound names ● Retention times for each target and surrogate compound at each standard concentration ● Mean retention times for each target and surrogate compound (if multi-point calibration) ● Retention time windows for each target and surrogate compound (QC limits) Calibration factor information: ● Instrument ID, GC column ● Dates of analysis ● Concentrations of the calibration standards ● Target compound and surrogate compound names ● Calibration factors for each target and surrogate compound at each standard concentration ● Mean calibration factor (for multi-point calibration) for each target and surrogate compound ● Percent Relative Standard Deviation for each target and surrogate compound ● QC limits - % RSD ● All outliers flagged Resolution information: ● Instrument ID, GC column ● Dates and times of analysis ● Laboratory sample ID ● Names of compounds for which resolution is measured ● Retention times for each of those compounds ● Percent resolution between each pair of compounds ● QC limits - % resolution ● All outliers flagged
CONTINUING CALIBRATION RESULTS (GC/MS)	● Instrument ID, laboratory file ID ● Purge method ● Date and time of continuing calibration analysis ● Date and time of initial calibration analysis associated with that continuing calibration ● Target compound and surrogate compound names ● Mean relative response factors from initial calibration for each target and surrogate compound ● Relative response factors from continuing calibration for each target and surrogate compound (each continuing calibration on a separate form) ● Percent differences for each compound ● QC limits for each target and surrogate compound (each continuing calibration on a separate form) - minimum RRF, maximum % D ● Concentrations of the continuing calibration standards ● All outliers flagged

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
CONTINUING CALIBRATION RESULTS (GC)	• Instrument ID, GC column • Laboratory sample ID • Dates and times of continuing calibration standards analysis • Date of associated initial calibration analysis • Target compound and surrogate compound names • Retention time for each target and surrogate compound • Calculated amount of standard • Nominal amount of standard • Relative Percent Difference for each compound • QC control limits - RPD • Percent breakdowns for compounds used to measure extent of breakdown (endrin and 4,4'-DDT) and combined breakdown • QC limits - percent breakdown
INTERNAL STANDARD RESULTS (GC/MS)	• Instrument ID, laboratory file ID • GC column, purge method • Date and time of continuing calibration standard analysis • Client sample identification numbers • Internal standard compound names • Retention times and area counts of the quantitation for each internal standard compound in the continuing calibration standard, field samples, MS/MSD samples, and blanks associated with that continuing calibration (separate form for each continuing or initial calibration) • QC limits - area counts and retention times • All outliers flagged
GC ANALYTICAL SEQUENCE	• Instrument ID, GC column • Initial calibration dates • List of Client sample ID numbers in that analytical sequence (in chronological order) for all standards, field samples, QC samples, and blanks • Laboratory sample ID numbers • Dates and times of analyses • Mean surrogate retention times - from initial calibration • Retention times of the surrogate compounds • QC limits of the surrogates - retention times • All outliers flagged

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
PESTICIDE CLEANUP RESULTS	Florisil Cartridge Check Results: • GC column • Florisil cartridge lot number (each lot on a separate form) • Date of check solution analysis • Names of compounds in the Florisil cartridge check solution • Amount of spike in the check solution • Amount of spike recovered in the check solution • Percent recoveries • QC limits - percent recoveries • All outliers flagged • Client sample ID numbers associated with that Florisil cartridge • Laboratory sample ID numbers associated with that Florisil cartridge • Dates of sample analysis GPC Calibration Results: • GPC column • Calibration date of GPC column • GC columns • Name of spike compounds added to GPC column • Amount of spike added • Amount of spike recovered • Percent recoveries • QC limits - % recoveries • All outliers flagged • Client sample ID numbers associated with the GPC column calibration • Laboratory sample ID numbers associated with the GPC column calibration • Dates of sample analyses
PESTICIDE/PCB IDENTIFICATION SUMMARY	• Instrument ID, GC columns • Dates of analysis • Client sample ID number (on a separate form for each sample) • Laboratory sample ID number • Target compound name detected • Retention time of compound on each column • Retention time windows • Concentration (mean concentration for multicomponent compounds) • Percent difference
METHOD DETECTION LIMIT STUDY RESULTS	• Target compound names • Concentrations of spikes added • Concentration detected for each MDL spike • Standard deviation and calculated MDL for each target compound The exact procedure utilized to generate the MDLs must be documented in detail in the narrative. The equation and associated constant values utilized to calculate the MDL for each analysis must be documented. The column, instrument ID, trap composition, and operating conditions must be clearly documented in the raw data.

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC RAW DATA

DOCUMENT	CONTENTS
RAW DATA	The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and matrix spike duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, all instrument printouts, strip chart recordings, chromatograms, quantitation reports, mass spectra, and other types of raw data generated by the laboratory for a particular project must be provided in the data package. Typical raw data for organic GC/MS analyses includes, but is not limited to the following: ● Reconstructed total ion chromatogram for each sample or sample extract, standards, QC samples, and blanks ● Instrument quantitation reports containing the following information: laboratory sample identification number, Client sample identification number, laboratory file ID, date and time of analysis, retention time and/or scan number of quantitation ion with measured area, analyte concentration, copy of area table from data system, GC/MS instrument ID, laboratory file ID, column, trap composition, and operating conditions ● Raw and enhanced mass spectra for all positive target compound results in field samples; daily continuing calibration standard reference spectra for all positive field sample results ● Mass spectra and three library searched best-match mass spectra for all tentatively identified compounds reported ● Instrument normalized mass listing and the mass spectrum for each tune. Typical raw data for organic GC analyses includes, but is not limited to the following: ● Chromatograms for field samples, calibration standards, QC samples, and blanks containing the following information: Client sample identification number, volume injected, date and time of injection, GC column identification, GC instrument identification, positively identified compounds must be labeled with the compound names either directly from the peak or on a printout of the retention times ● Chromatograms for both GC columns ● GC integration report or data system printout ● Manual worksheets

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
FIELD SAMPLE RESULTS	• Sample matrix type • Level of analysis (low, medium) • Percent moisture or percent solids • Date of sample receipt • Client sample identification number • Laboratory sample identification number • Target analyte names • Tabulated analytical results for identification (numerical detection/quantitation limits) and quantitation (positive hits) with concentration units • Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags) • Concentration qualifiers (indicating results less than the CRDL) • Analytical method used for each analyte (usually indicated with a symbol)
INITIAL AND CONTINUING CALIBRATION VERIFICATION RESULTS	• Sources of the initial and continuing calibration verification standards • Target analyte names • True values of the calibration verification standards • Concentrations found for the calibration verification standards • Percent recoveries • QC limits - percent recoveries • Analytical method used for each analyte (usually indicated with a symbol) The order of reporting the initial and continuing calibration verification standards for each analyte must follow the order in which the standards were analyzed.
CONTRACT REQUIRED DETECTION LIMIT STANDARD RESULTS	• Source of the CRDL standards • Target analytes • True values of the CRDL standard for each analyte • Concentrations found for each analyte • Percent recoveries for each analyte • QC limits (if known) The order of reporting the CRDL standard results for each analyte must follow the order in which they were analyzed.
BLANK RESULTS	• Matrix for which the preparation blank is associated • Concentration units for each blank type • Target analyte names • Initial and continuing calibration blank results • Preparation blank results • Concentration qualifiers • Analytical method used for each analyte (usually indicated with a symbol) The order of reporting the initial and continuing calibration blanks and preparation blanks for each analyte must follow the order in which they were analyzed.
ICP INTERFERENCE CHECK SAMPLE RESULTS	• ICP instrument ID number • Source of the ICS solutions • Target ICP analytes • True values of each target ICP analyte in the solution containing interferents only • True values of each target ICP analyte in the solution containing interferents and analytes • Concentrations of target ICP analytes detected in the solution containing interferents only • Concentrations of target ICP analytes detected in the solution containing interferents and analytes • Percent recoveries The order of reporting the interference check sample results for each analyte must follow the order in which they were analyzed.

CONTENTS - INORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
MATRIX SPIKE AND POST-DIGESTION SPIKE SAMPLE RESULTS	• Sample matrix • Level of analysis (low, medium) • Percent solids of the sample • Client sample identification number • Target analyte names • Concentrations of the spikes added to the sample • Concentrations found in the spiked sample • Concentration found in the unspiked sample • Percent recoveries • QC limits - percent recovery • All outliers flagged • Concentration qualifiers • Analytical method used for each analyte (usually indicated with a symbol) Separate forms are used to report matrix spike results and post-digestion spike results.
DUPLICATE SAMPLE RESULTS	• Sample matrix • Level of analysis (low, medium) • Percent solids of the original sample and duplicate sample • Client sample identification number • Target analyte names • Concentration of the original sample result • Concentration of duplicate sample result • Relative percent difference • QC limits • All outliers flagged • Concentration qualifiers • Analytical method used for each analyte (usually indicated with a symbol)
LABORATORY CONTROL SAMPLE RESULTS	• Source of the laboratory control sample • Matrix of the LCS • Target analyte names • True concentrations • Concentrations found • Percent recoveries • QC limits • Concentration qualifiers Because a laboratory control sample should be digested for each matrix and digestion batch, additional forms must be present as appropriate if more than one LCS for a matrix was analyzed.
METHOD OF STANDARD ADDITIONS RESULTS	• Client sample identification number • Concentrations of each MSA spike added • Absorbance detected in each MSA spike as well as the sample itself • Final concentration • Correlation coefficient • All outliers flagged Results for different samples for each analyte must be reported sequentially.
ICP SERIAL DILUTION RESULTS	• Sample matrix • Level of analysis (low, medium) • ICP instrument ID • Client sample identification number • Target analyte names • Concentrations of the undiluted sample result • Concentrations of the diluted sample result • Percent difference • QC limits - percent difference • All outliers flagged • Concentration qualifiers • Analytical method used for each analyte (usually indicated with a symbol)

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
INSTRUMENT DETECTION LIMITS	• Instrument ID numbers used for the IDL determination • Date the IDLs were determined • Wavelength and background used for each analyte • Target analyte names • Type of background correction used where applicable • Instrument detection limits • Contract required detection limits • Concentration units • Analytical method used for each analyte (usually indicated with a symbol
ICP INTERELEMENT CORRECTION FACTORS	• Wavelength for each analyte used for the determination • ICP instrument ID number • Date of interelement correction factor determination • Target ICP analyte names • Interfering analytes with which the interelement correction factors were determined • Interelement correction factors for each analyte
ICP LINEAR RANGES	• ICP instrument ID number • Date of the linear range determination • Integration time for each analyte • Target ICP analytes • Concentration of the upper limit of the linear range for each analyte
PREPARATION LOG	• Analytical method (each analytical method on a separate form) • Client sample ID number of all field samples, QC samples, standards, and blanks digested/distilled • Sample preparation date • Sample weight • Sample volume
ANALYSIS RUN LOG	• Instrument ID • Analytical method • Start and end dates of the analytical sequence • Client sample ID numbers in chronological order • Dilution factors • Time of analysis for each analytical sample and standard • Analytes for which the run sequence pertains Furnace AA QC analyses results are also typically provided on this form - see below.
FURNACE AA QC RESULTS	In addition to the information required on the Analysis Run Log (see above): • Percent relative standard deviation of duplicate injections (outliers usually indicated with flags on the tabulated field sample results summary form, Form 1) • Percent recoveries of post-digestion spikes The above information must be reported on separate forms for each furnace AA analyte.

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC RAW DATA

DOCUMENT	CONTENTS
RAW DATA	The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, for each reported value for a particular project, the laboratory must include all raw data used to obtain that reported value. Typical raw data for inorganic analyses include, but is not limited to the following: ● Instrument printouts, strip chart recordings, etc., for all field samples, QC samples, standards, and blanks containing the following information: laboratory sample identification number, Client sample ID number, date and time of analysis, absorbance/emission values, analyte concentration, instrument ID, lab file ID, and instrument operating conditions. ● Standard curve raw data, plotted standard curves, linear regression equations, and correlation coefficients.

ATTACHMENT VI

REGION I CSF COMPLETENESS EVIDENCE AUDIT PROGRAM

JULY 3, 1991

(See Attachment C of the Functional Guidelines)

Attachment H

"EPA Region I Performance Evaluation Program Guidance",
July 1996, Revision

EPA REGION I
PERFORMANCE EVALUATION PROGRAM GUIDANCE

July 1996

PREFACE

This document, the EPA Region I Performance Evaluation Program Guidance, July 1996, has been created to replace in its entirety, the U.S. EPA Region I Performance Evaluation Program Guidance, February 1995, Update. All documents that reference and/or utilize EPA Region I Performance Evaluation Program requirements must be revised to reflect these new procedures.

The use of single and/or double blind PE samples helps to ensure that environmental data collection activities result in the delivery of analytical data of known and documented quality, suitable for its intended use.

EPA REGION I
PERFORMANCE EVALUATION PROGRAM GUIDANCE

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1.0 CONTEXT FOR THE PERFORMANCE EVALUATION (PE) PROGRAM

This guidance provides the details on how to implement the EPA Region I PE Program requirements supporting the data quality system described in the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I, dated July 1996 or most recent revision. The use of single and/or double blind PE samples helps to ensure that environmental data collection activities result in the delivery of analytical data of known and documented quality, suitable for its intended use.

2.0 PURPOSE OF THE PE PROGRAM

The EPA Region I PE Program serves three major functions:

1. To identify a community of technically capable laboratories during laboratory pre-award evaluations,

2. To evaluate the performance of analytical laboratories over a period of time,

3. To provide information on a laboratory's ability to accurately identify and quantitate analytes of interest during the period of sample preparation and analysis.

In the third function, the EPA Region I PE Program works in conjunction with the tiered data validation approach that is described in the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses.

3.0 USE OF PE SAMPLES

3.1 Superfund Program

The EPA Region I PE Program applies to all Superfund fixed laboratory, field laboratory (full protocol analytical methods performed in a mobile or transportable field laboratory), and field screening analyses; regardless of the mechanism used to obtain analytical services, the funding source for the project, or the project lead (EPA or non-EPA entity) for the site work.

3.1.1 EPA Fund-lead and PRP/Federal Facility Oversight Projects

For EPA Fund-lead and PRP/Federal Facility Oversight Superfund projects, the EPA Region I PE Program applies to all analytical services obtained through Contract Laboratory Program (CLP) and non-CLP vehicles. Non-CLP vehicles include fixed laboratory, field laboratory and field screening analytical services provided directly by EPA or by EPA prime contractors and/or subcontractors under the Delivery of Analytical Services (DAS) mechanism. EPA-provided PE samples, which meet project Data Quality Objectives (DQOs), should be utilized when available as described in Section 4.0 of this guidance document. If appropriate PE samples meeting project DQOs are not available from EPA, then they should be obtained from commercial vendors.

3.1.2 Fund-lead Projects Performed by States or other Federal Agencies

For Fund-lead projects performed by States (under Cooperative Agreements) or other Federal Agencies (under Interagency Agreements) that utilize the CLP to obtain analytical services, EPA-provided PE samples should be utilized. When non-CLP vehicles are utilized to provide fixed laboratory, field laboratory, or field screening analytical services for these Superfund projects, then PE samples should be obtained from commercial vendors.

3.1.3 Non Fund-lead Projects

For Non Fund-lead Superfund projects undertaken by Potentially Responsible Parties (PRPs) or other Federal Agencies, the EPA Region I PE Program applies to all fixed laboratory, field laboratory, and field screening analytical services utilized for these projects. Appropriate PE samples meeting project DQOs must be utilized whenever environmental samples are collected. These PE samples are not generally available from EPA and should be obtained from commercial vendors.

3.1.4 EPA Region I PE Program Requirements for Superfund Projects

The following EPA Region I PE Program requirements apply to all Superfund projects:

- **!** One single or double blind PE sample should be used for each sample matrix, analysis parameter, and concentration level for each Sample Delivery Group (SDG) that is sent to a laboratory. An SDG is defined as a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. The PE samples should be counted as field samples in the 20 sample SDG total.

- **!** PE samples are required for all analytical testing when they are available from EPA or commercial vendors in the appropriate matrix and at the proper concentration level. Additionally, PE samples should contain as many target analytes as possible, but they must contain at least one of the target analytes, preferably a contaminant of concern at the site.

- **!** For soil/sediment/solid sampling events, it is not necessary to include an aqueous PE sample when the only aqueous samples are equipment and/or trip blanks **and** when a PE sample exists (from either EPA or a commercial vendor) for the soil/sediment/solid samples. However, an aqueous PE sample should be included with soil/sediment/solid samples when a soil/sediment/solid PE sample (from either EPA or a commercial vendor) does not exist for that analysis parameter.

The EPA-NE Data Validation (DV) Chemist should be contacted (as per Section 6.1.2) to obtain advice on identifying available commercial vendors of PE samples and choosing a proper PE sample.

3.2 Non-Superfund Programs

The EPA Region I QA Unit recommends that Non-Superfund programs utilize PE samples whenever environmental samples are collected. These PE samples are not available from EPA and should be obtained from commercial vendors. This recommendation applies to environmental sampling performed by EPA (OEME, OEP, etc.) and non-EPA entities (facilities, manufacturers, generators, States, other Federal Agencies, etc.) in support of Non-Superfund federal regulations such as RCRA, UST, CWA, NPDES, CAA, TSCA, FIFRA, etc.

The following PE Program requirements apply to all Non-Superfund projects:

- **!** One single or double blind PE sample should be used for each sample matrix, analysis parameter, and concentration level for each Sample Delivery Group (SDG) that is sent to a laboratory. An SDG is defined as a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. The PE samples should be counted

as field samples in the 20 sample SDG total.

- PE samples are required for all analytical testing when they are available from commercial vendors in the appropriate matrix and at the proper concentration level. Additionally, PE samples should contain as many target analytes as possible, but they must contain at least one of the target analytes, preferably a contaminant of concern

- For soil/sediment/solid sampling events, it is not necessary to include an aqueous PE sample when the only aqueous samples are equipment and/or trip blanks **and** when a PE sample exists from a commercial vendor for the soil/sediment/solid samples. However, an aqueous PE sample should be included with soil/sediment/solid samples when a soil/sediment/solid PE sample does not exist for that analysis parameter.

The EPA-NE DV Chemist should be contacted (as per Section 6.1.2) to obtain advice on identifying available commercial vendors of PE samples and choosing a proper PE sample.

4.0 APPLICATION OF PE SAMPLES

Attachment 1 provides a list of EPA-provided PE samples that are currently available through the Region I QA Unit for Fund-lead and PRP/Federal Facility Oversight Superfund work. Included for each PE material on the list is an example of an analytical application. Use of the PE samples is **NOT** limited to the example application. For instance, #90-001 or #95-001, Low/Medium Volatiles in Water, could be used for analysis by the CLP OLM03.1 Statement of Work, SW-846 method 8260, 40 CFR method 624, etc. Note that several catalogue numbers may exist for a particular method description and matrix. Generally, different catalogue numbers for a specific method description and matrix contain different analyte mixes and/or concentrations.

Note also that #90-005, metals in soil PE samples, now contain mercury plus the other metals. However, one bottle does not contain enough soil to carry out both mercury and metals preparation and analysis. If both analyses are to be performed on the soil, then two bottles of #90-005 must be requested when ordering.

If an aqueous PE sample is needed for mercury analysis, then #95-004 must be ordered since the aqueous #90-004 PE samples contain metals but do **not** contain mercury.

A list of PE samples that are available from commercial vendors is provided in Attachment 2 for use in Fund-lead Superfund projects, for Non Fund-lead Superfund projects, as well as for Non-Superfund projects. Tables I and II list the parameters and matrices for which various vendors can supply PE samples and Standard Reference Materials (SRMs), respectively. SRMs can be utilized as PE samples. Table III provides vendor telephone and telefax numbers. Individual vendors should be contacted directly to obtain current catalogue information. Current catalogue information must be consulted to ensure that particular PE samples will meet project DQOs for specific compounds/parameters, matrices and concentration levels. The list provided in Attachment 2 is not inclusive of all potential PE/SRM vendors and does not constitute an endorsement by EPA of any particular vendor or any specific PE sample. It is provided solely for reference in identifying potential commercial PE sample sources.

5.0 PLANNING FOR PE SAMPLE USE

The use of PE samples should be stipulated as an analytical Quality Control measure during the planning stage of each project. The utilization of PE samples in accordance with this guidance document, the EPA Region I Performance Evaluation Program Guidance, dated July 1996 or most recent revision, should be stipulated in every **DQO Summary Form, Quality Assurance Project Plan (QAPjP)** and/or **Sampling and Analysis Plan (SAP)** along with the frequency, analysis parameters, matrices, and concentration levels for which a PE sample will be used. The origin of the PE sample (EPA-provided or commercial vendor) should also be documented in the QAPjP and/or SAP.

Additionally, preparation and analysis of PE samples must be written into laboratory Technical Specifications as a QC requirement when obtaining analytical services from fixed and/or field laboratories.

PE samples are to be included in the sample count per SDG for CLP and DAS analyses, as well as any other analytical services mechanism. For example, 20 field samples and one equipment blank (for a specified concentration level, matrix, and analysis parameter) would require two SDGs and therefore two PE samples for a total of 23 samples.

6.0 RESPONSIBILITIES IN THE PE PROGRAM

6.1 Superfund Program

Figure 1 contains a flow chart that outlines the process, roles/responsibilities, and time frames for planning, obtaining, analyzing, scoring, and evaluating results for EPA-provided and commercial PE samples used in Superfund projects.

6.1.1 EPA-NE Performance Evaluation Chemist

The Performance Evaluation (PE) Chemist of the Region I Quality Assurance Unit (telephone # 617/860-4630, telefax # 617/860-4397) is responsible for the following activities:

- Providing a current list of EPA PE samples upon request,
- Supplying EPA PE samples to EPA Field Sampling Contractor Lead Chemists and EPA Field Sampling Personnel,
- Scoring analytical results for the EPA PE samples,
- Providing EPA PE sample score results to EPA Field Sampling Contractor Lead Chemists and EPA Field Sampling Personnel.

6.1.2 EPA-NE Data Validation Chemist

The EPA-NE Data Validation (DV) Chemist of the Region I Quality Assurance Unit (telephone # 617/860-4634) is responsible for the following activities:

- Tracking EPA PE samples and their analytical results,
- Notifying EPA Field Sampling Contractor Lead Chemists and EPA Field Sampling Personnel when EPA PE sample score results indicate laboratory performance problems,
- Trending laboratory performance based upon EPA PE sample score results,
- Providing advice on identifying commercial vendors of PE samples, choosing a proper PE sample, and evaluating resultant data quality.

6.1.3 EPA Field Sampling Contractor Lead Chemists and EPA Field Sampling Personnel

EPA Field Sampling Contractor Lead Chemists and EPA Field Sampling Personnel are responsible for the following activities (when CLP and/or non-CLP vehicles are used to obtain analytical services for EPA Fund-lead and PRP/Federal Facility Oversight projects):

- Determining PE sample needs during the project planning phase (scoping meetings, DQO Summary Forms, Quality Assurance Project Plans and/or Sampling and Analysis Plans),

- Identifying PE sample sources (EPA and commercial),

- Procuring commercial PE samples if necessary,

- Obtaining EPA PE samples from the EPA-NE PE Chemist according to the procedures outlined in Section 7.0 of this guidance document,

- Ensuring that every laboratory which is analyzing project samples receives and analyzes appropriate PE samples according to the frequency requirements described in Section 3.0 of this guidance document,

- Obtaining score results for EPA and/or commercial PE samples,

- Evaluating PE sample score results in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, and including a discussion of the PE sample score results in Tier I Validation Cover Letters and Tier II/III Data Validation Reports,

- Notifying the EPA-NE DV Chemist if EPA PE sample performance causes reduced payment or rejection of any CLP data.

6.1.4 States and Other Federal Agencies

6.1.4.1 Fund-lead CLP Projects

For Fund-lead projects performed by States or other Federal Agencies that utilize the CLP to obtain analytical services, the States and other Federal Agencies are responsible for performing the activities described in Section 6.1.3 of this guidance document.

6.1.4.2 Fund-lead Non-CLP Projects

When non-CLP vehicles are utilized for these projects (and, therefore, commercial PE samples must be used), then States or other Federal Agencies are responsible for the following activities:

- Determining PE sample needs during the project planning phase (scoping meetings, DQO Summary Forms, Quality Assurance Project Plans and/or Sampling and Analysis Plans),

- Identifying commercial PE sample sources,

- Procuring commercial PE samples,

- Ensuring that every laboratory which is analyzing project samples receives and analyzes appropriate commercial PE samples according to the frequency requirements described in Section 3.0 of this guidance document,

- Obtaining score results for commercial PE samples,

- Evaluating commercial PE sample score results in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, and including a discussion of the PE sample score results in Tier I Validation Cover Letters and Tier II/III Data Validation Reports.

6.1.5 Non Fund-lead Projects

For Non Fund-lead Superfund projects undertaken by PRPs or other Federal Agencies, the PRP or other Federal Agency is responsible for the following activities:

- Determining PE sample needs during the project planning phase (scoping meetings, DQO Summary Forms, Quality Assurance Project Plans and/or Sampling and Analysis Plans),
- Identifying commercial PE sample sources,
- Procuring commercial PE samples,
- Ensuring that every laboratory which is analyzing project samples receives and analyzes appropriate commercial PE samples according to the frequency requirements described in Section 3.0 of this guidance document,
- Obtaining score results for commercial PE samples,
- Evaluating commercial PE sample score results in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, and including a discussion of the PE sample score results in Tier I Validation Cover Letters and Tier II/III Data Validation Reports.

6.2 Non-Superfund Programs

EPA Site Managers and EPA Project Officers are responsible for ensuring that the EPA Region I PE Program requirements contained in Sections 3.0 through 5.0 of this guidance document are applied to environmental sampling performed by EPA (OEME, OEP, etc.) and non-EPA entities (facilities, manufacturers, generators, States, other Federal Agencies, etc.) in support of Non-Superfund federal regulations within EPA Region I.

The EPA or non-EPA entity performing sampling is responsible for:

- Determining PE sample needs during the project planning phase (scoping meetings, DQO Summary Forms, Quality Assurance Project Plans and/or Sampling and Analysis Plans),
- Identifying commercial PE sample sources,
- Procuring commercial PE samples,
- Ensuring that every laboratory which is analyzing project samples receives and analyzes appropriate commercial PE samples according to the frequency requirements described in Section 3.0 of this guidance document,
- Obtaining score results for commercial PE samples,

- Evaluating commercial PE sample score results in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, and including a discussion of the PE sample score results in Tier I Validation Cover Letters and Tier II/III Data Validation Reports.

The EPA-NE DV Chemist should be contacted (as per Section 6.1.2) to obtain advice on identifying available commercial vendors of PE samples, choosing a proper PE sample, or evaluating resultant analytical data quality.

7.0 DETAILED PROCEDURES FOR THE PE PROGRAM

7.1 Superfund Program

Specific procedures for obtaining and utilizing EPA-provided PE samples for the EPA Region I Performance Evaluation Program are provided below. These procedures must be followed by EPA Field Sampling Contractor Lead Chemists and EPA Field Sampling Personnel (whenever CLP and/or non-CLP vehicles are used to obtain analytical services for EPA Fund-lead and PRP/Federal Facility Oversight projects) and by States or other Federal Agencies that utilize the CLP to obtain analytical services for Fund-lead proje

1. The Lead Chemist (LC) from the EPA Field Sampling Contractor (ARCS, START, RACS, etc.), State, or other Federal Agency (ACOE, etc.), or the EPA Field Sampling Personnel (EFSP) telefaxes the EPA Region I PE Sample Request Forms to the EPA-NE PE Chemist at least one week prior to sampling. In an emergency, PE samples can be picked up within 24 hours of ordering, but this service cannot be guaranteed. The Lead Chemist should confirm by phone that the telefaxed request was received by the EPA-NE PE Chemist.

   The EPA Region I PE Sample Request Form must specify the catalogue numbers for requested PE samples, number of PE samples ordered, method description and applicable matrix, exact reference title/# for the analytical method which will be used to prepare and analyze the PE and field samples, and the requested date and time for pick-up. Any specific analytes or special concentrations needed for the project must be clearly indicated on the EPA Region I PE Sample Request Form in the "Required Analyte Concentration" field. If a specific analyte or special concentration, as requested by the Lead Chemist or EPA Field Sampling Personnel in the "Required Analyte Concentration" field, cannot be provided by an existing EPA PE sample, then the EPA-NE PE Chemist will notify the LC/EFSP by telephone. The LC/EFSP will then determine, based upon project DQOs, whether an EPA PE sample that does not contain that specific analyte or special concentration will be sufficient to meet project DQOs or whether a commercial PE sample will be utilized. Copies of blank and completed EPA Region I PE Sample Request Forms are provided in Attachment 3.

2. EPA PE samples with preparation instructions are received, verified, and logged out from the EPA-NE PE Chemist by the Lead Chemist or EPA Field Sampling Personnel at the pre-arranged date and time. The Lead Chemist or EPA Field Sampling Personnel ensures that PE samples are handled and stored properly until laboratory re

3. EPA sample numbers are assigned to the EPA PE samples during the sampling episode by the Lead Chemist (or sampling designee) or EPA Field Sampling Personnel. The EPA PE ampule numbers must be documented on the Traffic Report/Chain of Custody Forms to cross-reference EPA sample numbers and EPA PE ampule

4. The Lead Chemist (or sampling designee) or EPA Field Sampling Personnel then submits the EPA PE samples, along with the preparation instructions and field samples, to the laboratories performing the analytical work.

5. The laboratories analyze the EPA PE samples along with their respective SDGs of field samples according to the specified methods. For CLP, the laboratories provide the resultant data packages to the RSCC. For non-CLP, the laboratories provide the resultant data packages to the Lead Chemist.

6. When the RSCC submits the CLP data packages to the Lead Chemist or EPA Field Sampling Personnel, or when the Lead Chemist or EPA Field Sampling Personnel receives the non-CLP data package from the laboratory, then the Lead Chemist or EPA Field Sampling Personnel immediately (within 3 business days) telefaxes the EPA PE sample data (Form Is) to the EPA-NE PE Chemist. The corresponding EPA PE ampule number (ID#) must be written on the Form Is by the Lead Chemist or EPA Field Sampling Personnel. The complete analytical method reference (full method name, number, revision date, etc.) must also be written on the Form Is by the Lead Chemist or EPA Field Sampling Personnel.

7. The EPA PE Chemist scores the EPA PE data and telefaxes the results back to the Lead Chemist or EPA Field Sampling Personnel, usually within 2 business days.

8. The EPA PE sample score results are evaluated by the Lead Chemist or EPA Field Sampling Personnel, and the EPA PE sample score results are incorporated into the data validation process in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I, Sections 8.5.3 and 8.5.4. A discussion of the PE sample score results must be provided in Tier I Validation Cover Letters and Tier II/III Data Validation Reports.

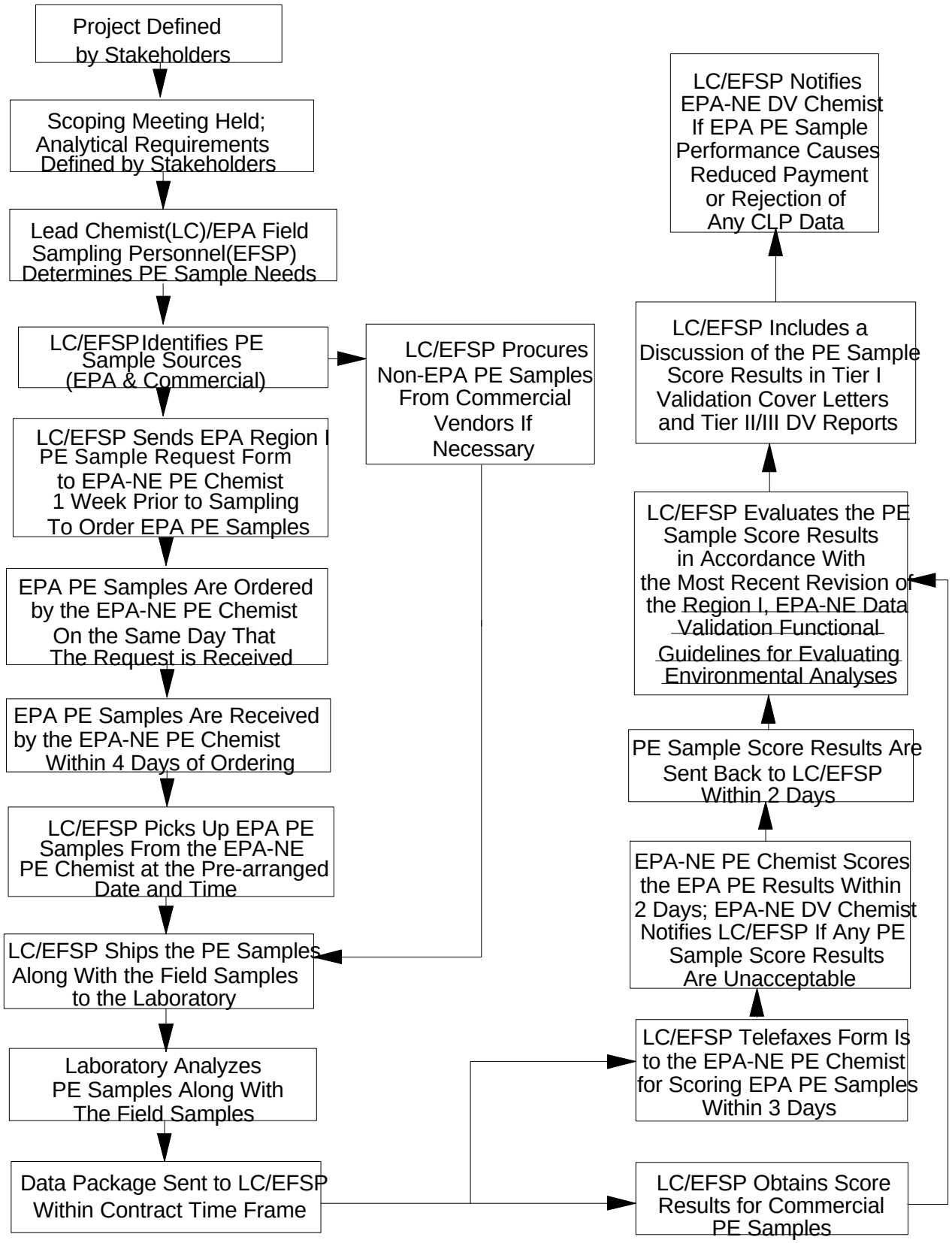
9. If poor performance on the EPA PE sample causes reduced payment or rejection of any CLP data, the Lead Chemist or EPA Field Sampling Personnel contacts the EPA-NE DV Chemist to initiate the reduced payment/data rejection process in accordance with the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I. For non-CLP data, the Lead Chemist does not contact the EPA-NE DV Chemist to initiate the reduced payment/data rejection process. For situations when resampling is recommended, the Lead Chemist or EPA Field Sampling Personnel (whichever performed the field sampling) must also contact the EPA Site Manager by telephone or electronic mail to alert them to the situation.

Similar procedures should be employed for obtaining and utilizing commercial PE samples for Fund-lead projects performed by States or other Federal Agencies that utilize non-CLP vehicles to obtain analytical services and for Non Fund-lead Superfund projects undertaken by PRPs or other Federal Agencies.

7.2 Non-Superfund Programs

EPA Site Managers and EPA Project Officers should establish SOPs for implementing use of PE samples in their Non-Superfund projects, containing similar activities and roles/responsibilities as described above for the Superfund Program. SOPs serve to augment the project's Quality Assurance documentation and to document project-specific procedures. SOPs are critical to producing environmental data that are consistent, comparable, credible and defensible.

FIGURE 1 - SUPERFUND PE PROGRAM PROCESS



ATTACHMENT 1

EPA REGION I PERFORMANCE EVALUATION SAMPLES
(7/96 Update)

PARAMETER	CATALOGUE NUMBER	METHOD DESCRIPTION & MATRIX	APPLICATION
ANIONS	91-006	Low Concentration Anions in Water	ILC01 & Rev.
METALS	91-004	Low Concentration Metals in Water	ILC01 & Rev.
	90-004	Low/Medium Metals in Water (No Mercury) (Use 95-004 for Mercury)	ILM04 & Rev.
	95-017	ICP/Graphite Furnace Metals (Low/Medium Metals in Water - No Mercury)	ILM04 & Rev.
	95-004	Low/Medium Mercury in Water	ILM04 & Rev.
	90-005	Low/Medium Metals in Soil (With Mercury) (Send two bottles when Metals and Mercury are required)	ILM04 & Rev.
	91-007	High Concentration Metals in Soil	HCIN
	91-008	High Concentration Metals in Soil/Oil	HCIN
	91-009	High Concentration Metals in Oil	HCIN
	91-010	High Concentration Metals in Oil/Water	HCIN
	94-020	ICC Industry Category Metals in Soil - Chemical and Allied Products (Various Levels Available)	ILM04 & Rev.
	94-021	ICC Industry Category Metals in Soil - Primary Metals Industries (Various Levels Available)	ILM04 & Rev.
	94-022	ICC Industry Category Metals in Soil - Mining (Various Levels Available)	ILM04 & Rev.
	94-023	ICC Industry Category Metals in Soil - Recyclers (Various Levels Available)	ILM04 & Rev.
	94-024	ICC Industry Category Metals in Soil- Other Waste Facilities (Various Levels Available)	ILM04 & Rev.
	95-009	ICC Industry Category Metals in Soil - Municipal Landfill (Various Levels Available)	ILM04 & Rev.
CYANIDE	91-005	Low Concentration Cyanide in Water	ILC01 & Rev.
	90-006	Low/Medium Cyanide in Water	ILM04 & Rev.
ASBESTOS	1 through 20	Asbestos Materials	EMSL-RTP Method

EPA REGION I PERFORMANCE EVALUATION SAMPLES
(7/96 Update)

PARAMETER	CATALOGUE NUMBER	METHOD DESCRIPTION & MATRIX	APPLICATION
VOLATILES	91-001	Low Concentration Volatiles in Water	OLC01, Method 524.2
	90-001	Low/Medium Volatiles in Water	OLM03 & Rev.
	95-001	Low/Medium Volatiles in Water	OLM03 & Rev.
	Requests to OEME	Volatiles in Air	TO-14, EMSL-RTP
SEMIVOLATILES	91-002	Low Concentration Semivolatiles in Water	OLC01 & Rev.
	90-002	Low/Medium Semivolatiles in Water	OLM03 & Rev.
	95-002	Low/Medium Semivolatiles in Water	OLM03 & Rev.
	95-010	ICC Industry Category Organics - General Manufacturing in Soil (PCBs High PPM Range)	OLM03 & Rev.
PESTICIDES/PCBs	91-003	Low Concentration Pesticides/PCBs in Water	OLC01 & Rev.
	90-003	Low/Medium Pesticides/PCBs in Water	OLM03 & Rev.
	95-003	Low/Medium Pesticides/PCBs in Water	OLM03 & Rev.
	91-011	Aroclor 1260 in Soil	OLM03 & Rev.
	91-012	Aroclor 1254 in Soil	OLM03 & Rev.
	91-013	Aroclor 1248 in Soil	OLM03 & Rev.

EPA REGION I PERFORMANCE EVALUATION SAMPLES
(7/96 Update)

PARAMETER	CATALOGUE NUMBER	METHOD DESCRIPTION & MATRIX	APPLICATION
DIOXINS/FURANS*	90-007	2,3,7,8-TCDD in soil (0.1-20 PPB)	GC/LRMS
	90-009	PCDD/PCDF in Soils (PPB Range PCDDs/PCDFs For Low Resolution MS)	GC/LRMS, DFLM01 & Rev.
	92-016	PCDD/PCDF With Interferences in Soil (PPB Range PCDDs/PCDFs and Interferences For Low Resolution MS)	GC/LRMS, DFLM01 & Rev.
	91-014	Interference Fortified Blank Soil (PPB Range Interferences For Low Resolution MS 2,3,7,8-TCDD and PCDD/PCDF Analyses)	GC/LRMS, DFLM01 & Rev.
	91-015	Blank Soil (For Low Resolution MS PCDD/PCDF Analyses)	GC/LRMS, DFLM01 & Rev.
	95-011	PCDD/PCDF in Soils (PPT Range PCDDs/PCDFs For High Resolution MS)	GC/HRMS, Method 1613B
	95-012	PCDD/PCDF With Interferences in Soil (PPT Range PCDDs/PCDFs with PPB Range Interferences For High Resolution MS) (Use with 95-013 as Blank)	GC/HRMS, Method 1613B
	95-013	Interference Fortified Blank Soil (PPB Range Interferences For High Resolution MS PCDD/PCDF Analyses) (Use with 95-012 as Spike)	GC/HRMS, Method 1613B
	95-014	Blank Soil (No PCDDs/PCDFs or Interferences >HRMS QL; For High Resolution MS PCDD/PCDF Analyses)	GC/HRMS, Method 1613B
	95-015	PCDD/PCDF in Incinerator Fly Ash (Various Levels Available)	Method 1613B & DFLM01 & Rev.
	95-016	PCDD/PCDF in "Environmental" Soil (Various Levels Available)	Method 1613B & DFLM01 & Rev.

*Dioxin/Furan Analyses require one Blank (or Interference Fortified Blank), and one Spike (or Interference Fortified Spike) at the appropriate concentration for the method. Note that blank and spike samples should be chosen so that the blank and spike pair either contains interferences or does not contain interferences, i.e., a spike containing interferences should not be paired with a blank that does not contain interferences and vice versa.

ATTACHMENT 2

EPA REGION I

PERFORMANCE EVALUATION

AND

STANDARD REFERENCE MATERIALS

VENDOR'S LIST

November 13, 1995
Revised November 22, 1995

7/96

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III List of PE/SRM Vendors . . . . . Att. 2-12

1.0 INTRODUCTION

The purpose of this report is to compile information concerning commercial vendors supplying Performance Evaluation (PE) Samples and Standard Reference Materials (SRM) for environmental analysis. The format of the report provides quick access to information regarding availability and source of the PE/SRM samples.

2.0 PERFORMANCE EVALUATION SAMPLES AND STANDARD REFERENCE MATERIALS VENDOR'S LIST

The information regarding commercial vendors who supply Performance Evaluation (PE) samples and Standard Reference Materials (SRM) was compiled into table format. To provide easy access to information, the data were classified by major analytical parameters and matrix with the particular vendor's name who supplies the described samples/materials. The above information is included in Tables I and II. Table III contains a list of PE/SRM vendors with their full name, telephone and telefax numbers.

The PE and SRM data were compiled based on the most current available catalogs (refer to Section 4.0 for details).

3.0 CONCLUSION

Upon review of available PE catalogues, it appears that some parameters/matrices do not have corresponding PE materials. Only the water matrix (including drinking water) is fully represented by the PE samples. Generally, the following matrices or parameters do not have PE samples (for specifics refer to Table I):

- Tissue - all parameters except dioxin
- Ash - all parameters
- Sediment - all parameters
- Pesticides in soil
- Herbicides in soil

4.0 REFERENCES

1. Analytical Products Group, Inc.: Performance Evaluation and Quality Control Drinking Water Standa
2. Analytical Products Group, Inc.: The Quality Assurance Challenge, 1995
3. Cambridge Isotope Laboratories: Environmental Contaminant Standards, 1994-1995
4. Chemcyclopedia, 1994
5. ChemService, 1995
6. Environmental Resource Associates (ERA): Environmental Quality Control Standards, 1995
7. Inorganic Ventures, Inc., 1995
8. National Institute of Standards and Technology (NIST): Standard Reference Materials, January 1994
9. Resource Technology Corporation (RTC): Standard Environmental Reference Materials, 1994
10. Scott Specialty Gases Catalog (Date unknown)
11. SPEX Industries, Inc.: Standards and Products for Inorganic Spectroscopy, 1995
12. ULTRA Scientific: Quality Control Standards, 1994

TABLE I
PERFORMANCE EVALUATION SAMPLE VENDOR'S LIST

PARAMETER	MATRIX	NAME OF VENDOR[1]
DEMAND (BOD, COD, TOC)	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
NUTRIENTS	Drinking Water	APG, Inorganic Ventures Inc., ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
HARDNESS	Drinking Water	Inorganic Ventures Inc., ULTRA Scientific
	Water	ERA, Inorganic Ventures Inc., ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
SOLIDS (TSS, TDS)	Drinking Water	APG, Inorganic Ventures Inc., ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
OIL & GREASE	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
MINERALS	Drinking Water	APG, Inorganic Ventures Inc., ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc,ULTRA Scientific
ANIONS	Water	ERA, SPEX
CATIONS	Water	ERA, Inorganic Ventures Inc., ULTRA Scientific
pH	Drinking Water	APG, ERA, Inorganic Ventures Inc.
	Water	APG, ERA, Inorganic Ventures Inc., SPEX
	Wastewater	Inorganic Ventures Inc.
TRACE METALS	Drinking Water	APG, ERA
	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
	Transformer Oil	ULTRA Scientific
METALS	Drinking Water	Inorganic Ventures Inc., ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Soil	ERA
	Sewage Sludge	ERA

TABLE I
PERFORMANCE EVALUATION SAMPLE VENDOR'S LIST

PARAMETER	MATRIX	NAME OF VENDOR[1]
INORGANICS BLANK	Sand	ERA
	Soil	ERA
TOTAL PHENOLICS	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
CYANIDE	Drinking Water	APG, SPEX, ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
	Soil	ERA
	Transformer Oil	ULTRA Scientific
RESIDUAL CHLORINE	Drinking Water	APG, Inorganic Ventures Inc., ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Wastewater	Inorganic Ventures Inc., ULTRA Scientific
CORROSIVITY/SODIUM	Water	SPEX
TURBIDITY	Drinking Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., SPEX, ULTRA Scientific
ALUMINUM - High Level	Water	APG
ASBESTOS	Water	ERA
FLUORIDE	Water	APG, SPEX
TOTAL ORGANIC HALIDES	Water	APG, SPEX
HEXAVALENT CHROMIUM	Water	APG
URANIUM	Water	APG

TABLE I
PERFORMANCE EVALUATION SAMPLE VENDOR'S LIST

PARAMETER	MATRIX	NAME OF VENDOR[1]
TRIHALOMETHANES	Drinking Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Water	ERA, Inorganic Ventures Inc., ULTRA Scientific
EDB/DBCP	Drinking Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Water	ERA, Inorganic Ventures Inc., ULTRA Scientific
VOLATILES	Drinking Water	APG, ERA, ChemService, Ultra Scientific
	Water	APG, ERA, ChemService, Inorganic Ventures Inc., ULTRA Scientific
	Wastewater	ULTRA Scientific
	Soil	ERA
	Transformer Oil	ULTRA Scientific
VOLATILES BLANK	Sand	ERA
	Soil	ERA
ACID EXTRACTABLES	Drinking Water	Inorganic Ventures Inc.
	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
BASE/NEUTRAL EXTRACTABLES	Water	APG, ERA, ULTRA Scientific
	Wastewater	ULTRA Scientific
SEMIVOLATILES	Drinking Water	APG, ERA, Inorganic Ventures Inc., ChemService, ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., ChemService, ULTRA Scientific
	Soil	ERA
	Transformer Oil	ULTRA Scientific
SEMIVOLATILES BLANK	Soil	ERA
PESTICIDES	Drinking Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Wastewater	ULTRA Scientific
CHLORDANE	Drinking Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Water	ERA, Inorganic Ventures Inc.
	Wastewater	ULTRA Scientific
HERBICIDES	Drinking Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Water	ERA, Inorganic Ventures Inc., ULTRA Scientific

TABLE I
PERFORMANCE EVALUATION SAMPLE VENDOR'S LIST

PARAMETER	MATRIX	NAME OF VENDOR[1]
CARBAMATE PESTICIDES	Drinking Water	APG, ERA, ChemService, Inorganic Ventures inc., ULTRA Scientific
	Water	ERA, ChemService, Inorganic Ventures Inc., ULTRA Scientific
TOXAPHENE	Drinking Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Water	ERA, Inorganic Ventures Inc., ULTRA Scientific
	Wastewater	ULTRA Scientific
PCBs	Drinking Water	APG, ERA, Inorganic Ventures Inc.
	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Wastewater	ULTRA Scientific
	Oil	ERA, ULTRA Scientific
	Soil	ERA, Cambridge Isotope Laboratories
	Fish	Cambridge Isotope Laboratories
PCB SCREENING (For Method 508A)	Drinking Water	ULTRA Scientific
	Wastewater	ULTRA Scientific
PCB AS DECACHLOROBIPHENYL	Drinking Water	ERA, APG, ERA
PAH	Water	APG, ERA
DIOXINS	Water	Cambridge Isotope Laboratories
	Soil	Cambridge Isotope Laboratories
	Fish	Cambridge Isotope Laboratories
BTEX	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Soil	ERA, Inorganic Ventures Inc.
GASOLINE	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Soil	ERA
DIESEL FUEL	Water	APG, ERA, Inorganic Ventures Inc., ULTRA Scientific
	Soil	ERA
TOTAL PETROLEUM HYDROCARBON	Water	APG, ERA, ULTRA Scientific
	Soil	ERA
CUSTOM MIXTURES	Air	Matheson Gas Products, Scott Specialty Gases

1 - Refer to Table III to obtain vendor's full name, telephone and telefax numbers.

TABLE II
STANDARD REFERENCE MATERIAL VENDOR'S LIST

INORGANICS		
STANDARD REFERENCE MATERIAL DESCRIPTION		NAME OF VENDOR[1]
Parameter	Matrix	
LEAD	Fuel	NIST
	Soil	NIST
	Paint Sludge	RTC
	Paint Waste	RTC
	Dust	RTC
MERCURY	Water	NIST
	Sediment	NIST
VANADIUM	Crude Oil	NIST
VANADIUM & NICKEL	Fuel Oil	NIST
TRACE ELEMENTS	Water	NIST
	Coal Fired Industrial Plant Ash	Inorganic Ventures Inc.
	Industrial Incinerator Ash	Inorganic Ventures Inc.
	Municipal Incinerator Ash	Inorganic Ventures Inc.
	Dry Soil	Inorganic Ventures Inc.
	Diatomaceous Earth Cake	Inorganic Ventures Inc.
	Sewage Sludge Amended Soil	Inorganic Ventures Inc.
	Paint Sludge	Inorganic Ventures Inc.
	Plating Sludge	Inorganic Ventures Inc.
	Contaminated Water Filter Media	Inorganic Ventures Inc.
	Paint Chips	Inorganic Ventures Inc.
	Dust	Inorganic Ventures Inc.
METALS	Ashes	RTC
	Soils	RTC
	Sludges	RTC
	Urban Particulates & Water Filtration Wastes	RTC

TABLE II
STANDARD REFERENCE MATERIAL VENDOR'S LIST

INORGANICS		
STANDARD REFERENCE MATERIAL DESCRIPTION		NAME OF VENDOR[1]
Parameter	Matrix	
TCLP METALS	Municipal Incinerator Ash	Inorganic Ventures Inc., RTC
	Superfund Site Soil	Inorganic Ventures Inc., RTC
GENERAL	Estuarine Sediment	NIST
	Urban Particulate Matter	NIST
	Used Pellet Autocatalyst	NIST
	Used Monolith Autocatalyst	NIST
	Simulated Rainwater	NIST
	Buffalo River Sediment	NIST
	San Joaquin Soil	NIST
	Montana Soil	NIST
	Sediments Lake	RTC
	Sediments Marine	RTC
	Sediments, Stream	RTC
	Soils	RTC
	Soil, Loam	RTC
	Soil, Sandy	RTC
	Sewage Sludge	RTC
	Fish Tissue	RTC
	Tuna Homogenate	RTC
	Cod Muscle	RTC
	Dogfish Liver	RTC
	Fish Tissue, Lyophilized	RTC
	Plankton	RTC
	Sargasso	RTC

TABLE II
STANDARD REFERENCE MATERIAL VENDOR'S LIST

ORGANICS		
STANDARD REFERENCE MATERIAL DESCRIPTION		NAME OF VENDOR[1]
Parameter	Matrix	
PHENOLS IN METHANOL	---	NIST
AROMATIC HYDROCARBONS/HEXANE, TOLUENE	---	NIST
HALOCARBONS FOR H_2O	---	NIST
PAHs	Separator Sludge	Inorganic Ventures Inc., RTC
	Contaminated Soil	Inorganic Ventures Inc., RTC
	Contaminated Soil/Sediment	Inorganic Ventures Inc., RTC
	Coal Tar	NIST
PRIORITY POLLUTANTS PAHs	---	NIST
NITRATED PAH IN METHANOL	---	NIST
NITROPYRENES IN CH_2Cl_2	---	NIST
CHLORINATED PESTICIDES/HEXANE	---	NIST
CHLORINATED PESTICIDES/ISOOCTANE	---	NIST
PESTICIDE, LINDANE	---	NIST
PESTICIDE, 4,4'-DDE	---	NIST
PESTICIDE, 4,4'-DDT	---	NIST
PCBs/ISOOCTANE	---	NIST
PCBs	Oil	NIST
	Transformer Oil	Inorganic Ventures Inc., RTC
	Soil	Inorganic Ventures Inc., RTC
	Soil/Sediment	Inorganic Ventures Inc.
	Human Serum	NIST
	River Sediment	NIST
CHLORINATED BIPHENYLS	---	NIST
ISOTOPE LABEL POLLUTANTS	---	NIST
DIOXIN	---	NIST

TABLE II
STANDARD REFERENCE MATERIAL VENDOR'S LIST

ORGANICS		
STANDARD REFERENCE MATERIAL DESCRIPTION		NAME OF VENDOR[1]
Parameter	Matrix	
GENERAL	Urban Dust	NIST
	Diesel Particulate Matter	NIST
	Mussel	NIST
	Oyster Tissue	NIST
	Shale Oil	NIST
	Petroleum Crude Oil	NIST
	Copepoda, Dried/PCBs & Pest	RTC
	Fish Tissue, Lyophilized/PCBs & Pest	RTC
	Sediment "Hot Spot"/PCBs & Pest	RTC
	Tuna Homogenate	RTC
	Marine Sediment	NIST
	Cod Liver Oil	NIST

TABLE II
STANDARD REFERENCE MATERIAL VENDOR'S LIST

ANALYZED GASES	
STANDARD REFERENCE MATERIAL DESCRIPTION	VENDOR'S NAME[1]
SO_2 Permeation Tube	NIST
NO_2 Permeation Device	NIST
Methane/Air	NIST
Methane + Propane/Air	NIST
SO_2/N_2	NIST
Propane/Air	NIST
CO_2/Air	NIST
CO_2/N_2	NIST
NO/N_2	NIST
CO_2, O_2/N_2	NIST
Organic Compounds/Nitrogen	NIST
Volatile Toxic Organics	NIST
Benzene/Nitrogen	NIST
Benzene, Toluene, Chlorobenzene, & Bromobenzene/Nitrogen	NIST
Carbon Tetrachloride, Chloroform, Tetrachloroethylene & Vinyl Chloride/N_2	NIST
CO_2/N_2O/Air	NIST
CO/Air	NIST
CO_2/N_2	NIST
NO/N_2	NIST
C_3H_8/N_2	NIST
Oxides of Nitrogen/Air	NIST
O_2/N_2	NIST
CO/N_2	NIST
IM Gases, 3 Components	NIST

1 - Refer to Table III to obtain vendor's full name, telephone and telefax numbers.

TABLE III
LIST OF PE/SRM VENDORS

NAME OF VENDOR	SUPPLIES (PE or SRM)	TELEPHONE NUMBER	TELEFAX NUMBER
1. Analytical Products Group, Inc. (APG)	PE	800-272-4442	614-423-5588
2. Cambridge Isotope Laboratories	PE	800-322-1174 508-749-8000	508-749-2768
3. ChemService	PE	800-452-9994 610-692-3026	610-692-8729
4. Environmental Resource Associates (ERA)	PE	800-372-0122 303-431-8454	303-431-0159
5. Inorganic Ventures, Inc.	PE & SRM	800-669-6799 908-901-1900	908-901-1903
6. Matheson Gas Products	PE	201-867-4100	201-867-4572
7. National Institute of Standards and Technology (NIST)	SRM	301-975-6776	301-948-3730
8. Resource Technology Corporation (RTC)	PE	307-742-5452	307-745-7936
9. Scott Specialty Gases	PE	617-245-8707	617-246-5452
10. SPEX Industries, Inc.	PE	800-522-7739 908-549-7144	908-603-9647
11. ULTRA Scientific	PE	800-338-1754	401-295-2330

ATTACHMENT 3

EPA REGION I
PERFORMANCE EVALUATION (PE) SAMPLE REQUEST FORM

Date of Request: _____
Requestor's Name: _____
Corporate Name: _____
Contract Name (if applicable): _____
Contract Number (if applicable): _____
Telephone Number: _____
Telefax Number: _____

Site Name and OU#: _____
Site ID # (CERCLIS, etc.): _____
Site Location: _____
Sampling Date(s): _____
Requested Pick-up Date and Time: _____

PE SAMPLES REQUIRED

CATALOGUE NUMBER	REQUIRED ANALYTE CONCENTRATION	NUMBER OF PE SAMPLES ORDERED	METHOD DESCRIPTION & MATRIX	ANALYTICAL METHOD

Example:

EPA REGION I
PERFORMANCE EVALUATION (PE) SAMPLE REQUEST FORM

Date of Request: 6/24/96
Requestor's Name: Ed Howard
Corporate Name: *3 X*
Contract Name (if applicable): *ARCS*
Contract Number (if applicable): *68-W8-0000*
Telephone Number: *000-222-1111*
Telefax Number: *000-222-2222*

Site Name and OU#: *Mac's Marsh, OU 2*
Site ID # (CERCLIS, etc.): *MAD000000000*
Site Location: *Debsville, MA*
Sampling Date(s): *7/3/96 and 7/10/96*
Requested Pick-up Date and Time: *6/30/96, 1:30 PM*

PE SAMPLES REQUIRED

CATALOGUE NUMBER	REQUIRED ANALYTE CONCENTRATION	NUMBER OF PE SAMPLES ORDERED	METHOD DESCRIPTION & MATRIX	ANALYTICAL METHOD
90-001 or 95-001		*3*	*L/M VOA in H_2O*	*OLM03 & Rev.*
90-002 or 95-002		*3*	*L/M SV in H_2O*	*OLM03 & Rev.*
90-003 or 95-003		*3*	*L/M P/P in H_2O*	*OLM03 & Rev.*
90-005	*Arsenic > 10 ppm*	*6*	*L/M Metals in Soil*	*ILM04 & Rev.*

Attachment I

"Standard Operating Procedures for Submitting Data for Reduced Payment/Data Rejection", September 9, 1991

ENVIRONMENTAL SERVICES DIVISION
60 Westview Street, Lexington, MA 02173-3185

MEMORANDUM

DATE: September 9, 1991

SUBJ: Standard Operating Procedures for Submitting Data for Reduced Payment/Data Rejection

FROM: Steve Stodola, Region I, Office of Quality Assurance

TO: Lead Chemists, ARC and TES Contractors

THRU: Heidi Horahan, ARC DPO

Several issues have come up recently regarding the submittal of data for reduced payment/data rejection. Please note the following:

1) The contact person for these issues is Steve Stodola at 617-860-4634. If necessary, use the voice mail to leave all the pertinent information - Case #, SDG #, Laboratory, Contractor (ARC or TES), Type of action needed reduced, payment or data rejection.

2) Review the data reduction/reduced payment flow chart and memo that was handed out at the lead chemist's meeting last September (1990). (See attachment.) Specifically, when it is determined that a package is <u>contractually</u> non-compliant, the lead chemist must contact the Region by phone by the end of the first week (or sooner, if possible). This process is very time critical because SMO must pay the invoices within 30 days unless we notify them of a problem.

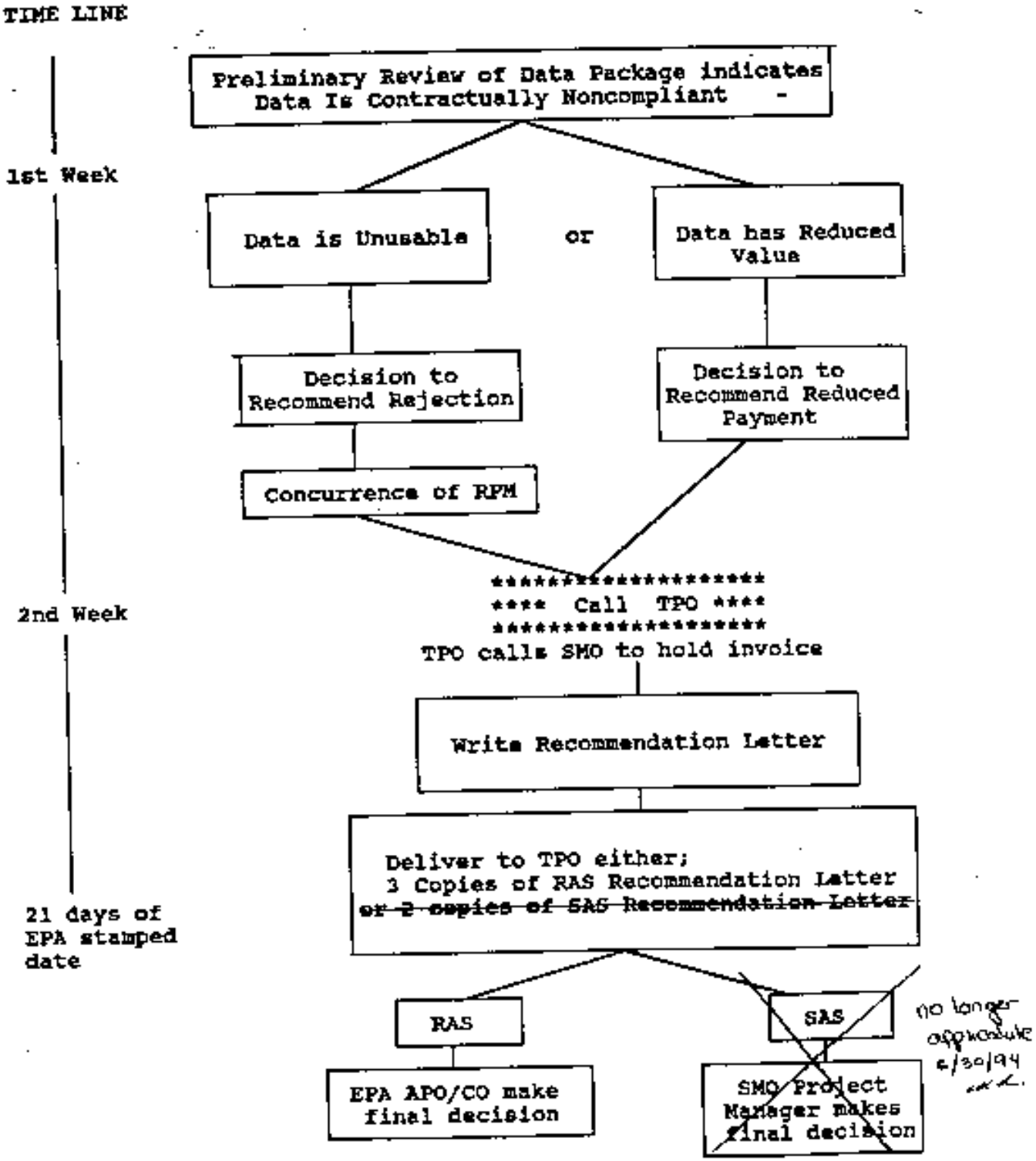
3) In the Recommendation Letter the lead chemist should not explicitly state "we recommend reduced payment (or data rejection)", since only the TPO can make the actual recommendation for reduced payment or data rejection. The letter should state that "the following information and documentation is provided to the Region to help in your evaluation of this case".

4) The Recommendation Letter must contain all the items mentioned in the memo handed out at the lead chemist's meeting. See RAS item 6) ~~and SAS item 7)~~.

5) The following items must be included with the attachments to the Recommendation Letter.

a) ~~SAS Request~~ N/A
b) Case Narrative
c) Traffic Report

DATA REJECTION/REDUCED PAYMENT OF RAS/SAS DATA /Contractor SOP
Sept 26, 1990



d) Chain of Custody
e) Phone Logs
f) Data Validation Worksheets - when appropriate
g) Raw data supporting the claims of noncompliance

ROUTINE ANALYTICAL SERVICES-DATA REJECTION/REDUCED PAYMENT

Background

Prior to the passage of the Amendments of 1988, the Prompt Payment Act required payment within 30 days of receipt of a valid invoice and interest payments for late payments. However, the Agency was granted a 15 day grace period before interest penalties were assessed. In October of 1988 the Prompt Payment Act Amendments were passed which abolished the 15 day grace period and required automatic interest payments after 30 days of receipt of a valid invoice.

What does this mean to EPA?

The data must be rejected or recommended for reduced payment (referred to as a qualified acceptance) within 30 days of receipt of a valid invoice. Otherwise, a constructive acceptance of the data occurs and the invoice is automatically paid when received.

What does this mean to you as a data validator?

It means that in order for you to recover analytical costs for the RPM on a data package you must submit in writing a letter detailing the contractual non-compliance of the data package no later than 21 day after the data package is received by EPA (stamped date).

RECOMMENDATION FOR REJECTION/REDUCED PAYMENT FOR RAS DATA

1. Perform preliminary review of the data package within the first week to determine if data package is contractually compliant with SOW.
2. Are the data unusable due to contract non-conformities?
   Determine if the entire SDG will be rejected or one or more fractions. Determine if the RPM has need of the data even if it is just to document that a sampling episode occurred. Once the data have been rejected EPA cannot use them. Obtain verbal concurrence to reject data from the RPM and document in a telephone log.
   Attach a copy of the telephone log to the letter recommending rejection of data.
3. Does the data have reduced value because of contractual non-conformities?
   If the data package is non-compliant but does not warrant rejection, submit recommendation for reduced payment. It is important at this point to assess whether or not it is worth pursuing a reduced payment action.
4. Call the TPO as soon as possible to notify her that a Data Rejection/Reduced Payment letter is being written.
5. TPO will call the SMO Section Leader, Contract Compliance Screening Group to indicate that Region I may request Data Rejection/Reduced Payment for the RAS case and to hold the invoice.
6. Within 21 days of the EPA stamped date write a letter to the TPO detailing contractual problems with the data package.

Include:

a. CASE and SDG numbers
b. Name of laboratory
c. Sample numbers
d. Copy of validation memo, if written
e. Parameters of issue
f. Specific references to the SOW by Exhibit, Section and page number
g. Resolution required (Data Rejection or Reduced Payment)
h. If recommendation is for rejection, attach all original data that are being rejected.
i. If recommendation is for reduced payment, attach copies of supporting documentation.

7. Send the recommendation letter and supporting documentation in triplicate to the TPO with the original data, if the data are to be rejected. Do not make copies of the original data if they are rejected.
   Note: Letters that do not cite specific contractual problems will not be forwarded by the TPO since non-contractual technical problems do not constitute reason for non-payment by the contracting officer.
8. The TPO will review the recommendation and if she concurs with it will write a cover letter to the Section leader, Contract Compliance Screening group, with a REJECTION/REDUCED VALUE COVER SHEET and send it by overnight delivery to SMO. A copy will be sent to the APO and one kept on file.
9. While it is the sole responsibility of the Region to reject or accept the data it remains the APO's responsibility to determine payment or non-payment based on our recommendations.
10. In the case of rejected data, the APO will document in a memo to the TPO indicating that the decision to reject data was either approved or not.
    If rejected, the entire analytical cost will be recovered for that sample or sample fraction.
11. In the case of data for reduced payment the APO will forward the recommendation to the EPA contracting officer. If the CO concurs with the recommendation she will negotiate a reduced payment with the laboratory. The decision to proceed with reduced payment will be documented in a memo to the TPO.
    A Reduced Value report may be obtained for samples from the contracting office through the APO.

Attachment J

Data Validation Report - Blank Forms

i. DQO Summary Form
ii. ORDA/IRDA Form
iii. Telephone Log or Regional/Laboratory Communication Form
iv. Data Validation Worksheets
v. Chain-of-Custody Form
vi. Traffic Report

A separate Form should be completed for each sampling event. Refer to Attachment A for instructions on completing this form, Attachment B for a complete list of the parameter codes and Attachment C for an example of a completed form.

1. EPA Program: TSCA CERCLA RCRA DW NPDES CAA
Other:_____
Projected Date(s) of Sampling_____
EPA Site Manager_____
EPA Case Team Members_____
_____
_____

Site Name_____
Site Location_____
Assigned Site Latitude/Longitude_____
CERCLA Site/Spill Identifier No. 01 _____ (Include Operable Unit)
Phase: ERA SA/SI pre-RI RI (phase I, etc.) FS RD RA post-RA
(circle one) Other:_____

2. QAPjP Title and Revision Date_____
_____
Approved by:_____ Date of Approval:_____
Title of Approving Official:_____ Organization*:_____
*If other than EPA, record date approval authority was delegated:_____

EPA Oversight Project (circle one) Y N
Confirmatory Analysis for Field Screening Y N
Are comparability criteria documented? Y N

Type of EPA Oversight (circle one) PRP or FF Other:_____
If EPA Oversight or Confirmatory: % splits_____

3. a.	Matrix Code[1]								
b.	Parameter Code[2]								
c.	Preservation Code[3]								
d.	Analytical Services Mechanism								
e.	No. of Sample Locations								
	Field QC:								
f.	Field Duplicate Pairs								
g.	Equipment Blanks								
h.	VOA Trip Blanks								
i.	Cooler Temperature Blanks								
j.	Bottle Blanks								
k.	Other:_____ _____								
l.	PES sent to Laboratory								
	Laboratory QC:								
m.	Reagent Blank								
n.	Duplicate								
o.	Matrix Spike								
p.	Matrix Spike Duplicate								
q.	Other:_____ _____								

4. Site Information
Site Dimensions_____
List all potentially contaminated matrices_____
Range of Depth to Groundwater_____
Soil Types: Surface Subsurface Other:_____
Sediment Types: Stream Pond Estuary Wetland Other:_____ Expected Soil/Sediment Moisture Content: High Low

When multiple matrices will be sampled during a sampling event, complete Sections 5-10 for each matrix. Matrix Code[1]_____

5. Data Use (circle all that apply) Site Investigation/Assessment PRP Determination Removal Actions
Nature and Extent of Contamination Human and/or Ecological Risk Assessment Remediation Alternatives
Engineering Design Remedial Action
Post-Remedial Action (quarterly monitoring) Other:_____

6. Summarize DQOs:_____

Complete Table if applicable

COCs	Action Levels	Analytical Method-Quantitation Limits

7. Sampling Method (circle technique) Bailer Low flow pump (Region I method: Yes No) Peristaltic Pump
Positive Displacement Pump Faucet or Spigot Other:_____
Split Spoon Dredge Trowel Other:_____

Sampling Procedures (SOP name, No., Rev. #, and date)_____
List Background Sample Locations_____
Circle: Grab or Composite
"Hot spots" sampled: Yes No

8. Field Data (circle) ORP pH Specific Conductance Dissolved O_2 Temperature Turbidity
Other:_____

9. Analytical Methods and Parameters

Method title/SOP name	Method/SOP Identification number	Revision Date	Target Parameters (VOA, SV, Pest/PCB, Metals, etc.)

10. Validation Criteria (circle one) 1. Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, III or IV
2. Other Approved Validation Criteria:_____
Validation Tier (circle one) I II III Partial Tier III: _____
Company/Organization Performing Data Validation_____Prime or Subcontractor (circle one)

11. Company Name_____ Contract Number_____
Contract Name (e.g. START, RACS, etc.)_____ Work Assignment No._____
Person Completing Form/Title_____ Date of DQO Summary Form Completion_____

Matrix Codes[1] - Refer to Attachment B, Part I
Parameter Codes[2] - Refer to Attachment B, Part II

Preservation Codes[3]

1. HCl to pH ≤ 2
2. HNO_3
3. $NaHSO_4$
4. H_2SO_4
5. Cool @ 4°C (± 2°)
6. NaOH
7. $K_2Cr_2O_7$
8. Freeze
9. Room Temperature (avoid excessive heat)
10. Other (Specify)
N. Not preserved

* - To supplement Matrix Codes and/or Parameter Codes contact the QA Unit

ATTACHMENT A

Guidance for Completion of DQO Summary Form

DISTRIBUTION:

1) Copies of completed DQO Summary Forms should be included in the QAPjP/SAP.

2) A. Copies of completed DQO Summary Forms for all CLP RAS work requested by EPA Site Managers, EPA contractors, including RACS, ROC, and START, and other Federal Agencies under Interagency Agreements, i.e., ACOE, and States under Cooperative Agreements should be sent with the quarterly sample projections to the Region I RSCC. Completed DQO Summary Forms for CLP RAS work must be received by the RSCC prior to the sampling event.

   B. Copies of completed DQO Summary Forms for non-CLP DAS work performed for EPA Site Managers and EPA contractors must be received by the Region I RSCC prior to the sampling event.

   C. DQO Summary Forms for non-CLP work performed under Interagency Agreements, Cooperative Agreements, and Grants must be completed prior to the sampling event, submitted to the "Authorizing Organization", as delegated by EPA, and included in the site documents.

3) Copies of completed DQO Summary Forms also must be included in the Data Validation Report or Tier I Validation Cover Letter (refer to Part I of the "Data Validation Manual" in the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses), December 1996, or most recent revision.

INSTRUCTIONS:

Note: A separate Form should be completed for each sampling event. For sampling events involving multiple environmental matrices, complete Sections 5-10 for each matrix and ensure that the two-letter matrix code is identified in Section 5. Enter the page number and total number of pages in the top right hand corner on the Form.

Section 1:

- Circle the appropriate EPA Program(s) involved in multi-media, multi-programmatic sampling events including, TSCA, CERCLA (i.e, Superfund), RCRA, DW (Drinking Water), NPDES, CAA (Clean Air), or fill in the blank for "Other:_____".
- List projected date(s) of sampling. The sampling dates should be inclusive of all matrices that will be sampled during this sampling event.
- Record the EPA Site Manager's name.
- List the names of the other EPA Case Team Members.
- Enter the site name. Use the NPL site name. If an NPL site name does not exist, then use the site name assigned under CERCLIS.
- Record the name of the city/town and State where the site is located in the "Site Location" field.
- Record the "Assigned Site Latitude/Longitude". Those numbers should be identical to those contained in CERCLIS database. Contact the EPA Site Manager to obtain correct Latitude/Longitude.
- Record the CERCLA site/spill identifier number, including the operable unit number. Contact the EPA Site Manager to obtain the correct identifier numbers.
- Circle the appropriate phase of Superfund site work (ERA: Environmental Risk Assessment, SA/SI: Site Assessment/Site Investigation, RI: Remedial Investigation, FS: Feasibility Study, RD: Remedial Design, RA: Remedial Assessment, post-RA: post-Remedial Assessment, i.e., quarterly monitoring). For non-Superfund site work, identify sampling event phase in the "Other" field.

Section 2:

- Record the complete title of the final QAPjP and revision date.
- Enter name of the Approving Official.
- Record date that the QAPjP was approved.
- Enter title of the Approving Official.
- Enter name of organization that has approval authority. This will be EPA, unless approval authority has been delegated by EPA to a State or other Federal Agency.
- If another organization has been delegated approval authority, then enter the date that EPA delegated approval authority (date of Quality Assurance Management Plan approval).
- Identify whether the project sampling event is an EPA oversight project, circle Yes or No.
- Indicate type of oversight by circling either Potentially Responsible Party (PRP) or Federal Facility (FF), or complete the blank for "Other:_____".
- Identify whether confirmatory sampling and analysis is being performed to verify field screening results, circle Yes or No.
- If EPA oversight or confirmatory analysis will be performed, record the percentage of split samples to be collected and analyzed.
- If EPA oversight or confirmatory analysis will be performed, identify whether comparability criteria are documented in the approved QAPjP or SAP, circle Yes or No.

Section 3:

a) List the two letter code for each matrix for samples that will be collected. Refer to Appendix B for a correct list of matrix codes. If a matrix does not have a corresponding code, then attach a description of the matrix to the DQO Summary Form.

Note: The matrix codes correspond to the matrix identifiers contained in the New England Sample Tracking System (NESTS) database. The current list of matrix codes are not intended to include all types of environmental matrices. However, they do represent groupings of similar-type matrices that potentially contain similar analytic interferences. For example, the matrix code GW (ground water) includes water from monitoring wells, supply wells, and public wells.

b) For each matrix, identify the analytical parameters for samples that will be collected by recording the appropriate parameter code. Refer to Appendix B for a current list of parameter codes. If an analytical parameter does not have a corresponding code, then the method title and/or SOP name, method and/or SOP identification number, and method and/or SOP revision date should be included and recorded in Section 9 of this Form.

Note: The parameter codes correspond to the analytical method parameters utilized in NESTS database. Appendix B includes a comprehensive list of analytical methods that have been used historically for Region I site work.

c) For each matrix and parameter, identify the preservation technique that will be used by recording the appropriate preservation code. Refer to the reverse side of this Form for a list of preservation codes.

d) Record the analytical service(s) mechanism that will be used for each matrix and parameter;

- CLP-RAS (CLP-Routine Analytical Service) This service may be utilized by EPA site managers, EPA contractors including, RACS, ROC, and START contracts. It may also be utilized under Interagency agreements, i.e., by the ACOE, and under Cooperative Agreements with the States.
- RACS-DAS (Remedial Alternative Contracting Strategy-Delivery of Analytical Services)
- ROC-DAS (Regional Oversight Contract-DAS)
- START-DAS (Superfund Technical Assessment and Remediation Contract-DAS)
- EPA-NERL (EPA-New England Regional Laboratory)

- Regional EPA-NE analytical contract
- State-Non-CLP
- Other Federal Agency Non-CLP
- If another analytical mechanism will be used, describe in detail on a separate page and attach to the Form.

e) Record the number of discrete locations that will be sampled for each parameter. The "No. of Sample Locations" count should include the site and background locations sampled.

- Record the number of each type of field QC sample that will be collected and sent to the laboratory for analysis for each matrix and parameter.

f) Record the number of Field duplicate sample pairs (which will equal "1" for each pair of field duplicates) that will be collected.
g) Enter the number of equipment/rinsate blanks.
h) Enter the number of VOA Trip blanks.
i) Enter the number of Cooler Temperature blanks that will be used.
j) Enter the number of Bottle Blanks that will be analyzed.
k) Describe any other field QC samples and the total number that were collected and that will be sent to the laboratory.
l) Enter the number of PESs that will be sent to the laboratory in accordance with EPA Region I Performance Evaluation Program Guidance, July 1996.

Note: The total of "e-l" equals the total number of samples sent to a laboratory for each matrix and parameter.

- Record the number of each type of laboratory QC sample that will be analyzed with the samples received.

m) Enter the minimum number of reagent blanks that will be analyzed.
n) Enter the number of laboratory Duplicates that will be analyzed.
o) Enter the number of matrix spikes that will be analyzed.
p) Enter the number of matrix spike duplicates that will be analyzed.
q) Describe any other laboratory QC samples and the total number that will be analyzed.

Section 4:

- Enter the approximate site dimensions with units.
- List all potentially contaminated matrices, regardless of whether or not they will be sampled during this sampling event.
- For well sampling, complete "Range of Depth to Groundwater" to ensure proper pump is utilized.
- For soil sampling, circle Surface or Subsurface or complete Other:_____.
- For sediment sampling, circle Stream, Pond, Estuary, Wetland, or complete Other:_____.
- For soil/sediment sampling, circle expected moisture content: High or Low. **Note: Analytical methods used for high moisture content samples should ensure that DQO-specified dry weight quantitation limits are achieved.**

Section 5:

When multiple matrices will be sampled during a sampling event, complete Sections 5-10 for each matrix and enter the Matrix Code.

- Identify the two-letter matrix code for which the information is provided in sections 5-10.
- Circle the potential uses for sample data such as, site investigation/assessment, PRP determination, removal actions, nature and extent of contamination, human and/or ecological risk assessment, remediation alternatives, engineering design, remedial action, post-remedial action, i.e., quarterly monitoring. A space is available for other potential uses of data.

Section 6:

- Briefly summarize the project DQOs. This section should describe the specific objectives of the sampling event, i.e., to identify health risks to children, ages 1-6, residing on the site who might be exposed to surface soils located in the area, or to characterize the extent of groundwater contamination. Identify the purpose of sampling, the decisions that will be made using the data, action level information, and any related information needed to identify that appropriate analytical and field sampling methods were chosen. Complete the table with the following information: contaminants of concern (COC), COC action levels and analytical method quantitation limits for each COC. **Note: Since this information will be used by data validators to identify potential data usability issues for the user, it is imperative that it is clear and concise.**

Section 7:

- Circle applicable sampling technique(s) used and/or complete "Other" to describe an innovative sampling technique or one that is not listed.
- Identify the SOPs that will be utilized for sample collection. Include SOP name, identification number and revision number and/or date.
- Record the discrete Background sample station location number(s) that will be sampled.
- Circle if samples will be "grab" or "composite".
- To indicate potential "Hot spots" on site, circle Yes or No.

Section 8:

- Identify the field data that will be collected including, ORP, pH, specific conductance, dissolved O_2, temperature, and turbidity. A space is available to indicate other field testing that will be performed.

Section 9:

- If an analytical method does not have a Parameter code (required information in Section 3), then the method title and/or SOP name, method and/or SOP identification number, and method and/or SOP revision date should be included. Attach a separate page if additional space is needed.
- Record the specific parameters required for analysis.

Section 10: **In accordance with Region I QA policy, all data must be validated in accordance with the most recent revision of Part I the "Data Validation Manual: The Data Quality System" of the Region I, EPA-NE Data Validation Functional Guidelines of Evaluating Environmental Analyses.**

- Circle the data validation criteria required by the QAPjP and/or SAP. In most cases, the QAPjP and/or SAP should cite the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines of Evaluating Environmental Analyses and identify the applicable Functional Guideline criteria procedures that will be used to validate the data: Part II-Volatile/Semivolatile Data Validation Functional Guidelines, Part III-Pesticide/PCB Data Validation Functional Guidelines, and Part IV-Inorganic Data Validation Functional Guidelines.
  If modified criteria or alternate data validation criteria will be utilized, the modified or alternate criteria must be documented in an approved QAPjP and/or SAP as stipulated in Part I, the "Data Validation Manual: The Data Quality System", December 1996 revision of the Region I, EPA-NE Data Validation Functional Guidelines of Evaluating Environmental Analyses, December 1996 revision.
- Circle the Region I Validation Tier that will be used.
- If a partial Tier III data validation is required, then the subset receiving a partial Tier III should be specified (e.g., benzene, VOA, etc).
- Identify the company performing the data validation. Circle either Prime or Subcontractor.

Section 11:

- Record the field sampling contractor company/organization name
- Contract number
- Name of contract
- Work assignment number
- Name and title of person completing Form
- Completion date of the DQO Summary Form

ATTACHMENT B - PART I

Matrix Codes[1]

Aqueous:
DW - Drinking Water
GW - Ground Water
LE - Leachate (includes porewater)
SW - Surface Water
WW - Waste Water (includes scrubber blowdown)

Solid:
SE - Sediment (includes tidal sediments)
SO - Soil

Biota:
BD - Bird Tissue
CF - Crawfish Tissue
FI - Fish (includes whole fish)
MU - Mussel (includes clam, quahog, and oyster tissue)
OF - Offal
PL - Plant
FF - Fish Fillet

Wastes:
AS - Ash (includes incinerator ash and boiler aggregate)
DU - Dust (includes concrete dust and fines)
OI - Oil (includes waste oil)
SL - Sludge
WD - Wood (includes chips, cuttings, and drillings)
WT - Waste (includes both solids and liquids)
ST - Still Bottoms

Miscellaneous:
AR - Air Samples
DN - DNAPLs
LN - LNAPLs
WI - Wipe Samples
PC - Paint Chips
CT - Concrete

ATTACHMENT B - PART II

PARAMETER CODES

PARAMETER CODE/METHOD IDENTIFICATION NUMBER	METHOD TITLE	REFERENCE	PARAMETER NAME
OLM03.1F	USEPA CLP Statement of Work for Organics Analysis - OLM03.1	1	Full organics (VOA, SV, P/P) CLP SOW Organic Analysis
OLM03.1P	USEPA CLP Statement of Work for Organics Analysis - OLM03.1	1	Pesticide/Aroclors Analysis CLP SOW Organic Analysis
OLM03.1S	USEPA CLP Statement of Work for Organics Analysis - OLM03.1	1	Semivolatile Organics Analysis CLP SOW Organic Analysis
OLM03.1V	USEPA CLP Statement of Work for Organics Analysis - OLM03.1	1	Volatile Organics Analysis CLP SOW Organic Analysis
1003	Halogenated Hydrocarbons	2	NIOSH 1003 Volatile on Charcoal Tubes
12/90-DI	USEPA CLP Statement of Work for Analysis of Polychlorinated Dibenzo-p-Dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF), DFLM1.0, Rev. 12/90	3	12/90 SOW Dioxin/Furan Analysis
130.1	Hardness, Total (mg/L) as $CaCO_3$, Colorimetric, Automated EDTA	4	Hardness-Colorimetric, Automated EDTA
130.2	Hardness, Total (mg/L) as $CaCO_3$, Titrimetric, EDTA	4	Hardness-Titrimetric, EDTA
13112007	Toxicity Characteristic Leaching Procedure and Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry	5 & 7	TCLP Extraction-Metals Analysis
13113.1F	Toxicity Characteristic Leaching Procedure and USEPA CLP Statement of Work for Organics Analysis - OLM03.1	5 & 1	TCLP Extraction-Full Organics Volatile, Semivolatile, Pesticide/PCB Analysis
13113.1P	Toxicity Characteristic Leaching Procedure and USEPA CLP Statement of Work for Organics Analysis - OLM03.1	5 & 1	TCLP Extraction-Pesticide/PCB Analysis
13113.1S	Toxicity Characteristic Leaching Procedure and USEPA CLP Statement of Work for Organics Analysis - OLM03.1	5 & 1	TCLP Extraction-Semivolatile Analysis
13113.1V	Toxicity Characteristic Leaching Procedure and USEPA CLP Statement of Work for Organics Analysis - OLM03.1	5 & 1	TCLP Extraction-Volatile Analysis
13118000	Toxicity Characteristic Leaching Procedure and Determination of Organic Analytes by Gas Chromatography	5	TCLP Extraction-Full Organics
13118080	Toxicity Characteristic Leaching Procedure and Determination of Organochlorine Pesticides and PCBs by Gas Chromatography	5	TCLP Extraction-Pesticide/PCB Analysis
13118240	Toxicity Characteristic Leaching Procedure and Determination of Volatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS)	5	TCLP Extraction-Volatile Analysis
13118270	Toxicity Characteristic Leaching Procedure and Determination of Semivolatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique	5	TCLP Extraction-Semivolatile Analysis

ATTACHMENT B - PART II

PARAMETER CODES

PARAMETER CODE/METHOD IDENTIFICATION NUMBER	METHOD TITLE	REFERENCE	PARAMETER NAME
160.1	Residue, Filterable, Gravimetric, Dried at 180 °C	4	Total Dissolved Solids (TDS)
160.2	Residue, Non-filterable, Gravimetric, Dried at 103-105 °C	4	Total Suspended Solids (TSS)
160.3	Residue, Total, Gravimetric, Dried at 103-105 °C	4	Total Solids
1613	Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilutions HRGC/HRMS	6	Dioxin/Furan High Resolution Analysis
200.7	Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma - Atomic Emission Spectrometry (Rev.4.4, 1994)	7	ICP Metals Analysis-Full List
200.7XX	Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma - Atomic Emission Spectrometry (Rev.4.4, 1994)	7	ICP Metals Analysis-XX Specific Metals
200.9/CD	Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry (Rev. 2.2, 1994)	7	Graphite Furnace-Cadmium
200.9/SB	Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry	7	Graphite Furnace-Antimony
200.9AS	Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry	7	Graphite Furnace-Arsenic
204.2/SB	Antimony AA, Furnace	4	Graphite Furnace-Antimony
206.2	Arsenic AA, Furnace	4	Graphite Furnace-Arsenic
213.2/CD	Cadmium AA, Furnace	4	Graphite Furnace-Cadmium
2320.B	Alkalinity, Titration Method	8	Titration Method-Alkalinity
2340B	Hardness by Calculation	8	Hardness-Calculation
2340C	Hardness, EDTA Titrimetric Method	8	Hardness Titrimetric, EDTA
2540B	Total Solids Dried at 103-105 °C	8	Total Solids
2540C	Total Dissolved Solids Dried at 180 °C	8	Total Dissolved Solids (TDS)
2540D	Total Suspended Solids Dried at 103-105 °C	8	Total Suspended Solids (TSS)
300.0C1	Ion Chromatography		Determination Inorganic Anions in AQ by IC
300.0F	Ion Chromatography		Ion Chrom.-Fluoride
300.0N03	Ion Chromatography		Ion Chrom.-Nitrate
310.1	Alkalinity Titrimetric (pH 4.5)	4	Titrimetric Alkalinity

ATTACHMENT B - PART II

PARAMETER CODES

PARAMETER CODE/METHOD IDENTIFICATION NUMBER	METHOD TITLE	REFERENCE	PARAMETER NAME
310.2	Alkalinity, Colorimetric, Automated, Methyl Orange	4	Colorimetric-Alkalinity
3113B/AS	Metals by Electrothermal Atomic Absorption Spectrometry	8	Graphite Furnace-Arsenic
3113B/CD	Metals by Electrothermal Atomic Absorption Spectrometry	8	Graphite Furnace-Cadmium
3113B/SB	Metals by Electrothermal Atomic Absorption Spectrometry	8	Graphite Furnace-Antimony
325.2	Chloride, Colorimetric, Automated Ferricyanide AA II	4	Colorimetric-Chloride
325.3	Chloride, Titrimetric, Mercuric Nitrate	4	Titrimetric-Chloride
335.2	Cyanide, Total, Titrimetric; Spectrophotometric	4	Titrimetric-Total Cyanide
340.2	Fluoride, Potentiometric, Ion Selective Electrode	4	Electrode-Fluoride
350.1	Nitrogen, Ammonia, Colorimetric, Automated Phenate	4	Colorimetric-Ammonia
350.2	Nitrogen, Ammonia, Colorimetric; Titrimetric; Potentiometric-Distillation Procedure	4	Colorimetric, Titrimetric, Electrode-Dist.-Ammonia
350.3	Nitrogen, Ammonia, Potentiometric, Ion Selective Electrode	4	Electrode-Ammonia
351.2	Nitrogen, Kjeldahl, Total, Colorimetric, Semi-Automated Block Digester, AA II	4	Colorimetric Semi-Auto-Total Kjeldahl N (TKN)
351.3	Nitrogen, Kjeldahl, Total, Colorimetric; Titrimetric; Potentiometric	4	Colorimetric, Titrimetric, Electrode-Total Kjeldahl N (TKN)
352.1	Nitrogen, Nitrate, Colorimetric, Brucine	4	Colorimetric-Nitrate
353.1	Nitrogen, Nitrate-Nitrite, Colorimetric, Automated, Hydrazine Reduction	4	Colorimetric, Auto., Hydr-Red.-Nitrate
353.2	Nitrogen, Nitrate-Nitrite, Colorimetric, Automated, Cadmium Reduction	4	Colorimetric, Auto., Cd-Red.-Nitrate
353.3	Nitrogen, Nitrate-Nitrite, Spectrophotometric, Cadmium Reduction	4	Spectro., Cd-Red-Nitrate
354.1	Nitrogen, Nitrite, Spectrophotometric	4	Spectrophotometric-Nitrite
365.1	Phosphorus, All Forms, Colorimetric, Automated, Ascorbic Acid	4	Colorimetric, Auto, Ascorbic Acid-Phosphorus
365.2	Phosphorus, All Forms, Colorimetric, Ascorbic Acid, Single Reagent	4	Colorimetric, Ascorbic Acid, 1 Reag-Phosphorus
365.3	Phosphorus, All Forms, Colorimetric, Ascorbic Acid, Two Reagent	4	Colorimetric, Ascorbic Acid, 2 Reag-Phosphorus
365.4	Phosphorus, Total, Colorimetric, Automated, Block Digestor AA II	4	Colorimetric, Auto.-Phosphorus
370.1	Silica, Dissolved, Colorimetric	4	Colorimetric-Silica
375.1	Sulfate, Colorimetric, Automated, Chloranilate	4	Colorimetric, Automated-Sulfate

ATTACHMENT B - PART II

PARAMETER CODES

PARAMETER CODE/METHOD IDENTIFICATION NUMBER	METHOD TITLE	REFERENCE	PARAMETER NAME
375.3	Sulfate, Gravimetric	4	Gravimetric-Sulfate
375.4	Sulfate, Turbidimetric	4	Turbidimetric-Sulfate
376.1	Sulfide, Titrimetric, Iodine	4	Titrimetric-Sulfide
376.2	Sulfide, Colorimetric, Methylene Blue	4	Colorimetric-Sulfide
403	Bicarbonate		Bicarbonate
405.1	Biochemical Oxygen Demand BOD (5 day, 20°C)	4	5 Days 20°C -BOD
410.1	Chemical Oxygen Demand, Titrimetric, Mid-Level	4	Titrimetric-COD Mid. Level
410.2	Chemical Oxygen Demand, Titrimetric, Low Level	4	Titrimetric-COD Low Level
410.3	Chemical Oxygen Demand, Titrimetric, High Level for Saline Waters	4	Titrimetric-COD High Level
410.4	Chemical Oxygen Demand, Colorimetric, Automated; Manual	4	Spectrophotometric-COD Manual/Auto
4110	Determination of Anions by Ion Chromatography	8	Anions
413.1	Oil and Grease, Total Recoverable, Gravimetric, Separatory Funnel Extraction	4	Gravimetric-Oil & Grease
413.2	Oil and Grease, Total Recoverable, Spectrophotometric, Infrared	4	Oil and Grease (O & G) - IR Spec.
415.1	Organic Carbon, Total, Combustion or Oxidation	4	Combustion or Oxidation-TOC
415.2	Organic Carbon, Total, UV Promoted, Persulfate Oxidation		TOC-Low Level, UV Promoted
418.1	Petroleum Hydrocarbons, Total Recoverable, Spectrophotometric, Infrared	4	IR Spec-TPH, Petroleum Hydrocarbons
418.1TPH	Petroleum Hydrocarbons, Total Recoverable, Spectrophotometric, Infrared	4	Total Petroleum Hydrocarbons
4500-P/E	Phosphorus, Ascorbic Acid Method	8	Ascorbic Acid-Phosphorus
4500-P/F	Phosphorus, Automated Ascorbic Acid Reduction Method	8	Auto. Ascorbic Acid-Phosphorus
4500F/C	Fluoride, Ion-Selective Electrode Method	8	Electrode-Fluoride
4500NO2B	Nitrogen (Nitrite) Colorimetric Method	8	Colorimetric-Nitrite
4500NO3E	Nitrogen (Nitrate) Cadmium Reduction Method	8	Cadmium Red. Manual-Nitrate
4500NO3F	Nitrogen (Nitrate) Automated Reduction Method	8	Cadmium Red. Auto.-Nitrate
4500NO3H	Nitrogen (Nitrate) Automated Hydrazine Reduction	8	Automated Hydrazine-Nitrate

ATTACHMENT B - PART II

PARAMETER CODES

PARAMETER CODE/METHOD IDENTIFICATION NUMBER	METHOD TITLE	REFERENCE	PARAMETER NAME
4500S/D	Sulfide, Methylene Blue Method	8	Methylene Blue Sulfide
4500S/F	Sulfide, Iodometric Method	8	Iodometric-Sulfide
4500S04C	Sulfate, Gravimetric Method with Ignition of Residue	8	Grav.+Ignition-Sulfate
4500S04D	Sulfate, Gravimetric Method with Drying of Residue	8	Grav.+Drying-Sulfate
4500SI/D	Silica, Molybdosilicate Method	8	Molybdosilicate-Silica
504.1	1,2-Dibromethane (EDB), 1,2-Dibromo-3-chloropropane (DBCP), and 1,2,3-Trichloropropane (123 TCP) in Water by Microextraction and Gas Chromatography (Rev. 1.1, 1995)	9	EDB, DBCP & 123TCP, Microextraction & GC
5210/B	Biochemical Oxygen Demand (BOD), 5 Day BOD Test	8	5 Day-BOD
5220/C	Chemical Oxygen Demand (COD), Closed Reflux, Titrimetric Method	8	Titrimetric-COD Mid Level
5220/D	Chemical Oxygen Demand (COD), Closed Reflux, Colorimetric Method	8	Spectrophotometric-COD Manual/Auto
524.2	Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry (Rev. 4.0, 1992)	9	Measurement of Purgeable Organic Compounds in Water - Capillary Column by GC/MS
524.2+	Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry (Rev. 4.0, 1992)	9	524.2 Plus Additional Compounds
525.2	Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (Rev. 2.0, 1995)	9	Determination of Organic Compounds in DW by Liquid Solid Extraction Capillary Column by GC/MS
5310/B	Total Organic Carbon (TOC) Combustion-Infrared Method	8	Combustion-Infrared-TOC
5310/C	Total Organic Carbon (TOC) Persulfate-Ultraviolet Oxidation Method	8	Persulfate-UV Oxidation-TOC
5310/D	Total Organic Carbon (TOC) Wet-Oxidation Method	8	Wet-Oxidation-TOC
551.1	Detection of Chlorination Disinfection Byproducts and Chlorinated Solvents, and Halogenated Pesticides/Herbicides in Drinking Water by Liquid/Liquid Extraction and Gas Chromatography with Electron-Capture Detection	9	Det. Chloro. Disin. Byprods, Chloro Solv. by LL&GC
5520/B	Oil and Grease Partition-Gravimetric Method	8	Gravimetric-Oil & Grease
5520/C&F	Oil and Grease Partition-Infrared Method and Hydrocarbons	8	IR Spec-TPH, Petroleum, Hydrocarbon
601	Purgeable Halocarbons (Trap-GC/Hall Detector-Electrolytic Conductivity Detector)	10	Purgeable Halocarbons Trap-GC/ELCD
602	Purgeable Aromatics (Trap-GC/PID)	10	Purgeable Aromatics Trap-GC/PID

ATTACHMENT B - PART II

PARAMETER CODES

PARAMETER CODE/METHOD IDENTIFICATION NUMBER	METHOD TITLE	REFERENCE	PARAMETER NAME
608	Organochlorine Pesticides and PCBs by (GC/ECD)	10	Organochlorine Pest PCB-GC/ECD
624	Purgeables (Trap-GC/MS)	10	Purgeable Trap-GC/MS
625	Base/Neutrals and Acids (GC/MS)	10	Base/Neutrals&Acids Extr. GC/MS
8015A	Nonhalogenated Volatile Organics by Gas Chromatography	5	Nonhalogenated Volatile Org GC
8080A	Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography (Rev. 1, 1994)	5	Organochlorine Pest.&PCB by GC/ECD
8240B	Volatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS) (Rev.2, 1994)	5	Volatile Organic Compounds by GC/MS
8270B	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique (Rev. 2, 1994)	5	Semivolatile Organic Compounds by GC/MS
8290	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High - Resolution Mass Spectrometry (HRGC/HRMS) (Rev.0, 1994)	5	PCDDS & PCDFS by HRGC/MS
ASTM2974	Standard Test Method for Moisture, Ash and Organic Matter of Peat and Other Organic Matter	11	TCOC - TOT Combustible Org Content
ASTMD422	Standard Test Method for Particle-Size Analysis of Soils	11	Grain Size Analysis
ILM040CN	USEPA CLP SOW for Inorganics Analysis - ILM04.0	12	Cyanide Inorganic CLP SOW
ILM040MT	USEPA CLP SOW for Inorganics Analysis - ILM04.0	12	Metals (no CN) Inorganic CLP SOW
ILM040TL	USEPA CLP SOW for Inorganics Analysis - ILM04.0	12	Metals & Cyanide Inorganic CLP SOW
TO-1	Determination of Volatile Organic Compounds in Ambient Air using Tenax Adsorption and GC/MS	13	VOC-AIR, Tenax Tubes
TO-14	Determination of Volatile Organic Compounds in Ambient Air Using Summa Passivated Canister Sampling and GC Analysis	13	VOC-AIR, Summa Canisters
TO-2	Determination of Volatile Organic compounds in Ambient Air using Carbon Molecular Sieve Adsorption and GC/MS	13	VOC-AIR, Carbon Molecular Sieve

NOTE: The method number is incorporated into the Parameter Code

REFERENCES:

1. USEPA CLP Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLM03.1, August 1994
2. NIOSH Manual of Analytical Methods (Second, Part I), NIOSH Monitoring Methods, Volume I.

ATTACHMENT B - PART II

PARAMETER CODES

3. USEPA CLP Statement of Work for Analysis of Polychlorinated Dibenzo-p-Dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF), DFLM01.0/DFLM01.1 - Rev. 12/90 and Rev. 9/91.
4. Methods for Chemical Analysis of Water and Wastes, Environmental Protection Agency, EPA-600/4-79-020
5. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, July 1992 and Updates
6. Method 1613: Tetra- Through Octa- Chlorinated Dioxins and Furans by Isotope Dilutions HRGC/HRMS, EPA 821-B-94-005, October 1994, Rev. B.
7. Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991, and Supplement I, EPA-600/R-94/111, May 1994.
8. Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995
9. Methods for the Determination of Organic Compounds in Drinking Water, December 1988, EPA/600/4-88/039 and Updates
10. Code of Federal Regulations, 40 CFR, Part 136, App. A
11. American Society for Testing and Materials
12. USEPA CLP Statement of Work for Inorganics Analysis, Multi-media, Multi-concentration, ILM04.0
13. EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-600/4-84-041, May, 1987.

REGION I, EPA-NE ORGANIC REGIONAL DATA ASSESSMENT (ORDA)*

CASE #: _____ SITE NAME: _____

LAB NAME: _____ # OF SAMPLES/MATRIX: _____

SDG #: _____ VALIDATION CONTRACTOR: _____

SOW #/CONTRACT #: _____ VALIDATOR'S NAME: _____

EPA-NE DV TIER LEVEL: _____ DATE DP REC'D BY EPA-NE: _____

TPO/PO: **ACTION ____ FYI ____ DV COMPLETION DATE: _____

ANALYTICAL DATA QUALITY SUMMARY

	VOA	SV	Pest/PCB
1. Preservation and Contractual Holding Times			
2. GC/MS / GC/ECD Instrument Performance Check			
3. Initial Calibration			
4. Continuing Calibration			
5. Blanks			
6. Surrogate Compounds			
7. Internal Standards			
8. Matrix Spike/Matrix Spike Duplicate			
9. Sensitivity Check			
10. PE Samples-Accuracy Check			
11. Target Compound Identification			
12. Compound Quantitation and Reported QLs			
13. Tentatively Identified Compounds			
14. Semivolatile Cleanup/Pesticide/PCB Cleanup			
15. Data Completeness			
16. Overall Evaluation of Data			

o = Data had no problems or were qualified due to minor contractual problems.
m = Data were qualified due to major contractual problems.
z = Data were rejected as unusable due major contractual problems.

ACTION ITEMS: (z items)_____

AREAS OF CONCERN: (m items)_____

COMMENTS: _____

*This form assesses the analytical data quality in terms of contractual compliance only. It does not assess sampling errors and/or non-contractual analytical issues that affect data quality.

**Check "ACTION" only if contractual defects resulted in reduced payment/data rejection recommendations.

Validator: _____ Date: _____

INSTRUCTIONS ON REVERSE SIDE

GUIDANCE FOR COMPLETING THE ORDA

The ORDA form provides the laboratory's CLP-TPO and other contract management personnel with an overview of the contractual analysis and reporting deficiencies found in an analytical data package and identifies those contractual deficiencies that resulted in reduced payment/data rejection recommendations/actions. The ORDA form is used to summarize analytical data quality only in terms of contractual compliance. Sampling errors and non-contractual analytical errors that affect data quality are not summarized on this form, but rather are documented in the Tier I Validation Cover Letter and Tier II/III Data Validation Reports. For instance, if the sampler did not ship the samples until after the holding time had expired, a notation would not be made on the ORDA form since the laboratory is not responsible for the sampler's actions.

The ORDA form should be completed as follows:

1. Fill in all of the header information (with the exception of the TPO Action/FYI field): Case Number, Site Name, Laboratory Name, number and matrix of samples in the data package, SDG Number, Validation Contractor, SOW#/Contract#, Data Validator's Name, EPA-NE Data Validation Tier Level (i.e., I, II, III or partial II/III), Date the Data Package was received by EPA-NE, and the Data Validation Completion Date.

2. Summarize the contractual problems discovered during data validation by fraction and by evaluation criteria in the "Analytical Data Summary " table, and in the "Action Items" and "Areas of Concern" sections as described in items 3 through 6 below. Use the Data Validation Memoranda as a guide when completing the ORDA form.

3. The following qualifiers must be utilized to document contractual problems on the ORDA forms.

o	=	Data had no problems or were qualified due to minor contractual problems
m	=	Data were qualified due to major contractual problems
z	=	Data were rejected as unusable due to major contractual problems

4. If the data were acceptable, or were qualified due to minor contractual problems, enter the qualifier "o" into the appropriate column (fraction) and row (evaluation criteria). No further documentation is necessary on the ORDA form. An example of a minor problem would be a semivolatile compound that slightly exceeded the SOW-specified %RSD initial calibration criterion.

5. If the data were qualified due to major contractual problems, enter the qualifier "m" into the appropriate column (fraction) and row (evaluation criteria). Use a different superscript (m^1, m^2, etc.) for each major contractual problem identified and provide a brief description of each major problem in the "Areas of Concern" section. An example of a major contractual problem resulting in data qualification would be a semivolatile internal standard that had extremely low area counts (below the lower limit of the SOW-specified acceptance criterion) and reanalysis was not performed.

6. If the data were rejected as unusable due to major contractual problems, enter the qualifier "z" in the appropriate column (fraction) and row (evaluation criteria). Use a different superscript (z^1, z^2, etc.) for each major contractual problem identified and provide a brief description of each major problem in the "Action Items" section. An example of a major contractual problem resulting in data rejection would be contractual holding time criteria that were exceeded for volatiles.

7. Complete the TPO Action/FYI field using the information contained in the "Action Items" and "Areas of Concern" sections. TPO Action should be indicated with a check mark (√) in the space following "Action" only if the contractual defects resulted in reduced payment or data rejection. If no TPO Actions are indicated, then a check mark (√) should be placed in the space following "FYI".

8. The validator who completed the ORDA form must sign his/her name in the "Validator" field and enter the ORDA completion date in the "Date" field.

For hardcopy of Telephone Log or
Regional/Laboratory Communication Form contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

Site Name _____
Reference No. _____

REGION I ORGANIC DATA VALIDATION

The following data package has been validated:

Lab Name _____
Case/Project No. _____
SDG No. _____
No. of Samples/Matrix _____

SOW/Method No. _____
Sampling Date(s) _____
Shipping Date(s) _____
Date Rec'd by lab _____

Traffic Report Sample Nos. _____
_____
_____
Trip Blank No. _____
Equipment Blank No. _____
Bottle Blank No. _____
Field Duplicate Nos. _____

PES Nos. _____

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, revision _____ was used to evaluate the data and/or approved modifications to the EPA-NE Functional Guidelines were used to evaluate the data and are attached to this cover page: (attach modified criteria from EPA approved QAPjP or amendment to QAPjP).

A Tier II or Tier III evaluation was used to validate the data (circle one). If a Tier II validation with a partial Tier III was used, then identify samples, parameters, etc. that received partial Tier III validation
_____
.
The data were evaluated based upon the following parameters:

- Overall Evaluation of Data
- Data Completeness (CSF Audit - Tier I)
- Preservation & Technical Holding Times
- GC/MS & GC/ECD Instrument Performance Check
- Initial & Continuing Calibrations
- Blanks
- Surrogate Compounds
- Internal Standards
- Matrix Spike/Matrix Spike Duplicate
- Field Duplicates
- Sensitivity Check
- PE Samples/Accuracy Check
- Target Compound Identification
- Compound Quantitation and Reported Quantitation Limits
- TICs
- Semivolatile and Pesticide/PCB Cleanup
- System Performance

Region I Definitions and Qualifiers:

A - Acceptable Data
J - Numerical value associated with compound is an estimated quantity.
R - The data are rejected as unusable. The R replaces the numerical value or sample quantitation limit.
U - Compound not detected at that numerical sample quantitation limit.
UJ - The sample quantitation limit is an estimated quantity.
TB, BB, EB - Compound detected in aqueous trip blank, aqueous bottle blank, or aqueous equipment blank associated with soil/sediment samples.

Validator's Name _____Company Name_____Phone Number _____

Date Validation Started _____ Date Validation Completed _____

EPA-NE
Data Validation Worksheet Cover Page - Page 2

Check if all criteria are met and no hard copy worksheet provided. Indicate NA if worksheet is not applicable to analytical method. Note: there is no standard worksheet for System Performance, however, the validator must document all system performance issues in the Data Validation Memorandum.

VOA/SV worksheets:

VOA/SV-Pest/PCB	COMPLETE SDG FILE (CSF) AUDIT	_____
VOA/SV-Pest/PCB-I	PRESERVATION AND HOLDING TIMES	_____
VOA/SV-II	GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING)	_____
VOA/SV-III	INITIAL CALIBRATION	_____
VOA/SV-IV	CONTINUING CALIBRATION	_____
VOA/SV-Pest/PCB-V-A	BLANK ANALYSIS	_____
VOA/SV-Pest/PCB-V-B	BLANK ANALYSIS	_____
VOA-VI	VOA SURROGATE SPIKE RECOVERIES	_____
SV-VI	SV SURROGATE SPIKE RECOVERIES	_____
VOA/SV-VII	INTERNAL STANDARD PERFORMANCE	_____
VOA/SV-Pest/PCB-VIII	MATRIX SPIKE/MATRIX SPIKE DUPLICATE	_____
VOA/SV-Pest/PCB-IX	FIELD DUPLICATE PRECISION	_____
VOA/SV-Pest/PCB-X	SENSITIVITY CHECK	_____
VOA/SV-Pest/PCB-XI	ACCURACY CHECK	_____
VOA/SV-Pest/PCB-XII	TARGET COMPOUND IDENTIFICATION	_____
VOA/SV-Pest/PCB-XIII	SAMPLE QUANTITATION	_____
VOA/SV-XIV	TENTATIVELY IDENTIFIED COMPOUNDS	_____
VOA/SV-XV	SEMIVOLATILE CLEANUP	_____
TABLE II-WORKSHEET	OVERALL EVALUATION OF DATA	_____

Pest/PCB worksheets:

VOA/SV-Pest/PCB	COMPLETE SDG FILE (CSF) AUDIT	_____
VOA/SV-Pest/PCB-I	PRESERVATION AND HOLDING TIMES	_____
Pest/PCB-IIA	GC/ECD INSTRUMENT PERFORMANCE CHECK-RESOLUTION	_____
Pest/PCB-IIB	GC/ECD INSTRUMENT PERFORMANCE CHECK-RETENTION TIMES	_____
Pest/PCB-IIC	GC/ECD INSTRUMENT PERFORMANCE CHECK-ACCURACY CHECK OF INITIAL CALIBRATION	_____
Pest/PCB-IID	GC/ECD INSTRUMENT PERFORMANCE CHECK-PESTICIDE DEGRADATION	_____
Pest/PCB-III	INITIAL CALIBRATION	_____
Pest/PCB-IV	CONTINUING CALIBRATION	_____
VOA/SV-Pest/PCB-V-A	BLANK ANALYSIS	_____
VOA/SV-Pest/PCB-V-B	BLANK ANALYSIS	_____
Pest/PCB-VI	SURROGATE COMPOUNDS: SPIKE RECOVERIES AND RETENTION TIME SHIFT	_____
Pest/PCB-VII	PESTICIDE CLEANUP	_____
VOA/SV-Pest/PCB-VIII	MATRIX SPIKE/MATRIX SPIKE DUPLICATE	_____
VOA/SV-Pest/PCB-IX	FIELD DUPLICATE PRECISION	_____
VOA/SV-Pest/PCB-X	SENSITIVITY CHECK	_____
VOA/SV-Pest/PCB-XI	ACCURACY CHECK	_____
Pest/PCB-XII	COMPOUND IDENTIFICATION	_____
VOA/SV-Pest/PCB-XIII	SAMPLE QUANTITATION	_____
TABLE II-WORKSHEET	OVERALL EVALUATION OF DATA	_____

I certify that all criteria were met for the worksheets checked above.

Signature:_____ Name:_____

Date: _____

The data validator generates a Data Validation Report, applicable to Data Validation Tiers II and III, that consists of the following components in the order specified below: (Refer to Section 11 for a description of each of the Data Validation Report components).

1. Organic Regional Data Assessment/Inorganic Regional Data Assessment (ORDA/IRDA) Form
2. Data Validation Memorandum
   a. Narrative
   b. Table I-Qualifier Recommendation Summary Table
   c. Table II-Overall Evaluation of Data
   d. Table III-Tentatively Identified Compounds
   e. Data Summary Tables
3. Standard Data Validation Worksheets
   a. Manual
   b. Automated Data Review Reports (i.e., CADRE)
4. Support Documentation
   a. Copy of non-CLP analytical method, e.g., DAS methods, modified EPA methods
   b. Copies of PES Score Reports/Vendor PES QC Acceptance Limits
   c. Copies of Telephone Logs/Communication Forms for:
      - RSCC communications
      - Requests for laboratory data resubmissions/clarifications
      - Communications with samplers resolving sampling problems
      - Communications with TPO/Lead Chemist to report contractually-deficient data for rejection/reduced payment
      - Communications with EPA Site Manager concerning possible data rejection
      - EPA Site Manager authorization for alternate DV tier
   d. Copies of data supporting recommendations for reduced payment resulting from CSF Audit and/or PE sample result evaluation
   e. Original data to support recommendations for data rejection/non-payment identified from Tier II or Tier III data validation
   f. Copies of field sampling notes and/or field report supplied by field sampler
   g. Copies of EPA-approved amendments to QAPjP and/or SAP describing modified criteria to be used for validating site data
5. CSF Completeness Evidence Audit
6. DQO Summary Form

The data validator is responsible for implementing all corrective actions required by the contractor Lead Chemist in response to EPA-NE data validation oversight findings.

Overall Evaluation of Data - Data Validation Memorandum - Table II

VOLATILE ORGANICS					
DQO (list all DQOs)	Sampling and/or Analytical Method Appropriate Yes or No	Measurement Error		Sampling Variability**	Potential Usability Issues
		Analytical Error	Sampling Error*		

* The evaluation of "sampling error" cannot be completely assessed in data validation.

** Sampling variability is not assessed in data validation.

Validator: _____ Date: _____

Overall Evaluation of Data - Data Validation Memorandum - Table II

SEMIVOLATILE ORGANICS					
DQO (list all DQOs)	Sampling and/or Analytical Method Appropriate Yes or No	Measurement Error		Sampling Variability**	Potential Usability Issues
		Analytical Error	Sampling Error*		

* The evaluation of "sampling error" cannot be completely assessed in data validation.

** Sampling variability is not assessed in data validation.

Validator: _____ Date: _____

COMPLETE SDG FILE (CSF) AUDIT

Organic Fractions:_____

Missing Information	Date Lab Contacted	Date Received

Validator: _____Date: _____

Sampler:_____ Company:_____ Contacted: Yes No Date:_____

I. PRESERVATION AND HOLDING TIMES - Circle sample numbers with exceeded technical holding times or omitted preservation.
List all required preservation codes and circle omitted preservation codes.
Circle all exceeded technical holding times.
Identify extraction technique after "# of Days"/(*Extraction Code).

Sample No. (TR No.)	Matrix	Pres. Code	Date Sampled	VOA			BNA					PEST/PCB				
				Date Analyzed	# of Days from Samp. to Anal.	Action	Date Extracted	# of Days from Samp. to Extr./(*)	Date Analyzed	# of Days from Extr. to Anal.	Action	Date Extracted	# of Days from Samp. to Extr./(*)	Date Analyzed	# of Days from Extr. to Anal.	Action

Preservation Code:
1. Cool @ 4°C (± 2°)
2. Preserve with HCl to at least pH 2
3. Protect from light
4. Freeze
5. Room Temperature (Avoid excessive heat)

(*Extraction Code:)
L/L - Liquid/Liquid
SON - Sonication
SEP - Separatory Funnel
SOX - Soxhlet
SPE - Solid Phase Extraction

Action Code:
J - Estimate (J) Detected Values
UJ - Estimate (UJ) Non-Detected Values
R - Reject (R) Non-Detected Values

Validator: _____ Date: _____

II. GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING)

List all Instrument Performance Checks that are outside method QC tuning acceptance criteria.

Volatile Instrument Performance Check (Compound Name)	Analysis Date and Time	Instrument	Ion(s) Affected	Percent Relative Abundance	QC Limits	Samples Affected	Action
Comments:							

Semivolatile Instrument Performance Check (Compound Name)	Analysis Date and Time	Instrument	Ion(s) Affected	Percent Relative Abundance	QC Limits	Samples Affected	Action
Comments:							

If tuning compounds and criteria are different from those specified in CLP SOW OLM03.1, then the validator should include a copy of the method-specific tuning criteria with this worksheet.

Validator:_____ Date:_____

III. INITIAL CALIBRATION - List all analytes that are outside calibration criteria.

Date of ICAL	Instrument	Parameter	Matrix	Compound	% RSD	$\overline{RRF}$	Samples Affected	Action

Comments:

Validator: _____ Date: _____

IV. CONTINUING CALIBRATION - List all analytes that are outside calibration criteria.

Date of ICAL	Date of CCAL	Instrument	Parameter	Matrix	Compound	%D	RRF	Samples Affected	Action

Comments:

Validator:_____ Date:_____

V. BLANK ANALYSIS

List the blank contamination below. Concentration Level:_____

Sampler: _____ Company: _____ Contacted: Yes No Date:_____

1. Laboratory: Method, Storage and Instrument Blanks

Date Extracted	Date Analyzed	Parameter/ Matrix	Sample No. (Blank Type)	Instrument/ Column	Compound	Conc. (units)

2. Field: Equipment (Rinsate), Trip and Bottle Blanks

Date Extracted	Date Analyzed	Parameter/ Matrix	Sample No. (Blank Type)	Instrument/ Column	Compound	Conc. (units)

Validator:_____ Date:_____

3. Blank Actions - List the maximum concentrations of blank compounds.

Compound	Type of Blank	Date Blank Sampled/Originated	Max. Conc. (units)	Action Level (units)	Sample QL	Samples Affected	Action

Comments: _____

_____

Validator:_____ Date:_____

VI. VOA SURROGATE SPIKE RECOVERIES - List all surrogate compound recoveries that are outside method QC acceptance criteria.

Method	Volatile Method QC Acceptance Criteria				
	Toluene-d_8	BFB	DCE-d_4	Other:	
OLM03.2	Water 88-110 Soil 84-138	Water 86-115 Soil 59-113	Water 76-114 Soil 70-121		
OLC02.1	NA	80-120	NA		
Other:					
Sample Number/Matrix	**% Recovery**	**% Recovery**	**% Recovery**	**% Recovery**	**Action**

Validator:_____ Date:_____

VI. SV SURROGATE SPIKE RECOVERIES - List all surrogate compound recoveries that are outside method QC acceptance criteria.

Method	Base/Neutral Method QC Acceptance Criteria					
	NBZ-d_5	2-FBP	TPH-d_{14}	1,2-DCB-d_4*	Other:	
OLM03.2	Water Soil 35-114 23-120	Water Soil 43-116 30-115	Water Soil 33-141 18-137	Water Soil 16-110 20-130		
OLC02.1	40-110	30-110	20-140	NA		
Other:						
Sample Number/Matrix	**% Recovery**	**% Recovery**	**% Recovery**	**% Recovery**	**% Recovery**	**Action**

Method	Acid Method QC Acceptance Criteria					
	Phenol-d_5	2-FP	2,4,6-TBP	2-CP-d_4*	Other:	
OLM03.2	Water Soil 10-110 24-113	Water Soil 21-110 25-121	Water Soil 10-123 19-122	Water Soil 33-110 20-130		
OLC02.1	15-115	15-110	15-130	NA		
Other:						
Sample Number/Matrix	**% Recovery**	**% Recovery**	**% Recovery**	**% Recovery**	**% Recovery**	**Action**

* Advisory Surrogates - OLM03.2

Validator:_____ Date:_____

VII. INTERNAL STANDARD PERFORMANCE

List the internal standards that are outside the area count and retention time method QC acceptance criteria.
IS Area Count method QC acceptance criteria:_____
IS Retention Time method QC acceptance criteria:_____

Sample Number (TR#)	Date and Time Analyzed	Instrument	Parameter	IS Outside Area Count and/or RT Criteria	__IS Area	RT Shift	Acceptable Range (IS area or RT shift)	Action

Validator:_____ Date:_____

VIII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE - List all MS/MSD analytes that are outside method QC acceptance criteria.

Use a separate worksheet for each MS/MSD pair.

Sample # _____ Matrix _____ Concentration Level _____

Parameter	Compound	MS %Rec	MSD %Rec	RPD	Method QC Limits		Concentration			% RSD	Action
					% Rec	RPD	Unspiked Sample	MS	MSD		

Validator: _____ Date: _____

IX. FIELD DUPLICATE PRECISION - List all field duplicate analytes that are outside criteria.

Use a separate worksheet for each field duplicate pair.

Sample Number _____ Duplicate Sample Number _____ Matrix _____

Parameter	Compound	Sample Conc.	Sample QL		Duplicate Conc.	Duplicate QL		RPD	QC Acceptance Criteria RPD or NA*	Action
			SQL	2xSQL		SQL	2xSQL			

* For instances where one duplicate result is ND (or reported less than the sample QL).

Does the MS/MSD data indicate acceptable laboratory precision? Y N

Comments: _____

_____

Sampler Name: _____ Contractor Name: _____ Date Contacted: _____

Reason for Contact and resolution obtained: _____

Validator: _____ Date: _____

X. SENSITIVITY CHECK (Method Detection Limit Study)

List all compounds, surrogates, and internal standards that are outside the MDL criteria.

- Has an appropriate MDL study been submitted with seven replicates for each compound and matrix of interest? **Y N**
- Date of Preparation/Analysis: _____ Within 1 year? **Y N**
- Instrument I.D.: _____ Same as samples? **Y N**
- Column I.D.: _____ Same as samples? **Y N**

Matrix	Compound	MDL > QL	Method QC Limits < 80% or > 120%	IS Outside Area Count and/or RT Criteria	RSD > 20%	Samples Affected	Action

If an MDL study has not been submitted, use only the LFB results to evaluate data.

(Laboratory Fortified Blank) - List all LFB compounds, surrogates and internal standards that are outside criteria.

- Has an appropriate and complete LFB been submitted at the proper frequency? **Y N**
- Does it contain all target compounds at the method-required QLs? **Y N**
- Was the LFB spiked with a standard from a source (vendor) independent of the calibration standard? **Y N**

Matrix	Compound	Method QC Limits < 60% or > 140% Other:	IS Outside Area Count and/or RT Criteria	Samples Affected	Action

Validator: _____ Date: _____

XI. ACCURACY CHECK (**Performance Evaluation Results**) - List all analytes that are outside criteria.

SDG No: _____ CASE: _____

Are more than one-half of the PES analytes within criteria for each parameter. **Y N**

PE Sample Number	Ampule Number	Parameter	Type of PES	Matrix	Analyte	Conc.	Region I EPA PES Scores*	Non-EPA PES Scores**	Samples Affected	Action

* For Region I PESs indicate the Region I PES Score Report Result: Action High; Action Low; TCL MISS; TCL CONTAMINANT; TIC HIT; TIC MISS; TIC CONTAMINANT

** For Non-EPA PESs indicate the Non-EPA PES Score: PES COMPOUND MISS; PES COMPOUND CONTAMINANT; PES COMPOUND HIT (% Recovery Limits)

Validator: _____Date: _____

XII. TARGET COMPOUND IDENTIFICATION - List the analytes that are outside the acceptance criteria.

Sample Number	Compound	___MS Ions	_____RRT	Action

Validator: _____ Date: _____

XIII. SAMPLE QUANTITATION

Recalculate, from the raw data, the concentrations for one positive detect and one reported sample quantitation limit for a non-detect in a diluted sample or soil sample per fraction. (Note: Although Section XIII, C.1.a, requires that one calculation for each fraction in each sample be performed, the validator is only required to reproduce an example, for each fraction, of one positive detect and one sample quantitation limit calculation on this worksheet.)

Do all soil/sediment samples have % solids greater than 30%? **Y N**
If no, list sample numbers_____

Fraction		Calculation
VOA		
Sample No.:		
Reported Compound:		
Reported Value:		
Not Detected Compound:		
Reported Quantitation Limit:		
BNA		
Sample No.:		
Reported Compound:		
Reported Value:		
Not Detected Compound:		
Reported Quantitation Limit:		
Pesticide/PCB		
Sample No.:		
Reported Compound:		
Reported Value:		
Not Detected Compound:		
Reported Quantitation Limit:		

Validator:_____ Date:_____

XIV. TENTATIVELY IDENTIFIED COMPOUNDS (TICs)

List the 5 TICs having the highest concentration for each sample parameter.

Sample Number	Parameter	Compound	_____RRT	__Est. Conc.	Action

Validator: _____ Date: _____

XV. SEMIVOLATILE CLEANUP - List all analytes that are outside method cleanup QC criteria.

Cleanup Procedure	Instrument # or Lot #	Date/Time GPC Calibrated or Check Solution Analyzed	Compound	% Rec	QC Limits	Samples Affected	Action

Did the GPC column meet; resolution requirements? Y or N
peak shape requirements? Y or N
retention time shift requirements? Y or N

Was the GPC calibration, Silica Gel cleanup checked at the method required frequency with correct compounds and concentrations? Y or N

Were all compounds less than QL for the GPC/Silica Gel/Acid-Partition blank? Y or N

Did the blank surrogate recoveries and IS area counts and RTs (if added) meet method QC acceptance criteria? Y or N

Comments:_____

_____

Validator:_____ Date:_____

For hardcopy of Traffic Reports contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

Attachment K

Example of Contract Compliance Screening (CCS) Report

U.S.E.P.A. CONTRACT LABORATORY PROGRAM
CONTRACT LABORATORY ANALYTICAL SERVICES SUPPORT - OPERATED BY DYNCORP
INORGANIC CONTRACT COMPLIANCE SCREENING SYSTEM

SEMI-AUTOMATED SCREENING RESULTS, OPTION PERIOD

```
************************************************************
************************************************************
************************************************************
****                                                    ****
****        S C R E E N I N G   P A C K A G E           ****
****      S U M M A R Y   I N F O R M A T I O N         ****
****                                                    ****
****                 ILM04.0 VER 1.4                    ****
****                                                    ****
****                  SDG:  MAKZ80                      ****
****                        RESUBMITTED                 ****
****                                                    ****
****                 CASE:  25931                       ****
****                                                    ****
****                  LAB:  SENTIN                      ****
****                                                    ****
****             CONTRACT:  68-D6-0001                  ****
****                                                    ****
****               REGION:  1                           ****
****                                                    ****
****        RESPONSE DATE:  02/02/98                    ****
****                                                    ****
****          MAIL DATE 1:  01/22/98                    ****
****                                                    ****
****          MAIL DATE 2:  02/05/98                    ****
****                                                    ****
****                                                    ****
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EFFECTIVE OCTOBER 1, 1997, THE CCS INTAKE REPORT WILL NO LONGER BE ROUTINELY DISTRIBUTED BY CLASS. IF YOUR LABORATORY WOULD LIKE TO RECEIVE THIS REPORT, PLEASE CONTACT EDWARD MESSER AT (703) 603-9047.

U.S.E.P.A. CONTRACT LABORATORY PROGRAM
CONTRACT LABORATORY ANALYTICAL SERVICES SUPPORT - OPERATED BY DYNCORP
INORGANIC CONTRACT COMPLIANCE SCREENING SYSTEM

CCS SCREENING RESULT OF DISKETTE

CCS DISCREPANCY LIST
ILM04.0 VERSION 1.4

VERSION 1.4 OF THE ILM04.X PC-CCS FOR WINDOWS IS AVAILABLE
ON EPA'S CLU-IN BULLETIN BOARD (301-589-8366) FROM DEC 01, 1997.
THE SELF EXTRACTING ZIPPED FILE IS LOCATED UNDER DIRECTORY
7 WITH THE FILE NAME ILM4V14.EXE
MODEM SETTINGS FOR CONNECTION TO THE CLU-IN BULLETIN BOARD SHOULD
BE: PARITY (N), BITS (8), STOP BITS (1). SHOULD YOU EXPERIENCE
ANY DIFFICULTY CONNECTING TO THE CLU-IN BULLETIN BOARD, OR IN
DOWNLOADING THE FILE, PLEASE CONTACT THE CLU-IN SYSOP AT
301-589-8368.

NO.	DESCRIPTION OF DISCREPANCY
1	

STATUS	LAB ACTION

NO.	DESCRIPTION OF DISCREPANCY
2	

STATUS	LAB ACTION

U.S.E.P.A. CONTRACT LABORATORY PROGRAM
CONTRACT LABORATORY ANALYTICAL SERVICES SUPPORT - OPERATED BY DYNCORP
INORGANIC CONTRACT COMPLIANCE SCREENING SYSTEM

SEMI-AUTOMATED SCREENING RESULTS, OPTION PERIOD

INORGANIC CONTRACT COMPLIANCE SCREENING SUMMARY
DEFECT COUNT BY SAMPLE AND CRITERION

EXPLANATORY NOTES

THIS REPORT SUMMARIZES BY SAMPLE THE TECHNICAL DEFECTS NOTED ON THE DISKETTE AND HARDCOPY DATA PACKAGE. THE "CYN" (CYANIDE) AND "MET" (MERCURY/METALS) COLUMNS SHOW WHETHER THE SAMPLE WAS (Y) OR WAS NOT (NA) ANALYZED. NUMBERS IN THE "CYN" OR "MET" COLUMNS INDICATE THE DAYS BY WHICH THE SAMPLE VIOLATED THE HOLDING TIME. NUMBERS IN THE "TECHNICAL/MANUAL DEFECTS" COLUMNS SHOW THE NUMBER OF NON-COMPLIANCE DEFECTS NOTED FOR A SAMPLE WITH REFERENCE TO A SPECIFIC CRITERION.

R E S U B M I S S I O N

CASE: 25931
LAB CODE: SENTIN
SDG NUMBER: MAKZ80

NO. OF SAMPLES: 7
CONTRACT: 68-D6-0001
REGION: 1

SOW: ILM04.0
RESPONSE DATE: 02/02/98
MAILDATE (2): 02/05/98

	HOLDING TIME		TECHNICAL/MANUAL DEFECTS																	
			CAUTION: THE DEFECTS IDENTIFIED BELOW MAY MASK OTHER DEFECTS WITHIN THE DATA. IT IS STRONGLY RECOMMENDED, THAT AFTER CORRECTING ANY DEFECT, THE DATA BE THOROUGHLY REVIEWED AND/OR PROCESSED THROUGH A CURRENT VERSION OF PC-CCS.																	
SAMPLE NO.	CYN	MET	DATA	ICV/ CCV	CRDL	ICB/ CCB	PREP. BLANK	ICS	SPIKE	PD SPIKE	DUP	LCS	MSA	SER. DIL.	IDL	IEC	ICP LIN. RANGE	PREP. DATA	RUN SEQ.	MAN. DEF.
LCSW	N	Y																		
MAKZ80	Y	Y																		
MAKZ81	Y	Y																		
MAKZ82	N	Y																		
MAKZ84	Y	N																		
MAKZ84D	Y	N																		
MAKZ84S	Y	N																		

U.S.E.P.A. CONTRACT LABORATORY PROGRAM
CONTRACT LABORATORY ANALYTICAL SERVICES SUPPORT - OPERATED BY DYNCORP
INORGANIC CONTRACT COMPLIANCE SCREENING SYSTEM
TEXTUAL/MANUAL DEFECT REPORT

THIS REPORT SUMMARIZES DEFECT MESSAGES. INFORMATION IN PARENTHESES AT THE END OF A DEFECT MESSAGE REFERENCES THE SOW PAGE NUMBER WHERE THE REQUIREMENTS ORIGINATED. FOLLOWING THE DEFECT MESSAGE IS A LIST OF ALL AFFECTED SAMPLES.

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*****                                                  *****
*****                                                  *****
*****    NO DEFECTS FOUND FOR TEXTUAL DEFECT REPORT    *****
*****    NO DEFECTS FOUND FOR MANUAL  DEFECT REPORT    *****
*****                                                  *****
*****                                                  *****
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U.S.E.P.A. CONTRACT LABORATORY PROGRAM
CONTRACT LABORATORY ANALYTICAL SERVICES SUPPORT - OPERATED BY DYNCORP
INORGANIC CONTRACT COMPLIANCE SCREENING SYSTEM

SEMI-AUTOMATED SCREENING RESULTS, OPTION PERIOD

LABORATORY CODE : SENTIN CASE : 25931 SDG: MAKZ80 DRD: 02/02/98

CRITERION	COMMENTS
AG01	THANK YOU FOR YOUR RESPONSE.
AG02.1	THANK YOU FOR YOUR RESPONSE.
AG03.1	THANK YOU FOR YOUR RESPONSE.
AI01	THANK YOU FOR YOUR RESPONSE.
AL01	THANK YOU FOR YOUR RESPONSE.

Attachment L

"Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review", February 1995

GUIDANCE DOCUMENT FOR COMPLETING REGION I DATA VALIDATION UTILIZING CADRE DATA REVIEW CADRE RELEASE 2.10

CONTRACT LABORATORY PROGRAM
ROUTINE ANALYTICAL SERVICES
VOLATILE AND SEMIVOLATILE ORGANICS

FEBRUARY 1995
REV: MARCH 14, 1995

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INTRODUCTION CADRE version 2.10

CADRE (Computer Aided Data Review and Evaluation) version 2.10 is a software program which is designed to aid in the validation of volatile and semivolatile CLP RAS data packages. CADRE is capable of interpreting the electronic deliverable which the laboratory is required to submit to CLASS under SOWs OLM01.9 and OLM03.1. CADRE performs a review of data quality by comparing the quality control results to a preprogrammed set of criteria. The criteria used for evaluation by CADRE are defined in the National Functional Guidelines for Organic Data Review (Draft, June 1991).

This document is designed to guide the validator in completion of a Region I Tier II data validation utilizing CADRE's findings. For each quality control parameter reviewed, CADRE will generate a worksheet reporting any problems found during the review. CADRE will also provide recommendations for qualification of the data based upon these problems. The recommendations made by CADRE are identical to those suggested in the National Functional Guidelines for Organic Data Review (Draft, June 1991).

In some instances, however, the recommendations of the National Functional Guidelines (and, therefore, the recommendations made by CADRE) may differ from those suggested in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses, November 1988. The actions recommended by CADRE on the data review worksheets should be followed unless stated otherwise in this guidance document. In the cases where specific actions are not stated by CADRE or included in this guidance document, all guidelines for review and data qualification set forth in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses, November 1988, are to be followed. Any deviations in the review process or qualifications placed on sample results must be clearly justified in the Data Validation Memorandum as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses.

NOTE: If pesticides/PCBs were analyzed along with the volatile and semivolatile organics for a particular SDG, CADRE will attempt to validate the pesticide/PCB fraction. CADRE will display its findings of the pesticide/PCB validation on the CADRE worksheets along with its findings of the validation of the volatile and semivolatile fractions. Currently, CADRE validates many pesticide/PCB parameters different than Region 1. Any pesticide/PCB results reported by CADRE on the CADRE worksheets should be ignored. All Region 1 pesticide/PCB analyses should be manually validated.

However, it should be noted that CADRE pesticide/PCB Data

Summary Tables will be generated during CADRE validation and should be used during validation. These Data Summary Tables will be delivered, along with the CADRE Data Validation Report, to the Field Sampling Contractor.

A completed set of example worksheets (CADRE and manual) is included in Attachment I. A full set of blank Region I data validation worksheets is included in Attachment II. A tabular summary of the manual review necessary to complete a Region I Tier II data validation is included in Attachment III. The differences between the National Functional Guidelines, CADRE, and the Region I Functional Guidelines criteria are summarized in a series of tables included in Attachment IV.

CADRE Data Summary Tables

CADRE will create Data Summary Tables summarizing the results reported by the laboratory on the Form Is. CADRE can generate either unqualified or qualified Data Summary Tables. The CADRE unqualified Data Summary Tables contain the Form 1 results and qualifiers as reported by the laboratory. The CADRE qualified Data Summary Tables contain the CADRE Form 1 results along with any qualifiers resulting from the CADRE validation process.

CADRE, wherever possible, will recalculate sample values. On occasion, CADRE will round values differently than the laboratory. CADRE is programmed to round results according to rules stated in SOWs OLM01.9 and OLM03.1. The differences occur when the laboratory reporting software uses rounding rules which vary from those stated in the SOW. In these cases, the results reported by CADRE (the CADRE Form I results) represent the true CLP values. If, however, the CADRE results and the laboratory Form I results differ due to slight rounding errors, the results reported by the laboratory will be included by the ESAT CADRE chemist on the qualified Data Summary Tables. This will insure consistency of sample result transcription from the hardcopy to electronic deliverables.

The CADRE qualified Data Summary Tables for the volatile and semivolatile fractions will be provided to the Field Sampling Contractor. These Data Summary Tables will contain qualifiers recommended by CADRE on the CADRE worksheets generated during the review of each QC parameter. The Field Sampling Contractor will be required to verify that all qualifiers have been correctly transcribed onto the Data Summary Tables by CADRE. The Field Sampling Contractor will also be required to place any qualifiers onto the Data Summary Tables which result from any required manual validation.

For the pesticide/PCB fractions and in instances where major discrepancies exist between the sample values reported on the CADRE qualified Data Summary Tables and the laboratory Form 1s, the CADRE unqualified Data Summary Tables will be sent to the Field Sampling Contractors along with a notice that the Data Summary Tables are unqualified. The unqualified Data Summary Tables distributed to the Field Sampling Contractors will contain only the Form I results and qualifiers as reported by the laboratory. The Field Sampling Contractors will be required to remove all laboratory qualifiers from the CADRE unqualified Data Summary Tables (such as the "B", "D", and "E" qualifiers), with the exception of the "J" qualifier,

for results detected below the CRQL, and the "U" qualifier, for non-detect results. The Field Sampling Contractor must then add any qualifiers resulting from the completion of the data validation.

Along with the hardcopy Data Summary Tables, a diskette containing the WordPerfect files for the Data Summary Tables will be provided to the Field Sampling Contractors.

Each CADRE Data Summary Table file will be named to identify the SDG, fraction and sample matrix. The CADRE files are created as ASCII files and are then transformed into WordPerfect files by the ESAT CADRE Chemist prior to being distributed to the Field Sampling Contractor. The file naming scheme is as follows: "SDG#XY.TXT".

SDG# = The SDG number for the CLP data package.

X = Fraction
B for Semivolatiles (BNA)
V for Volatiles
P for Pesticides
M for Metals

Y = Sample Matrix
A for Aqueous
S for Soil

The .TXT file extension is assigned by MSDOS, when the CADRE file is created as an ASCII file, to designate that the file is a text file. This extension is retained when the file is converted into WordPerfect format.

For example - SDG AEN06 has 5 soil samples and 4 water sample for volatiles, semivolatiles, and pesticides/PCBs analysis. The files for the Data Summary Tables would be named as follows:

<u>Semivolatiles</u>

AEN06BS.TXT for the semivolatile soil samples
AEN06BA.TXT for the semivolatile water samples

<u>Volatiles</u>

AEN06VS.TXT for the volatile soil samples
AEN06VA.TXT for the volatile water samples

Pesticides/PCBs

AEN06PS.TXT for the pesticide/PCB soil samples
AEN06PA.TXT for the pesticide/PCB water samples

A backup file for each Data Summary Table will also be included on the diskette. The backup file will be named similarly to the original file but the ".TXT" extension is replaced with a ".BKP" extension. This backup file will be included only to serve as a second copy of the Data Summary Tables, if for any reason the original becomes unusable.

The Data Summary Tables will also be submitted electronically in ASCII format. This format will be available for use in site databases if desired. The ASCII files will be named similarly to the original file but the ".TXT" extension will be replaced with a ".DB".

The CADRE Data Summary Tables have been formatted as WordPerfect files. This formatting includes creating a defined structure for the table boundaries.

In order to preserve the table boundaries, all edits to the summary tables **must be performed in the "typeover" mode.** After retrieving the file onto the computer screen, press the <INSERT> key. To verify that you are in typeover mode, the word "typeover" should appear on the bottom left hand corner of the computer screen.

COMPLETING THE CADRE Data Validation Report

Upon completion of the CADRE validation, a CADRE report which consists of the CADRE Worksheets, hardcopy and diskette CADRE Data Summary Tables, and CADRE Data Review Inventory Sheet will be shipped along with the CLP Data Package to the Field Sampling Contractor. Upon receipt of the CADRE report and CLP Data Package, the Field Sampling Contractor should complete the Region I Complete SDG File Receipt/Transfer Form and the Organics Complete SDG File (CSF) Inventory Sheet (Form DC-2) as usual, as well as the CADRE Data Review Inventory Sheet to verify data completeness. (Copies of these forms are included in Attachment V). The completed CADRE Data Review Inventory Sheet should be included with the Data Validation Report.

ROLES OF ESAT IN THE CADRE VALIDATION PROCESS

The following section describes the roles that ESAT personnel play in the procession of the CADRE validation.

1. Receipt of the Data Package From the EPA RSCC at ESD

   The EPA RSCC at ESD will transfer custody of the data package to the ESAT Lexington Data Preparer. The ESAT Lexington Data Preparer will log the data package into the CLP Sample Tracking System (CLPSTS) and will indicate on the Region 1 Complete SDG File (CSF) Receipt/Transfer Form if the data package is for ESD/ESAT, ARCS, or TES validation.

   The ESAT Data Preparer will then transfer custody of the data package to the ESAT CADRE Chemist.

2. Initiation of CADRE SDG Tracking Process

   Upon receipt of the data package, the ESAT CADRE Chemist will begin a CARD/CADRE SDG Tracking Form. (A copy of the CARD/CADRE SDG Tracking Form is included in Attachment VI). The purpose of this tracking form is to provide internal assurance that all of the necessary steps for generating the CADRE report have been completed and are documented. A copy of the CARD/CADRE SDG Tracking Form will be included in the CADRE report file for each SDG, which will be kept in the EPA ESD central files.

3. Downloading of Electronic Deliverable

   Upon receipt of the data package, the ESAT CADRE Chemist will download the SDGs from the CARD database to the CADRE PC.

4. Importing the SDG into CADRE

   ESAT will import the SDG into CADRE. After successfully importing the SDG into CADRE, ESAT will manually enter the sampling dates, sampling preservation for volatiles, laboratory sample numbers, and sampling locations. If any errors are detected by CADRE during the import process, ESAT will generate the CADRE import error reports.

5. Manual Data Entry and CADRE Data Review

The ESAT CADRE Chemist will manually enter any missing or discrepant data. After completion of all manual data entry, the ESAT CADRE Chemist will execute the CADRE review of the data and generate CADRE worksheets.

6. Generating and Formatting the Data Summary Tables

The ESAT CADRE Chemist will format the Data Summary Tables to conform to the current Region I Data Summary Table specifications. The one exception to this format is that currently there is no space on the CADRE Data Summary Tables to include a column for the SOW CRQLs. However, the sample-specific CRQLs will be listed for each compound which is not detected. The Data Summary Tables will be exported in ASCII format and converted into WordPerfect files. The CADRE qualified Data Summary Tables for the volatile and semivolatile fractions will be provided to the Field Sampling Contractor. These Data Summary Tables will contain qualifiers recommended by CADRE on the CADRE worksheets generated during the review of each QC parameter. The Field Sampling Contractor will be required to verify that all qualifiers have been correctly transcribed onto the Data Summary Tables by CADRE. The Field Sampling Contractor will also be required to place any qualifiers onto the Data Summary Tables which result from any required manual validation.

For the pesticide/PCB fractions and in instances where major discrepancies exist between the sample values reported on the CADRE qualified Data Summary Tables and the laboratory Form 1s, the CADRE unqualified Data Summary Tables will be sent to the Field Sampling Contractors along with a notice that the Data Summary Tables are unqualified. The unqualified Data Summary Tables distributed to the Field Sampling Contractors will contain only the Form I results and qualifiers as reported by the laboratory. The Field Sampling Contractors will be required to remove all laboratory qualifiers from the CADRE unqualified Data Summary Tables (such as the "B", "D", and "E" qualifiers), with the exception of the "J" qualifier, for results detected below the CRQL, and the "U" qualifier, for non-detect results. The Field Sampling Contractor must then add any qualifiers resulting from the completion of the data validation.

7. Delivery of CADRE report to Field Sampling Contractors

The ESAT CADRE Chemist will prepare the hardcopy CADRE report, which consists of the CADRE worksheets and CADRE Data Summary Tables. The ESAT CADRE Chemist will also prepare a diskette containing three copies of the file for the Data Summary Tables. One file is to be used as the working file for adding qualifiers to the summary tables. The second copy will be given a .BKP extension. This file is included as a backup file if needed. The third copy will be delivered in ASCII format for use in the site database if desired.

Prior to shipping the CADRE report and CLP Data Package, the ESAT CADRE Chemist will verify the completeness of the CADRE report by initiating the CADRE Data Review Inventory Sheet. This sheet will be delivered with the CADRE report to the Field Sampling Contractor.

The ESAT CADRE Chemist will send the CLP RAS Data Package, CADRE report, and diskette containing the files for the Data Summary Tables simultaneously to the appropriate Field Sampling Contractor.

8. Notification of Required Full Manual Data Validation

On some occasions, the diskette deliverable from the laboratory will not pass the Contract Compliance Screening (CCS), which is performed by the Contract Laboratory Analytical Services Support (CLASS) contractor. Those SDGs which do not pass the CCS screen will not be uploaded into the CARD database, and subsequently, will not be available for CADRE review.

Those SDGs must have a full manual validation performed by the Field Sampling Contractor. The ESAT CADRE Chemist will send those SDGs to the Field Sampling Contractor with a notification that manual validation must be performed.

9. Notification of Required Partial Manual Validation

On some occasions, CADRE will be unable to validate a portion of the SDG due to problems with the electronic deliverable. These affected parameters must be manually validated. The ESAT CADRE Chemist will send the remaining CADRE report and data package to the Field Sampling Contractors with a notice of which parameters must be manually validated.

10. Storage and Archival of CADRE Data

The ESAT Data Preparer will store a copy of all CADRE-generated worksheets, hardcopy and diskette CADRE Data Summary Tables, CADRE Error Reports, and the CARD/CADRE SDG Tracking Form in the EPA ESD central files. A copy of the CARD/CADRE SDG Tracking Form will also be kept by the ESAT CADRE Chemist to generate the weekly CADRE Status Report for EPA.

11. Weekly CADRE Status Reports

The ESAT CADRE Chemist will provide the EPA Data Validation Chemist and the CLP-TPO with a weekly report summarizing the CADRE activities for the previous week.

ROLES OF FIELD SAMPLING CONTRACTORS IN THE CADRE VALIDATION PROCESS

The following section describes the roles that the Field Sampling Contractors play in the validation of CLP RAS data utilizing CADRE.

1. Receipt of Hardcopy Data and CADRE Report

   The Field Sampling Contractors shall receive the hardcopy CLP Data Package and CADRE report, consisting of CADRE worksheets and CADRE Data Summary Tables, simultaneously from the ESAT CADRE Chemist. If no problems are encountered with CADRE, the CLP Data Package and CADRE report will be shipped by the ESAT CADRE Chemist from ESAT/ESD within 3 days of receipt of the CLP Data Package from the EPA RSCC. A diskette containing the WordPerfect file for the Data Summary Tables will accompany the hardcopy CADRE Data Summary Tables and worksheets. A CADRE Data Review Inventory Sheet which has been completed by the ESAT CADRE Chemist will also be shipped with the CADRE report.

2. Data Completeness Check

   Upon receipt of the data package, the Field Sampling Contractor shall complete the Organics Complete SDG File (CSF) Inventory Sheet (Form DC-2) as usual and the CADRE Data Review Inventory Sheet to verify data completeness. The completed CADRE Data Review Inventory Sheet should be included with the Data Validation Report.

   If any CADRE data are missing or discrepancies are detected in the CADRE report, then the Field Sampling Contractor should notify the Region I EPA Data Validation Chemist for correction or clarification.

   If any data are missing from the CLP Data Package, then the Field Sampling Contractor should contact the laboratory to obtain the missing data.

3. Completion of the Tier I Validation

   If only a Tier I validation was required in the QAPjP (Quality Assurance Project Plan) and/or SAP (Sampling and Analysis Plan), then the Field Sampling Contractor should complete the Tier I validation as described in the Region I CSF Completeness Evidence Audit Program, dated 7/3/91. This procedure was referenced in a memorandum titled "Region I CSF Completeness Evidence Audit Program" from the Region I CLP-

TPOs to Region I Contractors, dated 7/7/91.

If a Tier II or Tier III validation was not required in the QAPjP and/or SAP, then the CADRE report and diskette, including the completed CADRE Data Review Inventory Sheet, should be stored with the CLP data package.

4. Completion of the Tier II and Tier III validation

The Field Sampling Contractor shall complete the Tier II and Tier III data validation utilizing the findings of CADRE. The validation should be completed using the Region I Tiered Organic and Inorganic Data Validation Guidelines, dated 7/1/93, the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (as modified by Region I, 11/88) and the Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review (February 1995).

If CADRE review of an SDG is not possible, the Field Sampling Contractor will be sent the CLP Data Package with a notification that a manual validation must be performed. Currently, manual validation must also be performed for all CLP RAS Pesticides/PCB data. The Field Sampling Contractor shall perform the Tier II or Tier III data validation using the Region I Tiered Organic and Inorganic Data Validation Guidelines, dated 7/1/93 and the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (as modified by Region I, 11/88).

5. Writing of the Data Validation Report

The Field Sampling Contractor should complete the Data Validation Memorandum, Data Summary Tables, and worksheets as directed in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (as modified by Region I, 11/88) and the Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review (February 1995).

6. Delivery of Final Validation Report

The final validation report, including Data Validation

Memorandum, CADRE and Region 1 Worksheets, Data Summary Tables, CADRE Data Review Inventory Sheet, etc. should be addressed to the appropriate EPA RSCC representative (currently Christine Clark) at the Environmental Services Division (ESD) in Lexington, MA. The original validation report should be delivered to the RPM for the site at EPA WMD and a copy delivered to Christine Clark, EPA RSCC in Lexington, MA.

ROLES OF EPA IN THE CADRE VALIDATION PROCESS

The following section describes the roles that Region I EPA personnel play in the validation of CLP RAS data utilizing CADRE.

1. Receipt of Hardcopy Data at EPA ESD

   The transfer of the RSCC function from EPA Waste Management Division (WMD) to EPA Environmental Services Division (ESD) occurred in January 1995. All CLP Data Packages are currently supposed to be shipped to EPA ESD by the CLP laboratories.

2. Transfer of the Data Packages From EPA WMD to EPA ESD

   If any CLP Data Packages are mistakenly shipped to EPA WMD, then EPA WMD will transfer the data package to EPA ESD via the EPA internal mailing system.

   Upon receipt of the data package by the EPA RSCC, the EPA RSCC will initiate the custody tracking of the data package by filling out the EPA Region 1 Complete SDG File (CSF) Receipt/Transfer Form and will transfer custody of the data package to the ESAT Lexington Data Preparer.

3. CADRE Support to Field Sampling Contractors

   The EPA Data Validation Chemist shall provide CADRE support to the Field Sampling Contractors. This includes tasking ESAT to provide any CADRE resubmittals if necessary and/or answering CADRE questions raised by the Field Sampling Contractors.

4. Receipt and Review of Data Validation Reports From Field Sampling Contractors

   The EPA RSCC representative (currently Christine Clark) at the Environmental Services Division (ESD) in Lexington, MA shall receive a copy of the Data Validation Report. The EPA RPM for the site (located at EPA WMD) shall receive the original validation report.

   The Data Validation Report shall be reviewed by the EPA Data Validation Chemist for use in data validation oversight and laboratory analysis oversight activities.

REGION I TIERED APPROACH TO DATA VALIDATION

The data validation process can be broken down into three distinct levels: Tier I, Tier II, and Tier III.

Tier I: A Completeness Evidence Audit is performed to ensure that all laboratory data and documentation are present. Completeness Evidence Audits are performed in accordance with procedures contained in the Region I CSF Completeness Evidence Audit Program, dated 7/3/91. (This document is the currently used procedure referenced in the memorandum titled "Region I CSF Evidence Audit Program" from the Region I CLP-TPOs to Region I Contractors, dated 7/7/91.)

The validation procedures contained in this Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, dated February 1995, are not applicable for Tier I validation. The validation procedures contained in the Region I Tiered Organic and Inorganic Data Validation Guidelines, dated 7/1/93, should be followed. If only a Tier I validation was required in the QAPjP/SAP, then the CADRE report and diskette should be stored with the CLP Data Package for future use. The CADRE Data Review Inventory Sheet should be completed by the Field Sampling Contractor and supplied with a letter to the EPA RSCC at ESD in Lexington, MA and the site RPM stating that the QAPjP/SAP required only a Tier I validation. The technical justification for performing only a Tier I validation must also be documented in that letter.

Tier II: A Tier I Completeness Evidence Audit is performed, then the results of all Quality Control (QC) checks and procedures are evaluated and used to assess and qualify sample results. Tier II data validation is performed primarily from information contained on the tabulated data reporting forms.

The validation procedures contained in this Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, dated February 1995, and the Region I Tiered Organic and Inorganic Data Validation Guidelines, dated 7/1/93, are used in conjunction with the CADRE report to complete a Tier II validation for CLP RAS volatiles and semivolatiles.

Tier III: A full validation is performed. Tier III includes Tier I and Tier II procedures plus an in-depth examination of all raw data to check for technical, calculation, analyte identification/analyte quantitation, and transcription errors.

The validation procedures contained in this Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, dated February 1995, and the Region I Tiered Organic and Inorganic Data Validation Guidelines, dated 7/1/93, are used in conjunction with the CADRE report to complete the Tier II portion of the Tier III data validation. The validation procedures contained in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, modified 11/88, are used to complete the remainder of the Tier III data validation.

SECTION I DATA COMPLETENESS

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

CADRE generates import error reports if it detects errors or identifies that data are missing. All import errors are corrected by the ESAT CADRE Chemist prior to delivery of the CADRE report and CLP Data Package to the Field Sampling Contractor. Therefore, there is no interpretation required for this parameter.

II. FURTHER MANUAL REVIEW REQUIRED

Verifying the Completeness of the CLP Data Package

1) The contents of the CLP Data Package should be reviewed for completeness by completing the Organics Complete SDG File (CSF) Inventory Sheet (Form DC-2) as per the Region I CSF Completeness Evidence Audit Program, dated 7/3/91. This sheet is submitted by the laboratory with the CLP Data Package. This sheet must be signed by the person who performed the completeness check.

2) If any data from the CLP Data Package are missing or incorrect, the validator must request submission of this information from the laboratory.

3) The validator must complete the Region I Data Completeness Worksheet. The worksheet and any Records of Communication with the laboratory must be included with the Data Validation Report. For completing a Tier I validation, refer to the Roles of the Field Sampling Contractor section for guidance.

Verifying the Completeness of the CADRE Report

1) The CADRE report and Data Summary Tables should be reviewed for completeness and accuracy by completing the CADRE Data Review Inventory Sheet. This sheet is included with the CADRE report. The sheet must be signed by the person who performed the completeness check.

2) If any CADRE report data are missing or incorrect, then the validator must contact the Region I EPA Data Validation Chemist for correction and/or submission of the information.

3) The validator must complete the CADRE Data Review Inventory Sheet. The CADRE Data Review Inventory Sheet and any Records of Communication with the Region I EPA Data Validation Chemist must be included with the Data Validation Report. For completing a Tier I validation, refer to the Roles of the Field Sampling Contractor section for guidance.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

No action is required. All errors in electronic data completeness will be detected by CADRE during the import process and will be corrected by the ESAT CADRE Chemist prior to shipping the CADRE report and CLP Data Package to the Field Sampling Contractor.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review as required in Section II.

2) Include the CADRE Data Review Inventory Sheet with the Data Validation Report. The completed photocopy of the DC-2 form shall remain with the CLP Data Package.

3) If resubmittals were required, include any Records of Communication requesting these resubmittals with the Data Validation Report.

4) Include the Region I Data Completeness Worksheet after the Region I Review of Organic Contract Laboratory Data Package cover sheet and the CADRE Sample Listing page in the Data Validation Report.

5) Any CLP Data Package resubmittals shall remain with the CLP Data Package. Any CADRE resubmittals shall be included with the Data Validation Report.

SECTION II QUANTITATION LIMITS

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

CADRE produces a Quantitation Limit Report which lists any compounds detected below the CRQL. These results are already qualified (J) by the laboratory on the Form 1s. CADRE automatically transcribes the (J) qualifiers onto the CADRE Data Summary Tables. No further qualification of these results is required.

II. FURTHER MANUAL REVIEW REQUIRED

1) None.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

1) None.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Include the CADRE Quantitation Limit Report after the Region I Data Completeness Worksheet in the Data Validation Report.

2) Sample result qualifiers are already placed on the Data Summary Tables. No further action is required.

SECTION III HOLDING TIMES

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

1) **Holding Times**

The CADRE Holding Times Report for aqueous and soil samples displays the criteria used by CADRE for volatiles and semivolatiles when holding times are slightly or grossly exceeded. The criteria used by CADRE are identical to the criteria used by Region 1. If all holding times criteria are met, CADRE will display the message "No problems found for this qualification."

If holding times are exceeded, CADRE will state which samples exceeded holding times and the appropriate Region I action to be taken. The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables.

It should be noted that CADRE will list any volatile soil samples exceeding holding times twice on the Holding Times Worksheet. This is due to a difference in soil sample preservation designation between CADRE and Region 1. This does not affect the validation of holding times.

2) **Percent Solids**

The CADRE Percent Moisture Report displays the criteria used by CADRE for percent moisture (percent solid) determination. If all percent moisture criteria are met, CADRE will display the message "No problems found for this qualification." If the percent moisture exceeds the limits specified by Region 1, CADRE will list the samples affected and whether the percent moisture exceeded the primary or expanded criteria. CADRE will also list the appropriate Region 1 action to be taken. The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables.

II. FURTHER MANUAL REVIEW REQUIRED

1) If no error messages are reported, then no further manual review is required.

2) Manual review is required if CADRE reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE Holding Times Report or the CADRE Percent Moisture Report. The validator should refer to Section III to determine the extent of manual review required. Region 1 Holding Times Worksheets must be utilized to document the manual review.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

CADRE can display two possible error messages for Holding Times and one possible error message for Percent Moisture.

A. Possible Errors

Holding Times

1) Samples Missing Sampling Date.

- If CADRE does not find a sampling date, it will perform the holding times evaluation using the VTSR. CADRE will display an error message on the Holding Times Report stating that no sampling date was found.

2) Missing Holding Time Information.

- If any necessary information are missing (other than the sampling date), CADRE will not perform the review for this parameter. An example of this is when there is no sample preservation designated for volatile water samples.

Percent Moisture

1) Missing Percent Moisture Information

- If CADRE cannot find percent moisture information, CADRE cannot perform the review for this parameter.

B. Required Action

Holding Times

1) If either of the two holding time error messages are reported, manual review of the sampling paperwork and CLP Data Package must be performed to evaluate holding times. The sampler must be contacted to resolve discrepancies, if necessary. Manual review of the associated holding time information that was not reviewed by CADRE must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. A Region I Holding Times Worksheet must be completed to document the manual review.

Percent Moisture

1) A manual review of the percent moisture (percent solid) information in the CLP Data Package must be performed. The criteria and actions outlined in the memorandum sent to Region 1 Data Validators by the Region 1 CLP TPOs titled "Qualifying Soil/Sediment Data With Low Percent Solid", dated March 29, 1990 must be used. (A copy of this memorandum is included as Attachment VII.)

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review as required in Section II.

2) Include the CADRE Holding Times Report and any required Region I Holding Times Worksheets after the CADRE Quantitation Limit Report in the Data Validation Report. Include the CADRE Percent Moisture Report after the CADRE Holding Times Report (and Region 1 Holding Times Worksheets, if applicable) in the Data Validation Report.

3) Discuss any qualifications placed upon sample results in

the Data Validation Memorandum and provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

4) Include required qualifiers in the Recommendations Summary Table (Table I of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Tables as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION IV GC/MS TUNING

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

The CADRE Instrument Performance Check Report displays the criteria used by CADRE for review of the BFB and DFTPP instrument performance checks. The criteria used by CADRE are identical to the criteria used by Region I. The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables. If all criteria are met, CADRE will display the message "No problems found for this qualification".

II. FURTHER MANUAL REVIEW REQUIRED

1) If no error messages are reported, then no further manual review is required.

2) Manual review is required if CADRE reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE Instrument Performance Check Report. The validator should refer to Section III to determine the extent of manual review required. Region 1 GC/MS Tuning Worksheets must be utilized to document the manual review.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

A. Possible Errors

CADRE can display three possible error messages for GC/MS Tuning

1) The instrument tune is missing.

- CADRE will display this message if it is expecting to find and evaluate a BFB/DFTPP instrument performance check sample but could not locate one in the electronic deliverable.

2) The incorrect base mass was normalized.

- During the instrument performance check, the laboratory is required to normalize (use as 100% relative abundance) m/z 95 for BFB and m/z 198 for DFTPP. CADRE will display this message if it detects the base mass to be a mass other than m/z 95 for BFB or m/z 198 for DFTPP.

3) The instrument tune did not meet tuning criteria.

- CADRE will display this message if the primary BFB/DFTPP tuning criteria displayed on the Instrument Performance Check Report are not met.

B. **Required Action**

1) If any of these error messages are reported, manual review of the CLP Data Package must be performed to determine if the associated GC/MS Tuning information are present and meet tuning acceptance criteria. If the associated tuning information are missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated tuning information must be performed using the criteria and actions outlined in the Region 1 Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Region 1 GC/MS Tuning Worksheets must be utilized to document the manual review.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review as required in Section II.

2) Include the CADRE Instrument Performance Check Report and any required Region 1 GC/MS Tuning Worksheets after the CADRE Percent Moisture Report in the Data Validation Report.

3) Discuss any qualifications placed upon sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region I

Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

4) Include required qualifiers in the Recommendations Summary Table (Table I of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Tables as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION V CALIBRATIONS

I. INTERPRETATION OF THE CADRE WORKSHEETS AND FINDINGS

CADRE generates three reports to aid in the review of calibration data.

1) **Analytical Sequence**

The report titled Analytical Sequence contains a summary of all analyses associated with the SDG in chronological order by instrument and fraction. This report lists the BFB/DFTPP Instrument Performance Checks (IPCs) and calibrations, as well as all samples which were analyzed within the 12 hour time period following the BFB/DFTPP IPC.

2) **Calibration Listing**

The report titled Calibration Listing includes all the initial and continuing calibrations which are associated with the SDG along with the dates and times of analysis. This report lists all samples which were associated with each continuing calibration, and the calibrations are reported in chronological order by instrument and fraction.

This report also lists all compounds which failed to meet the initial calibration %RSD criteria of less than 30% and/or the continuing calibration %D criteria of less than 25%. The actual non-compliant %RSD and/or %D values are also reported. This report also includes a list of all calibration compounds which failed to meet the relative response factor (RRF) criteria of greater than 0.05. The actual non-compliant RRFs are also reported. If the RRF was out in an individual standard in the initial calibration, CADRE also identifies that individual standard.

3) **Calibration Report**

If all criteria are met, CADRE will display the message "No problems found for this qualification".

The Calibration Report displays the criteria used by CADRE when minimum RRFs and maximum %RSDs/%Ds have been slightly or grossly exceeded. If RRFs and/or %RSD/%D criteria are not

met, CADRE will state which samples and compounds are affected by non-compliant calibrations and the appropriate Region 1 action to be taken.

The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables.

II. FURTHER MANUAL REVIEW REQUIRED

1) If no error messages are reported on the CADRE Calibration Report, then no further manual review of the data is required.

2) Manual review is required if CADRE reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE Calibration Report. The validator should refer to Section III to determine the extent of manual review required. Region 1 Volatile Calibration Verification and/or Semivolatile Calibration Verification Worksheets must be utilized to document the manual review.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

CADRE can display four possible error messages for Calibrations.

A. Possible Errors

1) Samples with no instrument performance check.

- For each sample in the SDG, CADRE will attempt to locate the associated instrument performance check to determine if the instrument performance check passed the tuning criteria. If CADRE cannot locate the instrument performance check, it cannot associate a calibration standard with the instrument performance check standard. Thus, CADRE will not evaluate the calibration standard associated with the missing instrument performance check. Any sample(s) associated with the calibration standard(s) will not be evaluated for this parameter.

2) Samples with no associated calibration.

- If CADRE cannot associate a calibration standard with a sample, it cannot evaluate calibration criteria for that sample.

3) Samples associated with a continuing calibration for which no corresponding initial calibration is found.

- If CADRE cannot associate a continuing calibration with an initial calibration, it cannot calculate a %D. Therefore, it cannot evaluate the calibration criteria for that sample.

4) Missing calibration information.

- If CADRE cannot locate any necessary calibration information, other than the information listed above, it cannot perform an evaluation of that calibration. This may include but is not limited to the following information:

  - Missing a response factor for one or more compounds in the continuing calibration.

  - Missing the response factors for one or more points in the initial calibration.

B. **Required Action**

1) A manual review of the CLP Data Package must be performed to determine if the instrument performance check associated with the calibration(s) for the affected sample is present. If the instrument performance check is missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the instrument performance check and the associated calibration that was not reviewed by CADRE must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating

Organics Analyses. Region 1 Volatile Calibration Verification and/or Semivolatile Calibration Verification Worksheets must be utilized to document the manual review.

2) For errors 2-4 listed in Section III above, manual review of the CLP Data Package must be performed to determine if the associated calibration information are present and meet calibration acceptance criteria. If the associated calibration information are missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated calibration information must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Region 1 Volatile Calibration Verification and/or Semivolatile Calibration Worksheets must be utilized to document the manual review.

IV. **STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION**

1) Complete any manual review as required in Section II.

2) Include the CADRE Analytical Sequence, Calibration Listing, Calibration Report, and any required Region 1 Volatile Calibration Verification and/or Semivolatile Calibration Verification Worksheets after the CADRE Instrument Performance Check Reports and any required Region 1 GC/MS Tuning Worksheets in the Data Validation Report.

3) Discuss any qualifications placed upon sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (mod 11/88). Include, in the Data Validation Memorandum, a table of compounds not meeting the calibration criteria along with the samples associated with each calibration.

4) Include required qualifiers in the Recommendations Summary Table (Table I of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Tables as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION VI BLANKS

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

A. Laboratory Blanks

The CADRE Laboratory Blanks Report specifies the multipliers used for calculating action levels.

If all criteria are met, CADRE will display the message "No problems found for this qualification".

The CADRE Laboratory Blanks Report indicates which samples and compounds have been considered as non-detects due to method blank contamination. The CADRE Laboratory Blanks Report will also note whether the sample result is to be reported qualified as (U) or the sample result is to be raised to the CRQL and qualified as (U). The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables.

The Laboratory Blanks Report incorrectly qualifies equipment and trip blanks due to laboratory blank contamination. The qualification for these blanks should be ignored as Region I does not qualify equipment or trip blanks for laboratory blank contamination. For qualified Data Summary Tables, the validator needs to remove any qualifiers placed by CADRE on equipment/trip blanks due to laboratory blank contamination.

B. Equipment and Trip Blanks

CADRE distinguishes equipment and trip blanks from regular field samples. CADRE has the capability to evaluate these blank samples for contamination. This function is currently not being used. In many instances, the equipment and trip blank samples are not included in the same SDGs as the field samples. CADRE cannot evaluate these blanks if they are contained in a separate SDG from the associated field samples.

Manual review of the CADRE Data Summary Tables (or Form Is) for these blanks is required to determine the extent of contamination and to document the appropriate qualification of the regular field samples. Region I Blank Analysis Results Worksheets must be completed for these blanks. The review must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Actions should be applied to all samples (except other blanks) in the SDG. Equipment and trip blanks should not be qualified based upon laboratory blank contamination.

II. FURTHER MANUAL REVIEW REQUIRED

A. Laboratory Blanks

If no error messages are reported, then no further manual review of the laboratory blanks data is required.

B. Equipment and Trip Blanks

1) The sampler or sampling paperwork must be consulted to determine which samples, if any, are designated as equipment or trip blanks and to determine which regular field samples are impacted by these equipment/trip blanks.

2) A manual review of the CADRE Data Summary Tables (or Form Is) for these blanks must be performed. The criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses must be used for evaluation.

3) Sections 2 and 3 of the Region I Blank Analysis Results Worksheets must be completed and included in the Data Validation Report.

C. Manual Review Required Due To Errors Detected by CADRE

1) Manual review is required if CADRE reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE Laboratory Blanks Report. The validator should refer to Section III to determine

the extent of manual review required. Sections 2 and 3 of the Region 1 Blanks Analysis Worksheets must be utilized to document the manual review.

III. **REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE**

CADRE can display six possible error messages for Laboratory Blanks.

A. **Possible Errors**

1) No instrument performance check found for sample.

   - CADRE will not evaluate laboratory blank contamination for the sample if it does not find an instrument performance check (BFB/DFTPP) for that sample. CADRE will list the affected sample(s).

2) No calibration found for sample.

   - CADRE will not evaluate laboratory blank contamination for the sample if it does not find an associated initial and/or continuing calibration for that sample. CADRE will list the affected sample(s).

3) Samples with no associated laboratory blank.

   - CADRE will display this message if there was no laboratory blank associated with the specific sample(s) listed.

4) Invalid laboratory blank. Blank qualified (R) during a previous qualification.

   - CADRE will not evaluate a laboratory blank if the blank has been considered as unusable for other quality control parameters. For instance, if the blank results have been rejected (R) due to low surrogate recoveries.

5) Missing laboratory blank information.

   - CADRE will not evaluate laboratory blank

contamination for a sample if there is information missing which is required to associate laboratory blank contamination with that sample (e.g., if the laboratory does not report the weight of the blank, then CADRE cannot calculate the 5x/10x blank contamination levels).

6) No laboratory blank samples.

- CADRE will display this error message if it does not detect any laboratory blanks in the whole electronic deliverable.

B. **Required Action**

1) A manual review of the CLP Data Package is required to determine if the associated instrument performance check is present. If the instrument performance check is missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated instrument performance check (IPC) and laboratory blank(s) that were not reviewed by CADRE must be performed using the criteria and actions outlined in the Region 1 Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (mod 11/88). The Region 1 Blanks Analysis Results Worksheets (all three sections) must be completed to document the manual validation.

2) A manual review of the CLP Data Package is required to determine if the associated calibration is present. If the associated calibration is missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated calibration and laboratory blank(s) that were not reviewed by CADRE must be performed using the criteria and actions outlined in the Region 1 Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (mod 11/88). The Region 1 Blanks Analysis Results Worksheets (all three sections) must be completed to document the manual validation.

3) If the laboratory blank has been rejected (R) during the review of other quality control parameters, then professional judgement must be used to determine if qualification of any positive hits in any samples associated with the invalid laboratory blank is necessary. The reviewer must provide justification for sample result qualification as per the Region 1 Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

4) For errors 4-6 listed in Section III above, a manual review of the CLP Data Package is required to determine if the associated laboratory blank information are present and meet laboratory blank acceptance criteria. If the associated laboratory blank information are missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated laboratory blank information that was not reviewed by CADRE must be performed using the criteria and actions outlined in the Region 1 Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (mod 11/88). The Region 1 Blanks Analysis Results Worksheets (all three sections) must be completed to document the manual validation.

IV. **STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION**

1) Complete any manual review as required in Section II.

2) Include the CADRE Laboratory Blanks Report and any required Region I Blank Analysis Results Worksheets after the CADRE Calibration Report and any required Region 1 Volatile Calibration Verification and/or Semivolatile Calibration Verification Worksheets in the Data Validation Report.

3) Discuss any qualifications placed upon sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region 1 Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Include, in the memorandum, a table of the maximum concentrations of contaminants found in the laboratory, equipment, and trip

blanks.

4) Include required qualifiers in the Recommendations Summary Table (Table I of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Tables as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses. (Raise sample results to the CRQL if necessary).

SECTION VII SMCs/SURROGATES

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

The CADRE SMC/Surrogate Report displays the criteria used for surrogate review. The criteria used by CADRE are identical to the criteria used by Region I. CADRE reviews the advisory BNA surrogates but does not qualify data based on advisory recoveries.

If all criteria are met, CADRE will display the message "No problems found for this qualification".

The CADRE SMC/Surrogate Report will indicate any samples which require qualification due to poor surrogate recoveries and will indicate the qualifiers to be placed on the sample results. The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables.

II. FURTHER MANUAL REVIEW REQUIRED

1) If no error messages are reported, then no further manual review is required.

2) Manual review is required if CADRE reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE SMC/Surrogate Report. The validator should refer to Section III to determine the extent of manual review required. Region 1 Surrogate Spike Recoveries Worksheets must be utilized to document the manual review.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

CADRE can display three possible error messages for SMCs/Surrogates.

A. **Possible Errors**

1) Sample dilution factor exceeds criteria.

- This message will appear if the sample is analyzed at greater than a 1:10 dilution. CADRE will only review SMC/surrogate recoveries for samples analyzed at less than or equal to a 1:10 dilution.

2) Missing surrogate (system monitoring compound) data.

- This message will appear if CADRE cannot locate surrogate/SMC recoveries in the electronic deliverable.

3) Surrogate (system monitoring compound) percent recovery in method blank exceeds criteria.

B. **Required Action**

1) A manual review of the Form 2 contained in the CLP Data Package is required for all samples analyzed at greater than a 1:10 dilution to determine if surrogate recoveries are within acceptance limits. Manual review of the associated SMC/surrogate information must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. A Region I Surrogate Spike Recoveries Worksheet must be utilized to document the manual review.

2) A manual review of the Form 2 contained in the CLP Data Package must be performed to determine if the associated SMC/surrogate information are present and meet SMC/surrogate acceptance criteria. Manual review of the associated SMC/surrogate information must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. A Region I Surrogate Spike Recoveries Worksheet must utilized to document the manual review.

3) A manual review of SMC/surrogate data contained in the CLP Data Package must be performed for all samples associated with a laboratory blank containing outlier surrogate recoveries. Professional judgement should be used to qualify any affected sample data due to outlier laboratory blank surrogate recoveries as the per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. The reviewer must provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

IV. **STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION**

1) Complete any manual review as required in Section II.

2) Include the CADRE SMC/Surrogate Report and any required Region I Surrogate Spike Recoveries Worksheets after the CADRE Laboratory Blanks Report and any required Region I Blank Analysis Results Worksheets in the Data Validation Report.

3) Discuss any qualifications placed upon sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Include, in the memorandum, a table listing any samples and surrogates which failed to meet the acceptance criteria.

4) Include required qualifiers in the Recommendations Summary Table (Table 1 of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Tables as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION VIII MATRIX SPIKE/MATRIX SPIKE DUPLICATE

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

The CADRE Matrix Spike Report displays the criteria used for matrix spike evaluation. The criteria used for evaluation are identical to the criteria used by Region I.

If all matrix spike criteria are met, CADRE displays the message "No problems were found for this qualification" on the Matrix Spike Report.

If all criteria are not met, CADRE will indicate whether the percent recovery or RPD criteria were not met on the Matrix Spike Report. CADRE will also list the individual compounds which exceeded criteria.

Where possible, CADRE will recommend specific qualifications for MS/MSD deviations. If CADRE cannot recommend specific qualifications, CADRE will alert the reviewer that criteria were not met and list what manual review is necessary.

CADRE does not apply qualifiers to the qualified or unqualified Data Summary Tables for MS/MSD percent recovery or RPD deviations. Where possible, CADRE recommends qualifications. For qualified and unqualified Data Summary Tables, the validator needs to place the qualifiers suggested by CADRE and/or resulting from any required manual validation onto both Data Summary Tables.

II. FURTHER MANUAL REVIEW REQUIRED

1) If CADRE indicates that recovery criteria were not met, then a manual review of Form 3 contained in the CLP Data Package must be performed to determine if any qualification of the data is required. The criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses must be used. Region I Matrix Spike/Matrix Spike Duplicate Worksheets must be completed and included with the Data Validation Report.

2) CADRE does not evaluate the unspiked compounds in the sample, MS, and MSD. This review must be performed

manually. The criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses must be used. Region I Matrix Spike/Matrix Spike Duplicate Worksheets for unspiked compounds must be completed and included with the Data Validation Report. A CADRE-generated Data Summary Table which contains the sample results for the sample, MS, and MSD is included in the CADRE report along with the Matrix Spike Report. This Data Summary Table may be used to aid in completing the Region I Matrix Spike/Matrix Spike Duplicate Worksheet for the evaluation of MS/MSD unspiked compounds.

3) Manual review is also required if CADRE detects and reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE Matrix Spikes Report. The validator should refer to Section III to determine the extent of manual review required. Region 1 Matrix Spike/Matrix Spike Duplicate Worksheets must be utilized to document the manual review.

III. **REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE**

CADRE can display three possible error messages for MS/MSDs.

A. **Possible Errors**

1) Matrix spike (MS) and matrix spike duplicate (MSD) frequency not sufficient.

   - CADRE will display this message on the Matrix Spike Report if an MS/MSD pair was not analyzed at the required frequency of 1 per 20 samples per matrix.

2) Missing matrix spike information.

   - CADRE will display this message on the Matrix Spike Report if some (or all) of the information necessary to evaluate this parameter is missing. This may include but is not limited to:

     - True value of matrix spike added.

- Matrix (soil or water).
- QC limits for % recovery and/or RPD.

3) No matrix spike data.

- CADRE will display this error message on the Matrix Spike Report if it cannot find any matrix spike sample(s) in the electronic deliverable.

B. **Required Action**

1) As required in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, professional judgement must be used to determine if there is any affect on the data due to the insufficient frequency of MS/MSD analysis. The reviewer must justify, in the Data Validation Memorandum, any action taken.

2) For errors 2 and 3 listed in Section III above, manual review of the CLP Data Package must be performed to determine if the associated matrix spike information are present and meet matrix spike acceptance criteria. If the associated matrix spike information are missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated matrix spike information must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Region I Matrix Spike/Matrix Spike Duplicate Worksheets must be utilized to document any manual review.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review as required in Section II.

2) Include the CADRE Matrix Spike Report and any required Region I Matrix Spike/Matrix Spike Duplicate Worksheets after the CADRE SMC/Surrogate Report and any required Region I Surrogate Spike Recoveries Worksheets in the Data Validation Report. The CADRE generated sample, MS,

and MSD Data Summary Table must be included along with all other matrix spike worksheets.

3) Discuss any qualifications placed on sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Include, in the memorandum, a table listing all compounds which did not meet the matrix spike acceptance criteria.

4) Include the required qualifiers in the Recommendations Summary Table (Table 1 of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Table as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION IX FIELD DUPLICATES

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

CADRE does not evaluate field duplicate samples.

II. FURTHER MANUAL REVIEW REQUIRED

1) The reviewer must check the Organic Traffic Report/Chain-of-Custody Form, consult the sampler, or refer to the CLP Sample Tracking System (CLPSTS) to determine which samples in the SDG are field duplicates and to determine which regular field samples are impacted by these field duplicate samples.

2) A manual review of the CADRE Data Summary Tables (or Form 1s) for the field duplicates must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Region I Field Duplicate Precision Worksheets must be completed and included with the Data Validation Report.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

No action is required as this parameter is not reviewed by CADRE.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review required in Section II.

2) Include the Region I Field Duplicate Precision Worksheet after the CADRE Matrix Spikes Report and any required Region I Matrix Spike/Matrix Spike Duplicate Worksheets in the Data Validation Report.

3) Discuss any qualifications placed on the sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

4) Include the required qualifiers in the Recommendations Summary Table (Table I of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

5) Apply qualifiers to sample results on the Data Summary Tables as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION X INTERNAL STANDARDS

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

The CADRE Internal Standards Report displays the criteria used by CADRE for evaluation of internal standards. The criteria used by CADRE are identical to the criteria used by Region I.

If all criteria are met, CADRE will display the message "No problems found for this qualification".

CADRE will list, on the Internal Standards Report, the compounds in each sample which have been qualified due to poor internal standard recoveries or retention times which are outside of criteria. CADRE will also list the appropriate Region 1 action to be taken. The CADRE qualified Data Summary Tables will contain the correct qualifiers. For unqualified Data Summary Tables, the validator needs only to place the qualifiers suggested by CADRE onto the unqualified Data Summary Tables.

II. FURTHER MANUAL REVIEW REQUIRED

1) If no error messages are reported, then no further manual review is required.

2) Manual review is required if CADRE detects and reports any of the error messages listed in Section III. If an error is detected by CADRE, CADRE will report this error on the CADRE Internal Standards Report. The validator should refer to Section III to determine the extent of manual review required. Region 1 Internal Standard Performance Worksheets must be utilized to document the manual review.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

CADRE can display three possible error messages for Internal Standards.

A. **Possible Errors**

1) Samples with no internal standard.

- CADRE will display this message on the Internal Standards Report if it cannot locate the internal standards for a sample in the electronic deliverable.

2) Missing internal standards.

- CADRE will display this message on the Internal Standards Report if no internal standards for the SDG were located by CADRE in the electronic deliverable. This may occur if either the laboratory did not include Form 8 in the electronic deliverable or if CADRE failed to import Form 8.

3) Missing internal standard information.

- CADRE will display this message if a portion of the required information for internal standard evaluation is not present. This may include but is not limited to the following reasons:

  - If only the internal standard area information for the sample and/or associated calibration standard is missing.

  - If only the retention time information for the sample and/or associated calibration standard is missing.

  - If CADRE cannot associate the internal standard information for a sample with a calibration standard (i.e. either the calibration standard information are missing or the instrument performance check associated with the sample or calibration standard is missing).

B. **Required Action**

If any of these error messages are reported, manual review of the CLP Data Package must be performed to determine if the associated internal standard(s) information is present in the CLP Data Package. If any of the necessary information is missing from the CLP Data Package, then the laboratory must be contacted to submit the information. Manual review of the associated internal standard information must be performed using the criteria and actions outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Region 1 Internal Standard Performance Worksheets must be utilized to document the manual review.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review as required in Section II.

2) Include the CADRE Internal Standards Report and any required Region I Internal Standard Performance Worksheets after the Field Duplicate Precision Worksheets in the Data Validation Report.

3) Discuss any qualifications placed upon sample results in the Data Validation Memorandum and provide justification for sample result qualification as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

4) Include required qualifiers in the Recommendations Summary Table (Table I of the Data Validation Memorandum) as per the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

SECTION XI TENTATIVELY IDENTIFIED COMPOUNDS (TICs)

I. INTERPRETATION OF THE CADRE WORKSHEET AND FINDINGS

CADRE does not review Tentatively Identified Compound (TIC) results. Review of TIC results is not required for Tier II data validation. It is, however, required for Tier III data validation.

II. FURTHER MANUAL REVIEW REQUIRED

For Tier III data validation, the procedure stated in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses must be followed to evaluate Tentatively Identified Compounds.

For Tier II data validation, verify that target compounds are not reported as TICs in another fraction. Only a tabular summary of the detected TICs is required.

A tabular TIC summary should be included in the Data Validation Memorandum for both Tier II and Tier III data validations.

III. REQUIRED ACTION IF ERRORS ARE DETECTED BY CADRE

No action is required, as this parameter is not reviewed by CADRE.

IV. STEPS REQUIRED TO COMPLETE THE REGION I VALIDATION

1) Complete any manual review as required in Section II.

2) For Tier II data validation, summarize the TICs found in the TIC Summary Table in the Data Validation Report. Verify that target compounds are not reported as TICs in another fraction. No further review of TICs is to be performed.

3) Provide an explanation of any identifications which were changed by the reviewer or any TICs which were not included in the TIC Summary Table. The reviewer must justify any changes to the TIC results in the Data Validation Memorandum. This step is required only for Tier III data validation.

SECTION XII COMPLETING THE DATA VALIDATION REPORT

1) The Data Validation Memorandum must be completed as stated in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (Section 1.5). The completed CADRE Data Review Inventory Sheet must be included in the Data Validation Report.

2) Any manual review of the CLP Data Package which was performed must be documented by completing the Region I Data Validation worksheets. These worksheets should be included in the Data Validation Report along with the CADRE worksheets in the order specified by this guidance document.

3) The CADRE Data Summary Tables must be completed. The CADRE qualified Data Summary Tables for the volatile and semivolatile fractions will be provided to the Field Sampling Contractor. These Data Summary Tables will contain qualifiers recommended by CADRE on the CADRE worksheets generated during the review of each QC parameter. The Field Sampling Contractor will be required to verify that all qualifiers have been correctly transcribed onto the qualified Data Summary Tables by CADRE. The Field Sampling Contractor will also be required to place any qualifiers onto the Data Summary Tables which result from any required manual validation.

   For the pesticide/PCB fractions and in instances where major discrepancies exist between the sample values reported on the CADRE qualified Data Summary Tables and the laboratory Form 1s, the CADRE unqualified Data Summary Tables will be sent to the Field Sampling Contractors along with a notice that the Data Summary Tables are unqualified. The unqualified Data Summary Tables distributed to the Field Sampling Contractors will contain only the Form I results and qualifiers as reported by the laboratory. The Field Sampling Contractors will be required to remove all laboratory qualifiers from the CADRE unqualified Data Summary Tables (such as the "B", "D", and "E" qualifiers), with the exception of the "J" qualifier, for results detected below the CRQL, and the "U" qualifier, for non-detect

results. The Field Sampling Contractor must then add any qualifiers resulting from the completion of the data validation.

ATTACHMENT I

Completed CADRE and Region I Data Validation Worksheets

For a hardcopy of this Attachment contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT II

Blank Region I Data Validation Worksheets

For a hardcopy of this Attachment contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT III

Manual Review Necessary to Complete a Tier II Data Validation

SUMMARY OF CADRE DATA REVIEW - REQUIRED MANUAL REVIEW NECESSARY TO COMPLETE A REGION I TIER II DATA VALIDATION

QC CRITERIA	REVIEW PERFORMED BY CADRE	REQUIRED MANUAL REVIEW
DATA COMPLETENESS	● CADRE lists any errors or omissions which were detected in the electronic deliverable. These errors are corrected prior to data review. ● CADRE does not detect errors or omissions in the raw data.	● The Complete SDG File (CSF) form must be completed and signed by the reviewer. All supporting documentation must be present in the data package. The reviewer must request from the laboratory any data missing from the data package (For example if a hardcopy Form I is missing). The Region I Data Completeness Worksheet must be completed and included in the Data Validation Report along with any records of communication. ● The CADRE Data Review Inventory Sheet must be completed and signed by the reviewer. The reviewer must request from the EPA Data Validation Chemist any information missing from the CADRE report sent by ESAT. The CADRE Data Review Inventory Sheet must be included in the Data Validation Report.
HOLDING TIMES	● CADRE reviews holding times for waters and soils as per Region I guidelines.	● If CADRE detects no errors, no further manual review is required. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.
PERCENT SOLID	● CADRE evaluates percent solid content as per Region 1 Guidelines.	● If CADRE detects no errors, no further manual review is required. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.
GC/MS TUNING	● CADRE evaluates GC/MS tunes based on current Region I guidelines.	● If CADRE detects no errors, no further manual review is required. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.

SUMMARY OF CADRE DATA REVIEW - REQUIRED MANUAL REVIEW NECESSARY TO COMPLETE A REGION I TIER II DATA VALIDATION

QC CRITERIA	REVIEW PERFORMED BY CADRE	REQUIRED MANUAL REVIEW
CALIBRATIONS	● CADRE reviews calibration criteria based on current Region 1 Guidelines.	● If CADRE detects no errors, no further manual review is required. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.
BLANKS	● CADRE reviews and qualifies results for laboratory blanks based on current Region I guidelines. ● CADRE capability to review equipment or trip blanks is currently not being used.	● If CADRE detects no errors, no further manual review is required. ● A manual review of all equipment and trip blanks is necessary to assess contamination. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.
SURROGATES	● CADRE reviews and qualifies surrogate data based on current Region I guidelines. CADRE reviews the advisory surrogates for BNA but does not qualify data for outlier advisory surrogate recoveries. ● CADRE does not review surrogate recoveries if samples were analyzed at a dilution greater than 1:10.	● If CADRE detects no errors or if samples were analyzed at a dilution less than or equal to 1:10, no further manual review is required. ● A manual review of Form 2 is necessary for all samples analyzed at a dilution greater than 1:10. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.

SUMMARY OF CADRE DATA REVIEW - REQUIRED MANUAL REVIEW NECESSARY TO COMPLETE A REGION I TIER II DATA VALIDATION

QC CRITERIA	REVIEW PERFORMED BY CADRE	REQUIRED MANUAL REVIEW
MATRIX SPIKE	● CADRE reviews matrix spike data based on current Region I guidelines. ● CADRE does not qualify data for matrix spikes, but indicates which compounds did not meet matrix spike acceptance criteria.	● If all criteria were met for %R and RPD or if CADRE detects no errors, no further manual review is required for %R and RPD. ● If recovery criteria were exceeded, a manual review of Form 3 is necessary to determine if qualification of the data is required ● Manual review of the CADRE sample, MS, MSD Summary Table is required to assess the %RSD of unspiked compounds. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document. ● CADRE does not qualify results for MS/MSD deviations on the CADRE qualified Data Summary Tables. The validator needs to apply qualifiers to both the qualified and unqualified Data Summary Tables for any MS/MSD deviations.
FIELD DUPLICATES	● CADRE does not evaluate field duplicates.	● A manual review of the CADRE Data Summary Tables (or Form 1s) for the field duplicates is required to assess precision.
INTERNAL STANDARDS	● CADRE evaluates primary internal standard criteria based on the current Region I guidelines. For the criteria of "extremely low" areas counts, CADRE assigns a defined value of less than 20% of the internal standard area of the associated calibration.	● If CADRE detected no errors, no further manual review is required. ● Manual review is required if CADRE reports any errors during data review. Required review is stated in Section III of Guidance Document.
TENTATIVELY IDENTIFIED COMPOUNDS	● CADRE does not evaluate this criterion.	● No manual review for TICs is required for Tier II. A table summarizing the TICs detected must be completed. The reviewer must verify that target compounds are not reported as TICs in another fraction.

SUMMARY OF CADRE DATA REVIEW - REQUIRED MANUAL REVIEW NECESSARY TO COMPLETE A REGION I TIER II DATA VALIDATION

QC CRITERIA	REVIEW PERFORMED BY CADRE	REQUIRED MANUAL REVIEW
COMPOUND IDENTIFICATION AND QUANTITATION	● CADRE does not check any raw data but where possible does check and verify calculations and rounding procedures. ● If CADRE detects errors in calculations and rounding, CADRE will generate an error form and suggest its correct result. However, for consistency between the hardcopy and electronic data deliverables, the laboratory Form 1 result will be reported by the ESAT CADRE Chemist if the error is due to rounding. ● CADRE produces a worksheet which lists all compounds detected less than the CRQL.	● No manual review is required. Evaluation of compound Identification and Quantitation is not required for Tier II data validation.
OVERALL ASSESSMENT OF DATA FOR A CASE	● CADRE reviews each parameter independent of other parameters.	● No manual review is required. An overall summary of data qualifications should be included at the end of the Data Validation Memorandum as outlined in the Region I Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses.

ATTACHMENT IV

Criteria Comparison Between CADRE, National Functional Guidelines and Region I Functional Guidelines

CADRE METHOD COMPARISON
SEMI-VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
HOLDING TIMES	Yes	● Extraction: soil and water samples within 7 days of collection. ● Analytical: Both within 40 days of extraction. ● Action: (J) positive results and (UJ) non-detects. If holding time is grossly exceeded, may need to (R) non-detects.	● Extraction: **Primary:** water and soil samples within 7 days of collection. **Expanded:** water and soil samples within 14 days of collection. ● Analytical: **Primary:** both within 40 days of extraction. **Expanded:** both within 60 days of extraction. ● Action: (J) positive results and (UJ) non-detects outside primary criteria. (J) positive results and (R) non-detects outside expanded criteria.	● Extraction: water samples within 7 days of collection. Soil samples recommended within 14 days of collection. ● Analytical: Both within 40 days of extraction. ● Action: (J) positive results and (UJ) quantitation limits. If HT grossly exceeded, may need to (R) non-detects.
GC/MS TUNING	Yes	DFTPP m/z Ion Abundance Criteria 51 30.0 - 80.0% of m/z 198 68 less than 2.0% of m/z 69 69 present 70 less than 2.0% of m/z 69 127 25.0 - 75.0% of m/z 198 197 less than 1.0% of m/z 198 198 base peak, 100% relative abundance 199 5.0-9.0% of m/z 198 275 10.0-30.0% of m/z 198 365 greater than 0.75% of m/z 198 441 present, but less than 443 442 40.0-110% of m/z 198 443 15.0-24.0% of m/z 442 ● 12 hour tune period ● Action: (R) all data if mass calibration is in error. ● If ion abundance criteria not met, use professional judgement to determine if qualification is necessary. ● Ion abundance criteria used should reflect the most current version of the SOW.	DFTPP m/z Ion Abundance Criteria 51 30.0 - 80.0% of m/z 198 68 less than 2.0% of m/z 69 69 present 70 less than 2.0% of m/z 69 127 25.0 - 75.0% of m/z 198 197 less than 1.0% of m/z 198 198 base peak, 100% relative abundance 199 5.0-9.0% of m/z 198 275 10.0-30.0% of m/z 198 365 greater than 0.75% of m/z 198 441 present, but less than 443 442 40.0-110% of m/z 198 443 15.0-24.0% of m/z 442 ● 12 hour tune period. ● Action: Manual review of data is necessary to determine usability of data if mass calibration is in error, ion abundance criteria are not met, or if m/z 198 is not base peak. ● Will alert validator to perform manual review if it cannot associate samples with a tune file.	DFTPP m/z Ion Abundance Criteria 51 30.0 - 80.0% of m/z 198 68 less than 2.0% of m/z 69 69 present 70 less than 2.0% of m/z 69 127 25.0 - 75.0% of m/z 198 197 less than 1.0% of m/z 198 198 base peak, 100% relative abundance 199 5.0-9.0% of m/z 198 275 10.0-30.0% of m/z 198 365 greater than 0.75% of m/z 198 441 present, but less than 443 442 40.0-110% of m/z 198 443 15.0-24.0% of m/z 442 ● 12 hour tune period ● Action: (R) all data if mass calibration is in error. Use judgement if ion abundance criteria are not met.
INITIAL CALIBRATION	Yes	● All average RRFs must be ≥ 0.05. ● All %RSDs must be ≤ 30%. ● Action: If RRF < 0.05, (J) all positive results for that compound in samples associated with the initial and subsequent continuing calibrations. (R) non-detects for that compound in samples associated as mentioned. If %RSD > 30%, (J) positive results for that compound in associated samples. If %RSD > 50%, also (UJ) non-detects.	● All average RRFs must be ≥ 0.05. ● All %RSDs must be ≤ 30%. ● Action: If RRF < 0.05, (J) all positive results for that compound in samples associated with the initial and subsequent continuing calibrations. (R) non-detects for that compound in samples associated as mentioned. If %RSD > 30%, (J) positive results for that compound in associated samples. If %RSD > 50%, also (UJ) non-detects.	● Minimum RRF criteria specified per compound. ● %RSDs must be ≤ 20.5%. ● 19 selected compounds have no %RSD criteria, but RRFs must be ≥ 0.01. ● Action: RRF criteria judged in conjunction with the RSD. Professional judgement used to qualify data when RRF < minimum criteria. Data are qualified due to non-compliant %RSD criteria based on if the high, low or middle part of curve is out.

CADRE METHOD COMPARISON Continued
SEMI-VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
CONTINUING CALIBRATION	Yes	● RRFs must be ≥ 0.05. ● %Ds must be ≤ 25.0%. ● Action: If RRF < 0.05, (J) positive results and (R) non-detects for that compound in samples associated with the calibration. If %D > 25, (J) positive results for that compound in associated samples. If %D > 50, also (UJ) non-detects for that compound in associated samples.	● RRFs must be ≥0.05. ● %Ds must be ≤25. ● Action: If RRF < 0.05, (J) positive results and (R) non-detects for that compound in samples associated with the calibration. If %D > 25, (J) positive results for that compound in associated samples. If %D > 50, also (UJ) non-detects for that compound in associated samples.	● RRF criteria specified per compound. ● %Ds must be ≤ 25%. ● Selected compounds have no %D criteria but RRFs must be ≥ 0.01. ● Action: If RRF is between 0.01 and acceptance criteria, or above 0.01 for the selected compounds, (J) positive results and (UJ) non-detect results. If RRF < 0.01, (R) non-detects and (J) positive results with acceptable mass spectrum. If %D > 50, (J) positive results. Use professional judgement for non-detects.
BLANKS	Yes	● No contaminants should be present. ● One blank per matrix, concentration level and extraction batch. ● Equipment blanks are treated as method blanks. ● Action: If contaminant also detected in sample, qualify data (U) at the CRQL if concentration is < 5x blank level (10x for common lab contaminants (CLCs) and less than CRQL. Qualify (U) at raised detection limit if concentration is < 5x blank level (10x for CLCs) and greater than CRQL. Actions apply to any blank contamination (method or equipment).	● No contaminants should be present. ● One blank per matrix, concentration level and extraction batch. ● Equipment blanks are not validated by CADRE. ● Action: If contaminant also detected in sample, qualify data (U) at the CRQL if concentration is < 5x blank level (10x for common lab contaminants (CLCs) and less than CRQL. Qualify (U) at raised detection limit if concentration is < 5x blank level (10x for CLCs) and greater than CRQL. Actions apply only to samples associated with the contaminated blank (from Form 4).	● No contaminants should be present. ● One blank per matrix, concentration level and extraction batch. ● Field blanks are treated as method blanks. ● Lab Blank Action: If contaminant also detected in sample, qualify data (U) at the CRQL if concentration is < 5x blank level (10x for common lab contaminants (CLCs) and less than CRQL. Qualify (U) at raised detection limit if concentration is < 5x blank level (10x for CLCs) and greater than CRQL. ● Field blanks should not contain any TCL above its CRQL and should not contain the common phthalate contaminants at concentrations > 2x CRQL. ● Should not contain TICs. ● Field Blank Action: Qualify the same as for Region I Functional Guidelines except use only 5x level.

CADRE METHOD COMPARISON Continued
SEMI-VOLATILES

	Eval by Tier II	Region I Functional Guidelines modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
SURRO-GATES	Yes	% Recovery Soil Aqueous Nitrobenzene-d_5 23-120 35-114 2-Fluorobiphenyl 30-115 43-116 Terphenyl-d_{14} 18-137 33-141 Phenol-$_{d6}$ 24-113 10-94 2-Fluorophenol 25-121 21-100 2,4,6-Tribromophenol 19-122 10-123 ● Action: If at least 2 surrogates in a base/neutral or acid fraction are > 10%, but < CRR, (J) positive results and (UJ) quantitation limits in the sample. If < 10%, (J) positive results and (R) non-detect results in the sample. If recoveries are > CRR (J) positive results only.	% Recovery Soil Aqueous Nitrobenzene-d_5 23-120 35-114 2-Fluorobiphenyl 30-115 43-116 Terphenyl-d_{14} 18-137 33-141 Phenol-d_5 24-113 10-110 2-Fluorophenol 25-121 21-110 2,4,6-Tribromophenol 19-122 10-123 2-Chlorophenol-d_4 20-130 33-110 1,2-Dichlorobenzene-d_4 20-130 16-110 ● Action: If at least 2 surrogates in a base/neutral or acid fraction are > 10%, but < CRR, (J) positive results and (UJ) quantitation limits in the sample. If < 10%, (J) positive results and (R) non-detect results in the sample. If recoveries are > CRR (J) positive results only. ● CADRE does not take into consideration advisory surrogate recoveries when qualifying data. (i.e. does not include them as one of the two outlier surrogates required for qualification). ● CADRE will not evaluate surrogate recoveries on a sample if a dilution greater than 1:10 was performed on that sample.	% Recovery Soil Aqueous Nitrobenzene-d_5 23-120 35-114 2-Fluorobiphenyl 30-115 43-116 Terphenyl-d_{14} 18-137 33-141 Phenol-d_5 24-113 10-110 2-Fluorophenol 25-121 21-110 2,4,6-Tribromophenol 19-122 10-123 2-Chlorophenol-d_4 20-130 33-110 1,2-Dichlorobenzene-d_4 20-130 16-110 ● Action: If at least 2 surrogates in a base/neutral or acid fraction are out, but > 10%, (J) positive results for that fraction (B/N, or A) and (UJ) quantitation limits for that fraction. If < 10%, (J) positive results for that fraction and (R) or (J) non-detect results for that fraction.
MS/MSD	Yes	% Recovery Soil RPD Aqueous RPD Phenol 26-90 35 12-89 42 2-Chlorophenol 25-102 50 27-123 40 1,4-Dichlorobenzene 28-104 27 36-97 28 N-Nitroso-di-n-prop. 41-126 38 41-116 38 1,2,4-Trichlorobenzene 38-107 23 39-98 28 4-Chloro-3-methylphenol 26-103 33 23-97 42 Acenaphthene 31-137 19 46-118 31 4-Nitrophenol 11-114 50 10-80 50 2,4-Dinitrotoluene 28-89 47 24-96 38 Pentachlorophenol 17-109 47 9-103 50 Pyrene 35-142 36 26-127 31 ● Action: If recoveries are greater than limits (J) positive results in unspiked sample. If recoveries are less than limits but ≥ 10%, (J) positive results in unspiked sample. If recoveries are < 10%, (J) positive results and (R) non-detect results in unspiked sample. Use professional judgement to qualify results for high RSD between sample, MS, and MSD for non-spike compounds.	% Recovery Soil RPD Aqueous RPD Phenol 26-90 35 12-89 42 2-Chlorophenol 25-102 50 27-123 40 1,4-Dichlorobenzene 28-104 27 36-97 28 N-Nitroso-di-n-prop. 41-126 38 41-116 38 1,2,4-Trichlorobenzene 38-107 23 39-98 28 4-Chloro-3-methylphenol 26-103 33 23-97 42 Acenaphthene 31-137 19 46-118 31 4-Nitrophenol 11-114 50 10-80 50 2,4-Dinitrotoluene 28-89 47 24-96 38 Pentachlorophenol 17-109 47 9-103 50 Pyrene 35-142 36 26-127 31 ● Action: CADRE performs this evaluation but does not qualify sample results which did not meet criteria. CADRE does list which compounds failed criteria.	% Recovery Soil RPD Aqueous RPD Phenol 26-90 35 12-89 42 2-Chlorophenol 25-102 50 27-123 40 1,4-Dichlorobenzene 28-104 27 36-97 28 N-Nitroso-di-n-prop. 41-126 38 41-116 38 1,2,4-Trichlorobenzene 38-107 23 39-98 28 4-Chloro-3-methylphenol 26-103 33 23-97 42 Acenaphthene 31-137 19 46-118 31 4-Nitrophenol 11-114 50 10-80 50 2,4-Dinitrotoluene 28-89 47 24-96 38 Pentachlorophenol 17-109 47 9-103 50 Pyrene 35-142 36 26-127 31 ● Action: Use professional judgement to determine need for qualification of the data.

CADRE METHOD COMPARISON (Continued)
SEMI-VOLATILES

	Evaluated By Tier II?	Region I Functional Guidelines modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft 6/91
FIELD DUPLICATES	Yes	● RPD for aqueous samples must be < 30%. For soils, RPD must be < 50%. ● Action: If RPD limits are exceeded, (J) positive results for that compound in both samples. Use professional judgement to qualify all samples of the same matrix.	● Presently, CADRE does not evaluate field duplicates. CADRE does recognize field duplicate pairs but does not have an evaluation parameter. This option is scheduled for implementation in future releases.	● Criteria determined by each Region. ● Action: Action must be in accordance with Regional specifications.
INTERNAL STANDARDS	Yes	● Area counts may not vary by more than a factor of 2 from the associated calibration standard. The RTs may not shift more than 30 seconds. ● Action: If area counts are out, (J) positive results and (UJ) non-detect results for compounds quantitated using that internal standard. If area counts are extremely low, non-detects should be rejected (R). Use professional judgement if RT shifts by more than 30 seconds.	● **Primary:** Area counts may not vary by more than a factor of 2 from the associated calibration standard. The RTs may not shift more than 30 seconds. **Expanded:** areas counts may not decrease by more than a factor of 5. ● Action: If area counts are out of primary criteria low, (J) positive results and (UJ) non-detect results for compounds quantitated using that internal standard. If areas counts are out high, (J) positive results associated with that internal standard. If area counts are outside expanded criteria (J) positive results and (R) non-detects associated with that internal standard. Manual validation is required to qualify data for retention time shifts of more than 30 seconds.	● Area counts may not vary by more than a factor of 2 from the associated calibration standard. The RTs may not shift more than 30 seconds. ● Action: If area counts are out, (J) positive results and (UJ) non-detect results for compounds quantitated using that internal standard. If area counts are extremely low, non-detects should be rejected (R). Use professional judgement if RT shifts by more than 30 seconds.
TCL COMPOUND IDENTIFICATION	No	● Compound must be within ± 0.06 RRT units of the standard RRT. ● Mass spectra of the sample compound and a current lab generated standard must match according to specific ion criteria. ● Action: Use professional judgement.	● CADRE does not evaluate this criterion.	● Compound must be within ± 0.06 RRT units of the standard RRT. ● Mass spectra of the sample compound and a current lab generated standard must match according to specific ion criteria. These criteria are the same as those listed in the Region I Functional Guidelines. ● Action: Use professional judgement.

CADRE METHOD COMPARISON (Continued)
SEMI-VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft 6/91
COMPOUND QUANTITATION AND CRQLs	CRQLs Yes Compound Quantitation No	● See attached CRQL list for current soil and water CRQLs for each compound. ● RRF must be calculated based on internal standard specified in appropriate SOW. ● Quantitation must be based on the specific quantitation ion listed in the SOW. ● Quantitation is performed using the RRF obtained from the daily calibration standard. ● Action: Professional judgement is used to determine if reported compounds are false positives or if false negatives are reported.	● CADRE prepares a report listing all compounds which were reported below the CRQL and flags all those results as (J). ● CADRE does not evaluate raw data, therefore, cannot verify spectra.	● See CRQL list. ● RRF must be calculated based on internal standard specified in SOW OLM01. ● Quantitation must be based on the specific quantitation ion listed in the SOW. ● Quantitation is performed using the RRF obtained from the daily calibration standard. ● Action: Professional judgement is used to determine if reported compounds are false positives or if false negatives are reported.
TENTATIVELY IDENTIFIED COMPOUNDS	Yes	● Must report possible identity if the 20 largest non-TCL or surrogate peaks which have area counts > 10% of nearest IS. ● Action: Use professional judgement to determine if proper identifications or classifications have been made. TICs summarized in tabular format.	● CADRE does not evaluate nor list a summary of the reported TICs even though they are included on a reporting form.	● Must report possible identity if the 20 largest non-TCL or surrogate peaks which have area counts > 10% of nearest IS. ● Action: Use professional judgement to determine if proper identifications or classifications have been made.
SYSTEM PERFORMANCE	No	● Use professional judgement to evaluate effects of poor chromatography, abrupt shifts in baseline peak tailing or splitting, etc.	● CADRE does not evaluate this criterion. CADRE reports the findings of each parameter independent of other QC parameters.	● Use professional judgement to evaluate effects of poor chromatography, abrupt shifts in baseline peak failing or splitting, etc.

CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)
SEMI-VOLATILES

	Region I		CADRE		National Functional Guidelines	
Analyte	Water µg/L	Soil µg/Kg	Water µg/L	Soil µg/Kg	Water µg/L	Soil µg/Kg
1,2,4-Trichlorobenzene	10	330	10	330	10	330
1,2-Dichlorobenzene	10	330	10	330	10	330
1,3-Dichlorobenzene	10	330	10	330	10	330
1,4-Dichlorobenzene	10	330	10	330	10	330
2,2'-oxybis(1-Chloropropane)	10	330	10	330	10	330
2,4,5-Trichlorophenol	25	800	25	800	25	800
2,4,6-Trichlorophenol	10	330	10	330	10	330
2,4-Dichlorophenol	10	330	10	330	10	330
2,4-Dimethylphenol	10	330	10	330	10	330
2,4-Dinitrophenol	25	800	25	800	25	800
2,4-Dinitrotoluene	10	330	10	330	10	330
2,6-Dinitrotoluene	10	330	10	330	10	330
2-Chloronaphthalene	10	330	10	330	10	330
2-Chlorophenol	10	330	10	330	10	330
2-Methylnaphthalene	10	330	10	330	10	330
2-Methylphenol	10	330	10	330	10	330
2-Nitroaniline	25	800	25	800	25	800
2-Nitrophenol	10	330	10	330	10	330
3,3'-Dichlorobenzidine	10	330	10	330	10	330
3-Nitroaniline	25	800	25	800	25	800
4,6-Dinitro-2-Methylphenol	25	800	25	800	25	800
4-Bromophenyl-Phenylether	10	330	10	330	10	330
4-Chloro-3-Methylphenol	10	330	10	330	10	330
4-Chloroaniline	10	330	10	330	10	330
4-Chlorophenyl-Phenylether	10	330	10	330	10	330
4-Methylphenol	10	330	10	330	10	330
4-Nitroaniline	25	800	25	800	25	800
4-Nitrophenol	25	800	25	800	25	800
Acenaphthene	10	330	10	330	10	330
Acenaphthylene	10	330	10	330	10	330
Anthracene	10	330	10	330	10	330
Benzo(a)anthracene	10	330	10	330	10	330
Benzo(a)pyrene	10	330	10	330	10	330
Benzo(b)fluoranthene	10	330	10	330	10	330
Benzo(g,h,i)perylene	10	330	10	330	10	330
Benzo(k)fluoranthene	10	330	10	330	10	330
bis(2-Chloroethoxy)methane	10	330	10	330	10	330

CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) Continued
SEMI-VOLATILES

	Region I		CADRE		National Functional Guidelines	
Analyte	Water µg/L	Soil µg/Kg	Water µg/L	Soil µg/Kg	Water µg/L	Soil µg/Kg
bis(2-Chloroethyl)ether	10	330	10	330	10	330
bis(2-ethylhexyl)phthalate	10	330	10	330	10	330
Butylbenzylphthalate	10	330	10	330	10	330
Carbazole	10	330	10	330	10	330
Chrysene	10	330	10	330	10	330
Di-n-butylphthalate	10	330	10	330	10	330
Di-n-octylphthalate	10	330	10	330	10	330
Dibenz(a,h)anthracene	10	330	10	330	10	330
Dibenzofuran	10	330	10	330	10	330
Diethylphthalate	10	330	10	330	10	330
Dimethylphthalate	10	330	10	330	10	330
Fluoranthene	10	330	10	330	10	330
Fluorene	10	330	10	330	10	330
Hexachlorobenzene	10	330	10	330	10	330
Hexachlorobutadiene	10	330	10	330	10	330
Hexachlorocyclopentadiene	10	330	10	330	10	330
Hexachloroethane	10	330	10	330	10	330
Indeno(1,2,3,cd)pyrene	10	330	10	330	10	330
Isophrone	10	330	10	330	10	330
N-Nitroso-di-n-propylamine	10	330	10	330	10	330
N-Nitrosodiphenylamine (1)	10	330	10	330	10	330
Naphthalene	10	330	10	330	10	330
Nitrobenzene	10	330	10	330	10	330
Pentachlorophenol	25	800	25	800	25	800
Phenanthrene	10	330	10	330	10	330
Phenol	10	330	10	330	10	330
Pyrene	10	330	10	330	10	330

CADRE METHOD COMPARISON
VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines Modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
HOLDING TIMES	Yes	● Unpreserved water: Aromatic within 7 days, non-aromatic within 14 days of sample collection. ● Preserved water and soil: both within 14 days of sample collection. ● Action: (J) positive results and (UJ) non-detects. Use professional judgement when (R) rejecting data for grossly exceeded holding times.	● Unpreserved waters: **Primary:** within 7 days of collection for aromatics and 14 days of collection for non-aromatics. **Expanded:** within 14 days of collection for aromatics and 28 days of collection for non-aromatics. ● Preserved waters and soils: **Primary:** within 14 days of collection for all compounds. **Expanded:** within 28 days of collection for all compounds. ● Action: (J) positive results and (UJ) non-detects outside primary criteria. (J) positive results and (R) non-detects outside expanded criteria.	● Unpreserved & Preserved: Both water and soil within 14 days of sample collection. ● Action: (J) positive results, (UJ) non-detects. Use professional judgement when (R) rejecting data.
GC/MS TUNING	Yes	Bromofluorobenzene m/z Ion Abundance Criteria 50 8.0-40.0% of base peak 75 30.0-66.0% of base peak 95 base peak, 100% relative abundance 96 5.0-9.0% of base peak 173 < 2.0% of m/z 174 174 50-120% of m/z 95 175 4.0-9.0% of m/z 174 176 > 93.0% but < 101.0% of m/z 174 177 5.0-9.0% of m/z 176 ● 12 hour tune period. ● Action: (R) all data if mass calibration is in error. ● If ion abundance criteria not met, use professional judgement to determine if qualification is necessary. ● Ion abundance criteria used should reflect the most current version of the SOW.	Bromofluorobenzene m/z Ion Abundance Criteria 50 8.0-40.0% of m/z 95 75 30.0-66.0% of m/z 95 95 base peak, 100% relative abundance 96 5.0-9.0% of m/z 95 173 < 2.0% of m/z 174 174 > 50.0%-120.0% of m/z 95 175 4.0-9.0% of m/z 174 176 > 93.0%-101.0% of m/z 174 177 5.0-9.0% of m/z 176 ● 12 hour tune period. ● Action: Manual review of data is necessary to determine usability of data if mass calibration is in error or ion abundance criteria are not met. ● Will alert validator to perform manual review of the data if it cannot associate samples with a tune file.	Bromofluorobenzene m/z Ion Abundance Criteria 50 8.0-40.0% of m/z 95 75 30.0-66.0% of m/z 95 95 base peak, 100% relative abundance 96 5.0-9.0% of m/z 95 173 < 2.0% of m/z 174 174 > 50.0%-120.0% of m/z 95 175 4.0-9.0% of m/z 174 176 > 93.0%-101.0% of m/z 174 177 5.0-9.0% of m/z 176 ● 12 hour tune period. ● Action: (R) all data if mass calibration is in error. Use expanded criteria if necessary.

CADRE METHOD COMPARISON Continued
VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines Modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
INITIAL CALIBRATION	Yes	● All average RRFs must be ≥ 0.05. ● All %RSDs must be ≤ 30%. ● Action: If RRF < 0.05, (J) all positive results for that compound in samples associated with the initial and subsequent continuing calibrations. (R) non-detects for that compound in samples associated as mentioned. If %RSD > 30%, (J) positive results for that compound in associated samples. If %RSD > 50%, also (UJ) non-detects.	● All average RRFs must be ≥ 0.05. ● All %RSDs must be ≤ 30%. ● Action: If RRF < 0.05, (J) all positive results for that compound in samples associated with the initial and subsequent continuing calibrations. (R) non-detects for that compound in samples associated as mentioned. If %RSD > 30%, (J) positive results for that compound in associated samples. If %RSD > 50%, also (UJ) non-detects.	● Minimum RRF criteria specified per compound. ● %RSDs must be ≤ 20.5%. ● Acetone, 2-Butanone, Carbon Disulfide, Chloroethane, Chloromethane, 1,2-Dichloroethane(total)have no %RSD criteria, but RRFs must be ≥ 0.01. ● Action: RRF criteria judged in conjunction with the RSD. Professional judgement used to qualify data when RRF < minimum criteria. Data are qualified due to non-compliant %RSD criteria based on if the high, low or middle part of curve is out.
CONTINUING CALIBRATION	Yes	● RRFs must be ≥ 0.05. ● %Ds must be ≤ 25.0%. ● Action: If RRF < 0.05, (J) positive results and (R) non-detects for that compound in samples associated with the calibration. If %D > 25, (J) positive results for that compound in associated samples. If %D > 50, also (UJ) non-detects for that compound in associated samples.	● RRFs must be ≥0.05. ● %Ds must be ≤25. ● Action: If RRF < 0.05, (J) positive results and (R) non-detects for that compound in samples associated with the calibration. If %D > 25, (J) positive results for that compound in associated samples. If %D > 50, also (UJ) non-detects for that compound in associated samples.	● RRF criteria specified per compound. ● %Ds must be ≤ 25%. ● Selected compounds have no %D criteria but RRFs must be ≥ 0.01. ● Action: If RRF is between 0.01 and acceptance criteria, or above 0.01 for the selected compounds, (J) positive results and (UJ) non-detect results. If RRF < 0.01, (R) non-detects and (J) positive results with acceptable mass spectrum. If %D > 50, (J) positive results. Use professional judgement for non-detects.

CADRE METHOD COMPARISON Continued
VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines Modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
BLANKS	Yes	● No contaminants should be present. ● One method blank per matrix per concentration level per 12 hour tune per system. ● Equipment and trip blanks are treated as method blanks. ● Action: If contaminant also detected in sample, qualify data (U) at the CRQL if concentration is < 5x blank level (10x for common lab contaminants (CLCs) and less than CRQL. Qualify (U) at raised detection limit if concentration is < 5x blank level (10x for CLCs) and greater than CRQL. Actions apply to any blank contamination (method, equipment, or trip).	● No contaminants should be present. ● One method blank per matrix per concentration level per 12 hour tune per system. ● Equipment and trip blank contamination is not validated by CADRE. ● Action: If contaminant also detected in sample, qualify data (U) at the CRQL if concentration is < 5x blank level (10x for common lab contaminants (CLCs) and less than CRQL. Qualify (U) at raised detection limit if concentration is < 5x blank level (10x for CLCs) and greater than CRQL. Actions apply only to samples associated with the contaminated blank.	● No common contaminants should be present. ● One method blank per matrix per concentration level per 12 hour tune per system. ● Field blanks are treated as method blanks. ● No TCL compounds above 5x CRQL. ● Non-target compound must not have peak area > 10% of the nearest internal standard. ● Action: If contaminant also detected in sample, qualify data (U) at the CRQL if concentration is < 5x blank level (10x for common lab contaminants (CLCs) and less than CRQL. Qualify (U) at raised detection limit if concentration is < 5x blank level (10x for CLCs) and greater than CRQL.
SURROGATES	Yes	Analyte — QC Limits water — QC Limits soil Toluene-d_8 — 84-138 — 88-110 Bromofluorobenzene — 59-113 — 86-115 1,2-Dichloroethane,d_4 — 70-121 — 76-114 ● All blanks and samples must meet surrogate recovery criteria. ● Action: If any surrogates are > 10%, but < CRR, (J) positive results and (UJ) quantitation limits in the sample. If < 10%, (J) positive results and (R) non-detect results in the sample. If recoveries are > CRR (J) positive results only.	Analyte — QC Limits soil — QC Limits water Toluene-d_8 — 84-138 — 88-110 Bromofluorobenzene — 59-113 — 86-115 1,2-Dichloroethane,d_4 — 70-121 — 76-114 ● All blanks and samples must meet surrogate recovery criteria. ● Action: If any surrogates are > 10%, but < CRR, (J) positive results and (UJ) quantitation limits in the sample. If < 10%, (J) positive results and (R) non-detect results in the sample. If recoveries are > CRR (J) positive results only.	Analyte — QC Limits water — QC Limits soil Toluene-d_8 — 84-138 — 88-110 Bromofluorobenzene — 59-113 — 86-115 1,2-Dichloroethane,d_4 — 70-121 — 76-114 ● All blanks and samples must meet surrogate recovery criteria. ● Action: If surrogate recovery is < 10% (R) non-detects and (J) positive results. If recoveries are 10%-CRR (UJ) non-detects and (J) positive results. If recoveries are > CRR (A) non-detects and (J) positive results.
MS/MSD	Yes	% Recovery — Soil — RPD — Water — RPD 1,1-Dichloroethane — 59-172 — 22 — 61-145 — 14 Trichloroethene — 62-137 — 24 — 71-120 — 14 Benzene — 66-142 — 21 — 76-127 — 11 Toluene — 59-139 — 21 — 76-125 — 13 Chlorobenzene — 60-133 — 21 — 75-130 — 13 ● One MS/MSD analyzed per SDG, per matrix. ● Action: If recovery is < 10% (R) non-detects and (J) positive results in unspiked sample. If recoveries are 10%-CRR (UJ) non-detects and (J) positive results in the unspiked sample. If recoveries are > CRR (A) non-detects and (J) positive results in the unspiked sample. If RPD > CRR (J) positive results. Use professional judgement to qualify compounds for high RSD between sample, MS, and MSD for non-spike compounds.	% Recovery — Soil — RPD — Water — RPD 1,1-Dichloroethane — 59-172 — 22 — 61-145 — 14 Trichloroethene — 62-137 — 24 — 71-120 — 14 Benzene — 66-142 — 21 — 76-127 — 11 Toluene — 59-139 — 21 — 76-125 — 13 Chlorobenzene — 60-133 — 21 — 75-130 — 13 ● One MS/MSD analyzed per SDG, per matrix. ● Action: CADRE performs this evaluation but does not qualify sample results which did not meet criteria. CADRE does list the compounds which failed criteria.	% Recovery — Soil — RPD — Water — RPD 1,1-Dichloroethane — 59-172 — 22 — 61-145 — 14 Trichloroethene — 62-137 — 24 — 71-120 — 14 Benzene — 66-142 — 21 — 76-127 — 11 Toluene — 59-139 — 21 — 76-125 — 13 Chlorobenzene — 60-133 — 21 — 75-130 — 13 ● One MS/MSD analyzed per SDG, per matrix. ● Action: If recovery is < 10% (R) non-detects and (J) positive results. If recoveries are 10%-CRR (UJ) non-detects and (J) positive results. If recoveries are > CRR (A) non-detects and (J) positive results. If RPD > CRR (J) positive results. Use professional judgement for RSD in unspiked samples and only for the MS/MSD sample.

CADRE METHOD COMPARISON Continued
VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines Modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
FIELD DUPLICATES	Yes	● RPD for water is < 30%; RPD for soil is < 50%. ● Action: For duplicate samples, if RPD for water is > 30% (J) positive results. Also, if RPD for soil is > 50% (J) positive results. Use professional judgement to qualify all samples of the same matrix.	● Presently, CADRE does not evaluate field duplicates.	● Criteria determined by each Region. ● Action: Action must be in accordance with Regional specifications.
INTERNAL STANDARDS	Yes	● Area counts may not vary by more than a factor of 2 from the associated calibration standard. The RTs may not shift more than 30 seconds. ● Action: If area counts are out, (J) positive results and (UJ) non-detect results for compounds quantitated using that internal standard. If area counts are extremely low, non-detects should be rejected (R). Use professional judgement if RT shifts by more than 30 seconds.	● **Primary:** Area counts may not vary by more than a factor of 2 from the associated calibration standard. The RTs may not shift more than 30 seconds. **Expanded:** areas counts may not decrease by more than a factor of 5. ● Action: If area counts are out of primary criteria low, (J) positive results and (UJ) non-detect results for compounds quantitated using that internal standard. If areas counts are out high, (J) positive results associated with that internal standard. If area counts are outside expanded criteria (J) positive results and (R) non-detects associated with that internal standard. Manual validation is required to qualify data for retention time shifts of more than 30 seconds.	● Area counts may not vary by more than a factor of 2 from the associated calibration standard. The RTs may not shift more than 30 seconds. ● Action: If area counts are out, (J) positive results and (UJ) non-detect results for compounds quantitated using that internal standard. If area counts are extremely low, non-detects should be rejected (R). Use professional judgement if RT shifts by more than 30 seconds.
TCL COMPOUND IDENTIFICATION	No	● Compound must be within ± 0.06 (RRT) units of the standard (RRT). ● Mass spectra must meet criteria: 1. All ions present in the standard at a relative intensity > 10% must be present in the sample spectrum. 2. Relative intensities of ions specified above must agree ± 20%. 3. Ions > 10% in the sample spectrum but not present in the standard must be accounted for. 4. Technical judgement may be used if all criteria are not met. ● Action: Use professional judgement.	● CADRE does not evaluate this criterion.	● Compound must be within ± 0.06 (RRT) units of the standard (RRT). ● Mass spectra must meet criteria: 1. All ions present in the standard at a relative intensity > 10% must be present in the sample spectrum. 2. Relative intensities of ions specified above must agree ±20%. 3. Ions > 10% in the sample spectrum but not present in the standard must be accounted for. 4. Technical judgement may be used if all criteria are not met. ● Action: Use professional judgement.
COMPOUND QUANTITATION AND CRQLs	CRQLs Yes Quantitation No	● See attached CRQL list for current compound list and CRQLs for water and soil. ● RRF must be calculated based on internal standard specified in SOW OLM01. ● Quantitation must be based on the specific quantitation ion listed in the SOW. ● Quantitation is performed using the RRF obtained from the daily calibration standard. ● Action: Professional judgement is used to determine if reported compounds are false positives or if false negatives are reported.	● CADRE prepares a report listing all compounds which were reported below the CRQL and flags all those results as (J). ● CADRE does not evaluate raw data, therefore, cannot verify spectra.	● See CRQL list. ● RRF must be calculated based on internal standard specified in SOW OLM01. ● Quantitation must be based on the specific quantitation ion listed in the SOW. ● Quantitation is performed using the RRF obtained from the daily calibration standard. ● Action: Professional judgement is used to determine if reported compounds are false positives or if false negatives are reported.

CADRE METHOD COMPARISON Continued
VOLATILES

	Evaluated by Tier II?	Region I Functional Guidelines Modified 11/88	CADRE Version 2.10	National Functional Guidelines Draft, 6/91
TENTATIVELY IDENTIFIED COMPOUNDS	Yes	● The laboratory must conduct a mass spectral search of the NBS library and report the possible identity for the 10 largest VOA fraction peaks. Reported peaks are not surrogate, internal standards or TCL compounds, but have area height > 10% of the size of the nearest IS. ● Action: All TICs are flagged (J). TICs are summarized in tabular format	● CADRE does not evaluate nor list a summary of the reported TICs even though they are included on a reporting form.	● The laboratory must conduct a mass spectral search of the NBS library and report the possible identity for the 10 largest VOA fraction peaks. Reported peaks are not surrogate internal standards or TCL compounds, but have area height > 10% of the size of the nearest IS. ● Action: All TICs are flagged (J).
SYSTEM PERFORMANCE	No	● Use professional judgement to evaluate effects of poor chromatography, abrupt shifts in baseline peak tailing or splitting, etc.	● CADRE does not evaluate this criterion. CADRE reports the findings of each parameter independent of other QC parameters.	● Use professional judgement to evaluate effects of poor chromatography, abrupt shifts in baseline peak failing or splitting, etc.
OVERALL ASSESSMENT OF DATA	Yes	● Use professional judgement when assessing data.	● CADRE does not evaluate this criterion. CADRE reports the findings of each parameter independent of other QC parameters.	● Use professional judgement when assessing data.

CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)
VOLATILES

	Region I		CADRE		National Functional Guidelines	
Analyte	Water * µg/L	Soil * µg/Kg	Water µg/L	Soil µg/Kg	Water µg/L	Soil µg/Kg
1,1,1-Trichloroethane	10	10	10	10	10	10
1,1,2,2-Tetrachloroethane	10	10	10	10	10	10
1,1,2-Trichloroethane	10	10	10	10	10	10
1,1-Dichloroethane	10	10	10	10	10	10
1,1-Dichloroethene	10	10	10	10	10	10
1,2-Dichloroethane	10	10	10	10	10	10
1,2-Dichloroethene(Total)	10	10	10	10	10	10
1,2-Dichloropropane	10	10	10	10	10	10
2-Butanone	10	10	10	10	10	10
2-Hexanone	10	10	10	10	10	10
4-Methyl-2-Pentanone	10	10	10	10	10	10
Acetone	10	10	10	10	10	10
Benzene	10	10	10	10	10	10
Bromodichloromethane	10	10	10	10	10	10
Bromoform	10	10	10	10	10	10
Bromomethane	10	10	10	10	10	10
Carbon Disulfide	10	10	10	10	10	10
Carbon Tetrachloride	10	10	10	10	10	10
Chlorobenzene	10	10	10	10	10	10
Chloroethane	10	10	10	10	10	10
Chloroform	10	10	10	10	10	10
Chloromethane	10	10	10	10	10	10
cis-1,3-Dichloropropene	10	10	10	10	10	10
Dibromochloromethane	10	10	10	10	10	10
Ethyl Benzene	10	10	10	10	10	10
Methylene Chloride	10	10	10	10	10	10
Styrene	10	10	10	10	10	10
Tetrachloroethene	10	10	10	10	10	10
Toluene	10	10	10	10	10	10
trans-1,3-Dichloropropene	10	10	10	10	10	10
Trichloroethene	10	10	10	10	10	10
Vinyl Chloride	10	10	10	10	10	10
Xylenes (Total)	10	10	10	10	10	10

* - Indicates that currently Region I uses CRQLs from SOW 3/90.

ATTACHMENT V

Organics Complete SDG File (CSF) Inventory Sheet

For hardcopy of the Organics Complete SDG File (CSF) Inventory Sheet Contact:
Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

CADRE Data Review Inventory Sheet

Region I Complete SDG File Receipt/Transfer Form

CADRE DATA REVIEW
INVENTORY SHEET

The following items are required to be delivered from ESD to the Field Sampling Contractor in the CADRE data review report. Please verify delivery and receipt of each item by checking the appropriate column. This form is to be included with the Data Validation Report.

Case No:_____ SDG No:_____

		ESD	Field Sampling Contractor
1)	Sample Listing	_____	_____
2)	Quantitation Limit Report	_____	_____
3)	Holding Time Report	_____	_____
4)	Percent Moisture	_____	_____
5)	Instrument Performance Check Report	_____	_____
6)	Analytical Sequence	_____	_____
7)	Calibration Listing	_____	_____
8)	Calibration Report	_____	_____
9)	Laboratory Blanks Report	_____	_____
10)	SMC/Surrogate Report	_____	_____
11)	Matrix Spike Report	_____	_____
11)	MS/MSD Non-Spike Compounds Tables	_____	_____
12)	Internal Standards Report	_____	_____
13)	Volatile Data Summary Table	_____	_____
14)	Semivolatile Data Summary Table	_____	_____
15)	Pesticide/PCB Data Summary Table	_____	_____
16)	Diskette with Data Summary Table Files	_____	_____

CADRE Report
Prepared by:_____ Affiliation:_____ Date:_____

CADRE Report
Approved by:_____ Affiliation:_____ Date:_____

Received and
Audited by:_____ Affiliation:_____ Date:_____

REGION I

COMPLETE SDG FILE

RECEIPT/TRANSFER FORM

Case:_____ SDG#:_____ Data Package#:_____

Receipt Date	Received By: Name	Init.	Affiliation	CSF Activity	Custody Seals Present/Intact (On Receipt)	Released: To	Date
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		
					Y N Y N		

ATTACHMENT VI

CARD/CADRE SDG Tracking Form

CARD/CADRE SDG TRACKING FORM

CASE:_____ SDG:_____ NO. SAMPLES:_____ MATRIX:___ LABORATORY:_____
FIELD SAMPLING CONTRACTOR:_____
SITE:_____ PARAMETERS:_____
==========
DATE PKG. RECD AT ESD RSCC:_____ DATE PKG.RECD BY ESAT:_____
DATE DOWNLOAD ATTEMPTED:_____ WAS EXTRACT AVAILABLE?:_____
IF NOT AVAILABLE, WHY?_____
DATE DOWNLOADED:_____ CARD DOWNLOAD FILE NAME:_____
DOWNLOAD FORMAT:(ASF OR CARD)_____
DATE IMPORTED INTO CADRE:____ CADRE IMPORT FILE NAME:_____
==========
DATE IMPORTED INTO CADRE:_____ NO. FORMS (RECORDS) IMPORTED:_____
IMPORT TIME:____ NO. ERRORS DETECTED:__ ERROR REPORT PRINTED:_
EXPLANATION OF MANUAL ENTRY REQUIRED:_____
_____
TIME REQUIRED FOR MANUAL ENTRY:_____
==========
__ SUMMARY TABLES GENERATED FILENAME(S): BNA (S)_____ (W)_____
VOA (S)_____ (W)_____
PEST (S)_____ (W)_____
__ FORMATTED __ CONVERTED TO WP __ FLOPPIED
__ BACKUP MADE BACKUP FILENAME(S): BNA (S)_____ (W)_____
VOA (S)_____ (W)_____
PEST (S)_____ (W)_____
==========
___ ALL WORKSHEETS GENERATED

__ SAMPLE LISTING __ ANALYTICAL SEQUENCE __ HOLDING TIMES
__ CALIBRATION REPORT __ CALIBRATION LISTING __ IPC __ % MOISTURE
__ QUANTITATION LIMIT __ BLANKS _____ SMC/SURROGATES __ MS/MSD
__ INTERNAL STANDARD __ MS/MSD NON-SPIKE TABLES __ SUMMARY TABLES

CADRE REPORT __ DISKETTE OF DATA SUMMARY TABLES ___ DATABASE FILES
INCLUDED: __ HARDCOPY REPORT __ HARDCOPY DATA SUMMARY TABLES

DATE CADRE REPORT AND DATA PACKAGE PREPARED FOR SHIPPING:_____
CADRE REPORT AND DATA PACKAGE SENT TO FIELD SAMPLING CONTRACTOR BY:_____
DATE CADRE REPORT AND DATA PACKAGE SENT TO FIELD SAMPLING CONTRACTOR:_____

ATTACHMENT VII

Memorandum for Qualifying Soil/Sediment Data with Low Percent Solid

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION I
Environmental Services Division
60 Westview Street, Lexington, MA 02173-3185

MEMORANDUM

DATE: March 29, 1990

SUBJ: Qualifying Soil/Sediment Data with low Percent Solids

FROM: Moira Lataille/CLP-TPO
Deborah Szaro/CLP-TPO

TO: Data Validators

The RAS Inorganic and Organic SOWs may be used to analyze water and soil/sediment samples. However, what constitutes a soil/sediment sample is not addressed in either of the SOWs or the current CLP User's guide.

To maintain consistency in the validation of soil/sediment data, Region I will adhere to the definition of soil sample used by the Office of Water Regulations and Standards Industrial Technology Division.

Soil Samples--Soils, sediments and sludge samples containing more than 30% solids.(l)

Therefore, all soil data may be accepted when the percent solids are greater than 30%.

All positive results are to be approximated (J'd) when % solids are 10% or greater and less than or equal to 30%.

All positive results are to be rejected when % solids are less than 10%.

All non-detected results are to be rejected when % solids are less than or equal to 30%.

Summary

% Solids >30%
A = All results

% Solids ≤ 30% ≥ 10%
J = All positive results
R = All non-detects

% Solids < 10%
R = All results

Reference:

1. Method 1620: Metals by Inductively Coupled Plasma Atomic Emission Spectroscopy and Atomic Absorption Spectroscopy-Draft Sept 1989.

Attachment M

Example Tier I Validation Cover Letter

For hardcopy of Attachment M contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

Attachment N

Example Tier III Data Validation Reports

For hardcopy of Attachment N contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

Attachment O

"March 7, 1995 Memorandum to Heidi Horahan, ARCS DPO
re: CLP-SOW OLM03.1-New Contract Requirements."

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION I
60 WESTVIEW STREET, LEXINGTON, MA 02173

MEMORANDUM

DATE: March 7, 1995

SUBJ: CLP SOW OLM03.1 - New Contract Requirements

FROM: Moira M. Lataille
Deborah A. Szaro
CLP Technical Project Officers

TO: Heidi Horahan
ARCS Deputy Project Officer

The new CLP Statement of Work for Organic Analyses, OLM03.1, is currently being awarded to laboratories. There are two issues associated with the new CLP SOW that all field contractors should be made aware of.

First, there is new contract language (Exhibit A, 4.2.1.2.3) that requires the laboratory to monitor and record the temperature of the sample shipping cooler using a temperature blank. The SOW requires that "the temperature blank be clearly labeled: USEPA COOLER TEMPERATURE INDICATOR".

Therefore, all field contractors should <u>immediately</u> begin to include a temperature blank with <u>each</u> sample shipment cooler sent to a CLP laboratory. And each temperature blank must be identified and clearly labelled as indicated above. The validators should assess the impact of non-compliant shipment temperatures on data quality and document this in the Data Validation Memorandum.

Secondly, under the new contracts missed holding times are no longer considered to be a charge assessed under liquidated administrative damages. Therefore, missed holding times will no longer be assessed as a defect under CCS which previously resulted in an automatic reduced payment to the laboratory. AOB has directed the regions to assess the actual cost of this non-compliance to the region on a case-by-case basis.

Therefore, all field contractors should immediately begin to review all CLP CSFs to assess the impact of

missed holding times on data quality taking into account site-specific data quality objectives. If the data quality is adversely impacted by missed holding times, the validators should submit a request for data rejection or reduced payment in accordance with the CLP-TPO regional guidance.

Please forward this information to the field contractors at your earliest convenience. If you have any questions, we can be reached at (617) 860-4312.

Attachment P

"The Regional Sample Control Center Guidance for the Contract Laboratory Program (CLP) and Delivery of Analytical Services (DAS) Program for EPA-New England", November 1996

The Regional Sample Control Center (RSCC) Guidance for the Contract Laboratory Program (CLP) and Delivery of Analytical Services (DAS) program for EPA-New England

November 1996
RSCCGuid-01

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I. Introduction

A. Contract Laboratory Program (CLP) Background

The Superfund Contract Laboratory Program (CLP) was established in 1980 in response to the increased analytical needs created by the passage of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). It is a national program that consists of laboratories throughout the country which support the Environmental Protection Agency's (EPA) Superfund analytical needs. It provides cost effective data of known quality to be used in environmental decision making and in supporting Agency enforcement actions.

Prior to June 30, 1994 the CLP provided both Routine Analytical Services (RAS) through inorganic and organic Statements of Work (SOWs) and Special Analytical Services (SAS) through analytical subcontracts procured by the Sample Management Office Contract. Presently, the CLP provides organic and inorganic analytical services within the Routine Analytical Services program.

B. Delivery of Analytical Services (DAS) Background

As per the Superfund 90-Day Study, the Office of Emergency and Remedial Response (OERR) established a Delivery of Analytical Services (DAS) Task Force in April of 1991 to develop a Superfund long-term strategy for the delivery of analytical services. The final strategy decision for the delivery of special analytical services, which was made by the Deputy Assistant Administrator for the Office of Solid Waste and Emergency Response, was that special analytical services should be regionalized. This decision was transmitted to the Regions in a memorandum dated 01/27/93 from Henry Longest II, Director of the OERR. Thus, the ability to ship samples under the CLP Special Analytical Services (SAS) program, which had previously handled all the samples which could not be processed with the RAS program, ended June 30, 1994.

A Region I EPA DAS Workgroup formed in March 1993 to determine the best mechanism for obtaining special analytical services. The workgroup evaluated vehicles for obtaining these analytical services.

Given the high sample volumes, cost and the complexity of the analytical needs, EPA-New England decided on a two-phased implementation plan to obtain future Superfund special analytical services. EPA-New England's short-term approach was to direct the EPA field contractors to procure/obtain special analytical services for those sites that they worked on, either by subcontracting the analytical services or utilizing in-house corporate laboratory facilities. The long-term solution would be the procurement of a Regional Environmental Analyses Procurement (REAP) for special analytical services. EPA New-England will continue to use the national CLP RAS services whenever the analytical SOWs meet the project data quality objectives (DQOs).

C. Overview of the Contract Laboratory Program (CLP) Operations

The Contract Laboratory Program (CLP) is administered by a headquarters Administrative Project Officer (APO) with Technical Project Officer (TPO) support in the regions. The Technical Project Officer oversees the contract compliance of the laboratories in their region which have contracts under the CLP and is the first line of contact for the laboratory for resolution of all technical problems.

All CLP samples collected by EPA personnel, EPA contractors, States under Cooperative Agreements and other Federal Agencies under Interagency Agreements are tracked by the RSCC.

The Regional Sample Coordinator (RSC) places all Regional requests for CLP analyses. The requests are submitted to DynCorp Information & Engineering Technology, under the Contract Laboratory Analytical Services Support (CLASS) contract.

Analytical requests are processed one week prior to the anticipated sampling date. The specifics for obtaining CLP analyses are located in detail in the following sections of this document. The status of the field samples from the date of collection, submission to a laboratory, receipt of data and completion of data validation are tracked by the New England Sample Tracking System (NESTS) database maintained by the RSCC. Routinely, reports containing the tracking information are provided to the Lead Chemists. These reports are provided at various intervals from weekly to quarterly. See Section B, Activities, part 12 for a description of the reports.

Standardized sample identification paperwork, including sample labels, tags and Traffic Reports, are required for all CLP sampling. Accurate completion of standardized forms ensures that sample authenticity and sample custody are maintained.

D. Overview of Delivery of Analytical Services (DAS) Activities

Delivery of Analytical Services (DAS) activities refer to all EPA sampling activities or EPA contracted sampling activities for Superfund not analyzed through the CLP. DAS samples collected by EPA personnel and EPA contractors and submitted to either the U.S. EPA NERL, contracted laboratories or corporate laboratories are tracked by the RSCC. DAS samples collected under Interagency Agreements or Cooperative Agreements by States are not tracked by the RSCC.

The DAS analytical specification must ensure that the project data quality objectives (DQOs) for the sampling event are achieved. The organization procuring analytical services for the EPA is responsible for ensuring that usable data are delivered. To that end, the field contractor, State or other Federal Agency should review laboratory quality assurance plans, SOPs and other documentation outlining laboratory policies and procedures. In addition, technical systems audits, including on-site laboratory audits should be performed to monitor compliance with contract specifications. Performance Evaluation Samples (PESs) should also be analyzed by contracted laboratories to monitor performance prior to and/or during field sample analysis. The status of the field samples which includes the date of collection, submission to a laboratory, receipt of data and completion of data validation are tracked with the RSCC database. The field sampling contractor provides this information to the EPA RSC with the chain-of-custody form, the DAS Summary Form and DAS Data Receipt Notification form. Routinely, reports containing the tracking information are provided to the field sampling contractors. These reports are provided at various intervals from weekly to quarterly. See Section B, Activities, part 12 for a description of the reports.

II. Analytical Services Available

The Contract Laboratory Program (CLP) Routine Analytical Services (RAS) include Organic and Inorganic analyses for single-phase aqueous and soil/sediment samples.

A. Organic Routine Analytical Services (RAS)

The CLP Organic contracts are operating under the OLM03.1, or more recent version, of the USEPA Contract Laboratory Program Statement of Work for Organic Analysis. A copy of the SOW, Exhibit C, which has the analyte list and contract required quantitation limits is provided in Attachment I.

The SOW includes the analysis of soil and aqueous samples for volatile, semivolatile and pesticide/PCBs compounds. Analysis techniques include gas chromatography/mass spectrometer (GC/MS) and gas chromatography/electron capture detector (GC/ECD) procedures.

B. Inorganic Routine Analytical Services (RAS)

The CLP Inorganic contracts are working under the ILM04.0, or more recent version, of the USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis. A copy of the SOW, Exhibit C, which has the analyte list and contract required detection limits is provided in Attachment I. The SOW includes the analysis of soil and aqueous samples for metals, including cyanide and mercury. Analysis techniques include atomic absorption and inductively coupled plasma procedures.

C. Delivery of Analytical Services (DAS)

The Delivery of Analytical Services (DAS) program consists of primarily one method of laboratory procurement. The field sampling contractors procure analytical services in accordance with the "Region I ARCS Delivery of Analytical Services Pilot Program, Final Report Volume II. Appendices", 15 March 1994, or as otherwise directed by the EPA contract Project Officer.

III. Regional Sample Control Center (RSCC)

A. Overview

The Region I Sample Control Coordinator is:

Christine Clark
U.S. EPA, OEME
60 Westview St.
Lexington, MA 02173
(617)860-4615
Fax No. (617)860-4397

Each Region has established a RSCC to centralize scheduling of CLP sample analyses. The RSC routinely places all Regional requests for CLP analyses, coordinates with the Contract Laboratory Analytical Services Support (CLASS) contractor during sampling and sample shipment, and assists with resolving any problems/issues concerning the samples. The RSC is the point of contact for questions from sampling contractors and CLASS concerning Regional sampling efforts.

B. Activities

1. Quarterly CLP and DAS Sampling Projections

Prior to the beginning of each calendar quarter, CLASS requires quarterly projections from the Region. The RSC sends a letter requesting Quarterly Projections to all the potential regional samplers. The projections include Routine Analytical Services (RAS) samples and Delivery of Analytical Services (DAS) samples. These are broken down by analysis/matrix/month and summarized for the quarter.

The First Quarter of the fiscal year is October, November and December, Second Quarter is January, February and March, Third Quarter is April, May and June and Fourth Quarter is July, August and September.

The EPA RSCC sends a letter with the blank request form in accordance with the following schedule, see Attachment II:

First Quarter Request is mailed to sampler during the first week of July.

Second Quarter Request is mailed to sampler during the first week of October.

Third Quarter Request is mailed to sampler during the first week of January.

Fourth Quarter Request is mailed to sampler during the first week of April.

2. Weekly Scheduling of RAS Analysis

A sampling event is defined as "scheduled sampling at one site for a designated period of time".

To obtain RAS sample slots, the field sampling contractors must contact the RSCC as soon as it has been determined that samples will be analyzed through the CLP. Requests must be submitted no later than close of business, the Tuesday prior to the sampling date.

Requests submitted to the RSCC after the deadline of Tuesday, close of business, prior to the week of sampling are transmitted to CLASS as late requests. Whenever possible, CLASS will accommodate late analysis requests; however, these assignments are not guaranteed.

The RAS analysis requests must be submitted by facsimile on the "Region I Weekly RAS Request Form" provided as Attachment III. **The project "EPA-NE-DQO Summary Form" located in Attachment J of "Part I", Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses must be provided with the request.** Receipt of the transmittal by the RSCC must be confirmed with telephone communication initiated by the requestor.

The Region I Weekly RAS Request Form must contain the following information:

1. Site Name (from the National Priority List)
2. Location (Town, State)
3. The Contract the work is requested under
4. Analysis Turnaround Time Required
5. CERCLIS #
6. Purpose Code (Located on the Traffic Report form.)
7. Site Spill ID# with Operable Unit (A six digit code which begins with 01, for Region 1.)
8. Action Code (A two digit code from the project Work Assignment.)
9. Matrix
10. Number of Samples
11. Analysis/Parameter Code
12. Initials of the Contractor making request.

Note: The terms Analysis/Parameter Code and Analytical Method are used interchangeably in this document.

RAS laboratory requests are for one week, if more than one week requests are needed, the Region I CLP TPO must be contacted. Authorization must be obtained by Wednesday at 3:00 p.m. two weeks prior to sampling. The TPO may be contacted at (617) 860-4379. The CLP TPO notifies the RSC of authorization approval. The RSC will not submit a request for two week assignments without TPO authorization. A two week request indicates sampling during both weeks, not just the second week.

Routine data package turnaround time is 35 days from the last sample/per SDG submitted for analysis. Fast turnaround time 14 day contracts may be requested for both Inorganic and Organic samples. However, 14 day turnaround must be requested at the time of RSCC notification of sampling event and written on the "Regional Weekly RAS Request Form". The RSC contacts the requesters by COB the Friday following the initiation of a request with the laboratory assignment and case number.

3. Notification of DAS Events

The Region I field sampling contractors' responsibilities for DAS events include informing the RSC of the sampling information by using the "Region I Weekly DAS Summary Form" provided as Attachment III. The DAS notification must be submitted within seven working days of DAS sample shipment. **The project "EPA-NE-DQO Summary Form" located in Attachment J of "Part I", Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, must be provided with the request.** DAS notification includes the same information as for RAS analyses.

Notification includes:

1. Site Name (from the National Priority List)
2. Location (Town and State)
3. The Contract the work is requested under
4. Data Package Turn Around Time (Date samples shipped and date data package due.)
5. CERCLIS #
6. Purpose Code (Located on the Traffic Report Form.)
7. Site Spill ID# with Operable Unit (A six digit code which begins with 01, for Region 1.)
8. Action Code (A two digit code from the project Work Assignment.)
9. Matrix
10. Number of Samples
11. Analysis/Parameter Code
12. Initials of the Contractor making request.
13. DAS Case Number
14. Laboratory Code (Standardized by the Field Sampling Contractor with the full reference provided.)

Note: The terms Analysis/Parameter Code and Analytical Method are used interchangeably in this document.

4. Making Changes to CLP Analytical Requests

The RSC must be notified of all changes to sample shipments. Changes include the number of samples shipped, a change in the date of sample shipment, a change in the analysis requested, cancelling and the reason for cancelling or postponing a sampling event, etc. An extension for sample shipment may be requested but, it is not

guaranteed that the same laboratory will be available. If the laboratory is not available the case will be closed. The sampler must send a new RAS Request Form to the RSCC again by close of business, the Tuesday before the week of sampling, and a new case number and laboratory will be assigned.

5. Sample Documentation

Within 7 working days of sample shipment, the CLP clients must provide copies of RAS sampling documentation to the RSC. See Attachment IV.

The RAS sampling documentation includes a copy of sample Traffic Reports/Chain-of-Custody forms identifying sample numbers, and the QA/QC samples.

The DAS sampling documents include "Region I Weekly DAS Summary Form" and chain of custody documentation must be submitted to the RSC within 7 working days of DAS sample shipment. The documents must provide field and laboratory sample numbers, identify the QA/QC samples, provide the analytical method, the number of samples and matrix.

The original Traffic Reports/Chain-of-Custody forms must not be sent to the RSCC. If original Traffic Reports/Chain-of-Custody forms are received by the RSCC, a copy of the paperwork is generated and filed with the case file. The original documents are returned to the CLP or DAS client to be included with the final data package.

6. Receipt of CLP RAS Data & DAS Data Receipt Notification

Upon receipt of a CLP laboratory data package the RSC date stamps the first page of the data package and initiates a "Complete SDG File Receipt/Transfer Form", Attachment V.

The CLP data package is then identified in the NESTS database. The EPA site name, NESTS data package number, the CLP client name with the contract are recorded on the first page of the data packages.

The data package is sent within 24 hours of its receipt to the CLP client who performed the sampling for validation purposes.

The RSC is notified of DAS data receipt with the "DAS Data Receipt Notification Form" from the field sampling contractor, see Attachment VI. The form must be completed and sent to the RSC via facsimile within one day of data package receipt.

Note: If data packages for Dioxin/Furan analysis are received by the field sampling contractor, forward the data within 24 hours to the RSC. The RSC will submit the data for validation. OEME performs data validation on all Region I Dioxin/Furan data.

7. CLP Contract Compliance Screening (CCS) & Laboratory Response to CCS

CLASS checks laboratory data packages for compliance with CLP contract requirements. CLASS completes the Contract Compliance Screening (CCS) Reports. A copy of the CCS Report is sent to the laboratory and another copy is sent to the Region. The Regional CCS copy is sent to the CLP client. The CCS Reports must be included with the laboratory data package in the CLP client's project files.

The RSC receives copies of the laboratory's response to CCS reports. The responses are forwarded to the CLP clients to be included with the laboratory data package in the CLP client's project files.

8. Data Validation Reports and Tier I Validation Cover Letters

Refer to Sections 10.0 through 14.0 of the Data Validation Manual, Part I of Region I, EPA-NE Data Validation Functional Guidelines for Evaluation of Environmental Analyses, for format and distribution of Data Validation Reports and Tier I Validation Cover Letters.

9. Notification to CLASS for Sample Shipment

All RAS shipment information must be reported to the Region I CLASS Coordinator, Neil Rogers. The sampler must telephone Neil Rogers at (703)519-1019 or by facsimile at (703)519-8626 by 5:00 p.m. on the day of shipment.

CLASS must be notified for shipments placed on Friday for Saturday delivery no later than 3:00 p.m. on Friday. If CLASS is not notified by 3:00 p.m. on Friday it is considered a late notification and receipt by the laboratory is not be guaranteed.

The required shipment information includes:

1. Case Number
2. Date of telephone message or facsimile
3. Shipper - Region I
4. Lab receiving samples
5. Number of samples and analysis
6. Matrix and concentration of samples shipped
7. Date of shipment
8. Expected date of delivery
9. Courier/Airbill numbers
10. Identify whether or not shipping is complete
11. Special comments

10. Distribution of CLP Sampling Documentation

The RSCC manages the distribution of CLP and regional sampling documentation to Region I CLP clients.

On an as-needed basis, CLP clients telephone the RSCC to request CLP sample Sample Tags, Custody Seals, Inorganic and Organic Traffic Reports/Chain-of-Custody forms and Inorganic, Organic Labels, see Attachment IV. The CLP clients must call one week in advance of requiring supplies.

11. Problem Resolution and Information Services

The RSC assists both CLASS and CLP clients when questions and/or problems arise regarding laboratory or field activities.

When sampling problems occur and/or samples are not shipped as scheduled, CLP clients must notify the RSC with the reason for the change by telephone as soon as a change or problem has been identified. The RSC documents this information in a telephone logbook and communicates the changes to CLASS.

If laboratories encounter problems during sample receipt or analysis, the laboratory contacts CLASS, who then contacts the RSC to discuss the problem resolution. If appropriate, the RSC will contact the affected CLP client to resolve the situation. <u>The field samplers cannot contact the laboratory until data have been received.</u>

CLP client questions concerning sample shipment, sample analysis, laboratory contacts, the status of data deliverables, and final data packages are relayed to the RSC. The RSC is the regional information center for incoming telephone calls, correspondence, and other inquiries regarding CLP operations for EPA New England.

CLP clients should contact the National Technical Information Service (NTIS) in Springfield, Virginia 22161, Telephone (703)487-4650 or 1-800-553-NTIS, Facimile: (703)321-8547 or (703)321-9038, directly for CLP Statements of Work.

12. Reports Provided by the RSCC to the Lead Chemists

The RSC provides reports to the Lead Chemist for each EPA field sampling contractor. The Lead Chemist is responsible for confirming that the information is accurate and/or updating the information for the RSCC within seven days of receipt. These reports must contain complete and accurate data records.

Routine Analytical Services (RAS) Reports:

1. RAS Outstanding Analysis Status Report - Monthly

The RAS Outstanding Analysis Status Report identifies Region I CLP samples which were shipped to CLP laboratories for analysis. This report is generated monthly.

The Lead Chemist verifies that this report identifies all the samples they have shipped and that the ship date, laboratory, Case number, number of samples, contractor/contract and due date correlate with his/her records.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC within seven days of report receipt.

If samples have been identified as outstanding, at the laboratory for longer than the turnaround time, then the Lead Chemist must telephone the RSC within three days of report receipt to identify the late data by Case Number, SDG and laboratory. The RSC will ascertain a delivery date.

2. RAS Outstanding Data Validation Memoranda Status Report - Monthly

The RAS Outstanding Data Validation Memoranda Status Report is a summary of Region I CLP cases which have been in data review for more than 21 days. This report is generated monthly.

The lead chemist verifies that this report identifies all the cases which are presently in data validation and that the Case number, contractor/contract, laboratory, number of samples and the data package received date correlate with his/her records.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC, within seven days of report receipt.

If CLP cases have been identified which have been in data review for more than 21 days, then the Lead Chemist must provide to the RSC, within seven working days of report receipt, a letter which identifies by Case Number, SDG, and laboratory the Outstanding Data Validation Memoranda with the scheduled date by which validation will be complete.

DAS Activities Status Reports:

1. DAS Outstanding Analysis Status Report - Weekly

The DAS Outstanding Analysis Report identifies DAS samples which were shipped to DAS laboratories for analysis. This report is generated weekly.

The Lead Chemist verifies that this report identifies all the samples that they have shipped and that the ship date, laboratory, Case number, number of samples, contractor/contract and due date correlate with his/her records.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC within seven days of report receipt.

If samples have been identified as outstanding, at the laboratory for longer than the turnaround time, then the Lead Chemist must pursue obtaining the data. The Lead Chemist must provide to the RSC a letter which identifies the late data by Case Number, SDG and laboratory. The Lead Chemist must provide within seven working days the date that the data are anticipated or received.

2. DAS Outstanding Data Validation Memoranda Status Report - Monthly

The DAS Outstanding Data Validation Memoranda Status Report is a summary of DAS cases which have been in data review for more than 21 days. This report is generated monthly.

The Lead Chemist verifies that this report identifies all the casaes which are presently in data validation and that the Case Number, contractor/contract, laboratory, number of samples and the data package received date correlate with his/her records.

If there are discrepacies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC, within seven working days of report receipt.

If Cases have been identified which have been in data review for more than 21 days the Lead Chemist must provide to the RSC, within seven working days of report receipt, a letter which identifies by Case Number, SDG, and laboratory the Outstanding Data Validation Memoranda with the scheduled date by which validation will be complete.

3. Summary of All Data Packages Report - Quarterly

The Summary of All Data Packages Report identifies the overall status of each DAS Case Number and SDG number. This report is generated quarterly.

The Lead Chemist verifies that this report identifies all of DAS activities for the quarter, showing late data/late validation trends, as well as data packages and/or validation reports which have not been received by the RSC.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC, within seven days of report receipt.

If samples have been identified as outstanding, at the laboratory for longer than the turnaround time, then the Lead Chemist must pursue obtaining the data. The Lead Chemist must provide to the RSC a letter which identifies the late data by Case Number, SDG and laboratory. The Lead Chemist must provide within seven working days the date that the data are anticipated or received.

If Cases have been identified which have been in data review for more than 21 days the Lead Chemist must provide to the RSC, within seven working days of report receipt, a letter which identifies by Case Number, SDG, and laboratory the Outstanding Data Validation Memoranda with the scheduled date by which validation will be complete.

4. QA/QC Tracking Report - Bimonthly

The QA/QC Tracking Report lists all the field QC samples assigned to each DAS sampling event. This report is generated bimonthly.

The Lead Chemist verifies that field QC samples are taken at the proper frequency and that the field QC sample numbers are accurate.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC, within seven days of report receipt.

If it is identified that field QC samples were not provided at the proper frequency, the Lead Chemist must submit a letter identifying the data by Case Number, SDG and Laboratory. The letter must define the discrepancy and provide an explanation as to why the frequency requirements were not met.

5. Lab Performance With WMD Comments Report - Bimonthly

The Lab Performance with WMD Comments Report is a summary of comments extracted from the DV memo cover letter and/or the "Data Completeness Worksheet" regarding either the DAS analysis method or problems/deficiencies with the data package. This report is generated bimonthly.

The Lead Chemist verifies that this report identifies cases which require the initiation of corrective action measures with laboratories.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC, within seven days of report receipt.

If there are problems which require follow-up, either in progress or new initiatives, by the Lead Chemist regarding issues in the report, then the Lead Chemist must submit a letter which identifies the data by Case Number, SDG and laboratory to the RSC within seven working days of report receipt describing the actions and the status of these activities.

6. DAS Activity Report - Monthly

The DAS Activity Report lists the matrices and analysis/parameter code requested for each site during a specified time frame. It is generated monthly.

The Lead Chemist verifies that the level and type of DAS activities for each site are reported accurately.

If there are discrepancies between the Lead Chemist's records and the report, then the Lead Chemist must provide updated information to the RSC, within seven days of report receipt.

If Cases have been identified which the data are erroneous or data have not been provided to the RSC then the Lead Chemist must provide all information identified with Case Number, SDG and laboratory to the RSC to make the records complete and/or accurate.

IV. Definitions/Acronyms

CADRE	Computer Aided Data Review and Evaluation
CCS	Contract Compliance Screening
CERCLIS	Comprehensive Environmental Recovery Compensation and Liability Information System
CLASS	Contract Laboratory Analytical Services Support
CLP	Contract Laboratory Program
CLP Client	User of CLP Services
CSF	Complete SDG File
DAS	Delivery of Analytical Services
DAS Client	User of DAS services
DPN	Data Package Number
EPA	U.S. Environmental Protection Agency
ESAT	EPA Region I Environmental Services Assistance Team: Lockheed Engineering and Sciences Corporation
ESD	EPA Region I Environmental Services Division
NESTS	New England Sample Tracking System
OEME	Office of Environmental Measures and Evaluation, formerly Environmental Services Division
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan (or QAPjP)
RAS	Routine Analytical Services

RPO	Regional Project Officer
RSC	Regional Sample Coordinator
RSCC	Regional Sample Control Center
SCC	ESAT Sample Control Coordinator
SDG	CLP Case Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TPO	Technical Project Officer

Attachment I

CLP SOW Exhibit C

EXHIBIT C

TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

NOTE: Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

All CRQLs are rounded to two significant figures.

The CRQL values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D.

For soil samples, the moisture content of the samples must be used to adjust the CRQL values appropriately.

Exhibit C - Target Compound List and Contract Required Quantitation Limits

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1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS 3

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS 4

3.0 PESTICIDES/AROCLORS TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS 7

1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

			Quantitation Limits			
			Water	Low Soil	Med. Soil	On Column
	Volatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)
1.	Chloromethane	74-87-3	10	10	1200	(50)
2.	Bromomethane	74-83-9	10	10	1200	(50)
3.	Vinyl Chloride	75-01-4	10	10	1200	(50)
4.	Chloroethane	75-00-3	10	10	1200	(50)
5.	Methylene Chloride	75-09-2	10	10	1200	(50)
6.	Acetone	67-64-1	10	10	1200	(50)
7.	Carbon Disulfide	75-15-0	10	10	1200	(50)
8.	1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9.	1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10.	1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11.	Chloroform	67-66-3	10	10	1200	(50)
12.	1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13.	2-Butanone	78-93-3	10	10	1200	(50)
14.	1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15.	Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16.	Bromodichloromethane	75-27-4	10	10	1200	(50)
17.	1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18.	cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19.	Trichloroethene	79-01-6	10	10	1200	(50)
20.	Dibromochloromethane	124-48-1	10	10	1200	(50)
21.	1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22.	Benzene	71-43-2	10	10	1200	(50)
23.	trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24.	Bromoform	75-25-2	10	10	1200	(50)
25.	4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26.	2-Hexanone	591-78-6	10	10	1200	(50)
27.	Tetrachloroethene	127-18-4	10	10	1200	(50)
28.	1,1,2,2- Tetrachloroethane	79-34-5	10	10	1200	(50)
29.	Toluene	108-88-3	10	10	1200	(50)

			Quantitation Limits			
			Water	Low Soil	Med. Soil	On Column
	Volatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)
30.	Chlorobenzene	108-90-7	10	10	1200	(50)
31.	Ethylbenzene	100-41-4	10	10	1200	(50)
32.	Styrene	100-42-5	10	10	1200	(50)
33.	Xylenes (total)	1330-20-7	10	10	1200	(50)

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Semivolatiles	CAS Number	Quantitation Limits Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
34.	Phenol	108-95-2	10	330	10000	(20)
35.	bis-(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)
36.	2-Chlorophenol	95-57-8	10	330	10000	(20)
37.	1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)
38.	1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)
39.	1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)
40.	2-Methylphenol	95-48-7	10	330	10000	(20)
41.	2,2'-oxybis (1-Chloropropane)[1]	108-60-1	10	330	10000	(20)
42.	4-Methylphenol	106-44-5	10	330	10000	(20)
43.	N-Nitroso-di-n-propylamine	621-64-7	10	330	10000	(20)
44.	Hexachloroethane	67-72-1	10	330	10000	(20)
45.	Nitrobenzene	98-95-3	10	330	10000	(20)
46.	Isophorone	78-59-1	10	330	10000	(20)
47.	2-Nitrophenol	88-75-5	10	330	10000	(20)
48.	2,4-Dimethylphenol	105-67-9	10	330	10000	(20)
49.	bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)
50.	2,4-Dichlorophenol	120-83-2	10	330	10000	(20)
51.	1,2,4-Trichloro-benzene	120-82-1	10	330	10000	(20)
52.	Naphthalene	91-20-3	10	330	10000	(20)
53.	4-Chloroaniline	106-47-8	10	330	10000	(20)
54.	Hexachlorobutadiene	87-68-3	10	330	10000	(20)
55.	4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)
56.	2-Methylnaphthalene	91-57-6	10	330	10000	(20)

[1]Previously known by the name bis(2-Chloroisopropyl) ether.

	Semivolatiles	CAS Number	Quantitation Limits Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
57.	Hexachlorocyclo-pentadiene	77-47-4	10	330	10000	(20)
58.	2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)
59.	2,4,5-Trichlorophenol	95-95-4	25	830	25000	(50)
60.	2-Chloronaphthalene	91-58-7	10	330	10000	(20)
61.	2-Nitroaniline	88-74-4	25	830	25000	(50)
62.	Dimethylphthalate	131-11-3	10	330	10000	(20)
63.	Acenaphthylene	208-96-8	10	330	10000	(20)
64.	2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
65.	3-Nitroaniline	99-09-2	25	830	25000	(50)
66.	Acenaphthene	83-32-9	10	330	10000	(20)
67.	2,4-Dinitrophenol	51-28-5	25	830	25000	(50)
68.	4-Nitrophenol	100-02-7	25	830	25000	(50)
69.	Dibenzofuran	132-64-9	10	330	10000	(20)
70.	2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71.	Diethylphthalate	84-66-2	10	330	10000	(20)
72.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73.	Fluorene	86-73-7	10	330	10000	(20)
74.	4-Nitroaniline	100-01-6	25	830	25000	(50)
75.	4,6-Dinitro-2-methylphenol	534-52-1	25	830	25000	(50)
76.	N-Nitroso-diphenylamine	86-30-6	10	330	10000	(20)
77.	4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
78.	Hexachlorobenzene	118-74-1	10	330	10000	(20)
79.	Pentachlorophenol	87-86-5	25	830	25000	(50)
80.	Phenanthrene	85-01-8	10	330	10000	(20)
81.	Anthracene	120-12-7	10	330	10000	(20)
82.	Carbazole	86-74-8	10	330	10000	(20)

	Semivolatiles	CAS Number	Quantitation Limits Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
83.	Di-n-butylphthalate	84-74-2	10	330	10000	(20)
84.	Fluoranthene	206-44-0	10	330	10000	(20)
85.	Pyrene	129-00-0	10	330	10000	(20)
86.	Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87.	3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88.	Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89.	Chrysene	218-01-9	10	330	10000	(20)
90.	bis(2-Ethylhexyl) phthalate	117-81-7	10	330	10000	(20)
91.	Di-n-octylphthalate	117-84-0	10	330	10000	(20)
92.	Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93.	Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
94.	Benzo(a)pyrene	50-32-8	10	330	10000	(20)
95.	Indeno(1,2,3-cd)-pyrene	193-39-5	10	330	10000	(20)
96.	Dibenzo(a,h)-anthracene	53-70-3	10	330	10000	(20)
97.	Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)

3.0 PESTICIDES/AROCLORS TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[2,3]

	Pesticides/Aroclors	CAS Number	Quantitation Limits Water ug/L	Soil ug/Kg	On Column (pg)
98.	alpha-BHC	319-84-6	0.050	1.7	5
99.	beta-BHC	319-85-7	0.050	1.7	5
100.	delta-BHC	319-86-8	0.050	1.7	5
101.	gamma-BHC (Lindane)	58-89-9	0.050	1.7	5
102.	Heptachlor	76-44-8	0.050	1.7	5
103.	Aldrin	309-00-2	0.050	1.7	5
104.	Heptachlor epoxide[4]	111024-57-3	0.050	1.7	5
105.	Endosulfan I	959-98-8	0.050	1.7	5
106.	Dieldrin	60-57-1	0.10	3.3	10
107.	4,4'-DDE	72-55-9	0.10	3.3	10
108.	Endrin	72-20-8	0.10	3.3	10
109.	Endosulfan II	33213-65-9	0.10	3.3	10
110.	4,4'-DDD	72-54-8	0.10	3.3	10
111.	Endosulfan sulfate	1031-07-8	0.10	3.3	10
112.	4,4'-DDT	50-29-3	0.10	3.3	10
113.	Methoxychlor	72-43-5	0.50	17	50
114.	Endrin ketone	53494-70-5	0.10	3.3	10
115.	Endrin aldehyde	7421-93-4	0.10	3.3	10
116.	alpha-Chlordane	5103-71-9	0.050	1.7	5
117.	gamma-Chlordane	5103-74-2	0.050	1.7	5
118.	Toxaphene	8001-35-2	5.0	170	500

[2]There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides/Aroclors.

[3]The lower reporting limit for pesticide instrument blanks shall be one-half the CRQL values for water samples.

[4]Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is reported on the data reporting forms (Exhibit B).

	Pesticides/Aroclors	CAS Number	Quantitation Limits Water ug/L	Soil ug/Kg	On Column (pg)
119.	Aroclor-1016	12674-11-2	1.0	33	100
120.	Aroclor-1221	11104-28-2	2.0	67	200
121.	Aroclor-1232	11141-16-5	1.0	33	100
122.	Aroclor-1242	53469-21-9	1.0	33	100
123.	Aroclor-1248	12672-29-6	1.0	33	100
124.	Aroclor-1254	11097-69-1	1.0	33	100
125.	Aroclor-1260	11096-82-5	1.0	33	100

EXHIBIT C

INORGANIC TARGET ANALYTE LIST

INORGANIC TARGET ANALYTE LIST (TAL) - TABLE 1

Analyte	Contract Required Detection Limit[1,2] (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

(1) Subject to the restrictions specified in Exhibits D and E, any analytical method specified in ILM04.0, Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Detection Limit. This is illustrated in the example below:

For lead: Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 220
Contract Required Detection Limit (CRDL) = 3

The value of 220 may be reported even though the instrument detection limit is greater than CRDL. The instrument or method detection limit must be documented as described in Exhibits B and E.

(2) The CRDLs are the minimum levels of detection acceptable under the contract Statement of Work.

Attachment II

Quarterly Projections

THIRD QUARTER RAS FY95 PROJECTIONS

INORGANIC ANALYSES	April	May	June
35 Day TA Full Metals With Cyanide			
35 Day TA Cyanide Only			
35 Day TA Metals Only			
14 Day TA Full Metals With Cyanide			
14 Day TA Cyanide Only			
14 Day TA Metals Only			
ORGANIC ANALYSES			
35 Day TA Full TCL			
35 Day TA VOA Only			
35 Day TA BNA Only			
35 Day TA Pesticide/PCB Only			
14 Day TA Full TCL			
14 Day TA VOA Only			
14 Day TA BNA Only			
14 Day TA Pesticide/PCB Only			
DIOXIN ANALYSES			
Fast TA 2,3,7,8-TCDD			

THIRD QUARTER DAS FY95 PROJECTIONS

Contractor/Contract:

Site Name	Parameter/Method	Matrix	April	May	June

Attachment III

RAS Weekly Request Form and DAS Summary Form

REGION 1 WEEKLY RAS REQUEST FORM

REQUESTS FOR THE WEEK OF:

CASE #/ LAB ASSIGNED	SITE NAME/ TOWN & STATE/ TURN AROUND TIME	CERCLIS #/ PURPOSE CODE/ SITE ID-ACTION CODE -OPERABLE UNIT	MATRIX	# OF SAMPLES	PARAMETER	CONTRACTOR/ CONTRACT	CLASS
O: I:			AQ AQ AQ AQ AQ AQ SOIL SOIL SOIL SOIL SOIL SOIL		VOA SEMI-VOA PEST/PCB METALS CYANIDE METALS w/CYANIDE VOA SEMI-VOA PEST/PCB METALS CYANIDE METALS w/CYANIDE		
O: I:			AQ AQ AQ AQ AQ AQ SOIL SOIL SOIL SOIL SOIL SOIL		VOA SEMI-VOA PEST/PCB METALS CYANIDE METALS w/CYANIDE VOA SEMI-VOA PEST/PCB METALS CYANIDE METALS w/CYANIDE		
O: I:			AQ AQ AQ AQ AQ AQ SOIL SOIL SOIL SOIL SOIL SOIL		VOA SEMI-VOA PEST/PCB METALS CYANIDE METALS w/CYANIDE VOA SEMI-VOA PEST/PCB METALS CYANIDE METALS w/CYANIDE		

REGION I WEEKLY DAS SUMMARY FORM

WEEK OF:_____

DAS #/SDG #/LABCODE/TAT	Site ID#-Action Code- Operable Unit/Site Name/Location	No. of Samples	Matrix	Parameter	QC Sample Nos.	Contractor/Contract
DAS#: SDG#: LAB: TAT:	SITE ID: ACTION CODE: OPERABLE UNIT: NAME: CITY: STATE:					
DAS#: SDG#: LAB: TAT:	SITE ID: ACTION CODE: OPERABLE UNIT: NAME: CITY: STATE:					
DAS#: SDG#: LAB: TAT:	SITE ID: ACTION CODE: OPERABLE UNIT: NAME: CITY: STATE:					
DAS#: SDG#: LAB: TAT:	SITE ID: ACTION CODE: OPERABLE UNIT: NAME: CITY: STATE:					
COMMENTS:						

Attachment IV

Sampling Paperwork

For hardcopy of Attachment IV example documents contact:

Steve Stodola, U.S. EPA Region I
TEL: 617-918-8634
EMAIL: stodola.steve@epamail.epa.gov

DAS DATA RECEIPT NOTIFICATION

Site Name:_____

Contractor/Contract:_____

DAS#:_____

Lab Code:_____

SDG No.:_____

No. of Samples:_____

DAS Sample Nos.:_____

Blanks:_____

Duplicates:_____

Ship to Lab Date:_____

Data Package Receipt Date:_____

CSF Receipt Date:_____

PE Sample Numbers	Matrix	Parameter	Sample Count
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ATTACHMENT A

Guidance for Completion of DQO Summary Form

DISTRIBUTION:

1) Copies of completed DQO Summary Forms should be included in the QAPjP/SAP.

2) A. Copies of completed DQO Summary Forms for all CLP RAS work requested by EPA Site Managers, EPA contractors, including RACS, ROC, and START, and other Federal Agencies under Interagency Agreements, i.e., ACOE, and States under Cooperative Agreements should be sent with the quarterly sample projections to the Region I RSCC. Completed DQO Summary Forms for CLP RAS work must be received by the RSCC prior to the sampling event.

   B. Copies of completed DQO Summary Forms for non-CLP DAS work performed for EPA Site Managers and EPA contractors must be received by the Region I RSCC prior to the sampling event.

   C. DQO Summary Forms for non-CLP work performed under Interagency Agreements, Cooperative Agreements, and Grants must be completed prior to the sampling event, submitted to the "Authorizing Organization", as delegated by EPA, and included in the site documents.

3) Copies of completed DQO Summary Forms also must be included in the Data Validation Report or Tier I Validation Cover Letter (refer to Part I of the "Data Validation Manual" in the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses), December 1996, or most recent revision.

INSTRUCTIONS:

Note: A separate Form should be completed for each sampling event. For sampling events involving multiple environmental matrices, complete Sections 5-10 for each matrix and ensure that the two-letter matrix code is identified in Section 5. Enter the page number and total number of pages in the top right hand corner on the Form.

Section 1:

- Circle the appropriate EPA Program(s) involved in multi-media, multi-programmatic sampling events including, TSCA, CERCLA (i.e, Superfund), RCRA, DW (Drinking Water), NPDES, CAA (Clean Air), or fill in the blank for "Other:_____".
- List projected date(s) of sampling. The sampling dates should be inclusive of all matrices that will be sampled during this sampling event.
- Record the EPA Site Manager's name.
- List the names of the other EPA Case Team Members.
- Enter the site name. Use the NPL site name. If an NPL site name does not exist, then use the site name assigned under CERCLIS.
- Record the name of the city/town and State where the site is located in the "Site Location" field.
- Record the "Assigned Site Latitude/Longitude". Those numbers should be identical to those contained in CERCLIS database. Contact the EPA Site Manager to obtain correct Latitude/Longitude.
- Record the CERCLA site/spill identifier number, including the operable unit number. Contact the EPA Site Manager to obtain the correct identifier numbers.
- Circle the appropriate phase of Superfund site work (ERA: Environmental Risk Assessment, SA/SI: Site Assessment/Site Investigation, RI: Remedial Investigation, FS: Feasibility Study, RD: Remedial Design, RA: Remedial Assessment, post-RA: post-Remedial Assessment, i.e., quarterly monitoring). For non-Superfund site work, identify sampling event phase in the "Other" field.

Section 2:

- Record the complete title of the final QAPjP and revision date.
- Enter name of the Approving Official.
- Record date that the QAPjP was approved.
- Enter title of the Approving Official.
- Enter name of organization that has approval authority. This will be EPA, unless approval authority has been delegated by EPA to a State or other Federal Agency.
- If another organization has been delegated approval authority, then enter the date that EPA delegated approval authority (date of Quality Assurance Management Plan approval).
- Identify whether the project sampling event is an EPA oversight project, circle Yes or No.
- Indicate type of oversight by circling either Potentially Responsible Party (PRP) or Federal Facility (FF), or complete the blank for "Other:_____".
- Identify whether confirmatory sampling and analysis is being performed to verify field screening results, circle Yes or No.
- If EPA oversight or confirmatory analysis will be performed, record the percentage of split samples to be collected and analyzed.
- If EPA oversight or confirmatory analysis will be performed, identify whether comparability criteria are documented in the approved QAPjP or SAP, circle Yes or No.

Section 3:

a) List the two letter code for each matrix for samples that will be collected. Refer to Appendix B for a correct list of matrix codes. If a matrix does not have a corresponding code, then attach a description of the matrix to the DQO Summary Form.

Note: The matrix codes correspond to the matrix identifiers contained in the New England Sample Tracking System (NESTS) database. The current list of matrix codes are not intended to include all types of environmental matrices. However, they do represent groupings of similar-type matrices that potentially contain similar analytic interferences. For example, the matrix code GW (ground water) includes water from monitoring wells, supply wells, and public wells.

b) For each matrix, identify the analytical parameters for samples that will be collected by recording the appropriate parameter code. Refer to Appendix B for a current list of parameter codes. If an analytical parameter does not have a corresponding code, then the method title and/or SOP name, method and/or SOP identification number, and method and/or SOP revision date should be included and recorded in Section 9 of this Form.

Note: The parameter codes correspond to the analytical method parameters utilized in NESTS database. Appendix B includes a comprehensive list of analytical methods that have been used historically for Region I site work.

c) For each matrix and parameter, identify the preservation technique that will be used by recording the appropriate preservation code. Refer to the reverse side of this Form for a list of preservation codes.

d) Record the analytical service(s) mechanism that will be used for each matrix and parameter;

- CLP-RAS (CLP-Routine Analytical Service) This service may be utilized by EPA site managers, EPA contractors including, RACS, ROC, and START contracts. It may also be utilized under Interagency agreements, i.e., by the ACOE, and under Cooperative Agreements with the States.
- RACS-DAS (Remedial Alternative Contracting Strategy-Delivery of Analytical Services)
- ROC-DAS (Regional Oversight Contract-DAS)
- START-DAS (Superfund Technical Assessment and Remediation Contract-DAS)
- EPA-NERL (EPA-New England Regional Laboratory)

- Regional EPA-NE analytical contract
- State-Non-CLP

- Other Federal Agency Non-CLP
- If another analytical mechanism will be used, describe in detail on a separate page and attach to the Form.

e) Record the number of discrete locations that will be sampled for each parameter. The "No. of Sample Locations" count should include the site and background locations sampled.

- Record the number of each type of field QC sample that will be collected and sent to the laboratory for analysis for each matrix and parameter.

f) Record the number of Field duplicate sample pairs (which will equal "1" for each pair of field duplicates) that will be collected.
g) Enter the number of equipment/rinsate blanks.
h) Enter the number of VOA Trip blanks.
i) Enter the number of Cooler Temperature blanks that will be used.
j) Enter the number of Bottle Blanks that will be analyzed.
k) Describe any other field QC samples and the total number that were collected and that will be sent to the laboratory.
l) Enter the number of PESs that will be sent to the laboratory in accordance with EPA Region I Performance Evaluation Program Guidance, July 1996.

Note: The total of "e-l" equals the total number of samples sent to a laboratory for each matrix and parameter.

- Record the number of each type of laboratory QC sample that will be analyzed with the samples received.

m) Enter the minimum number of reagent blanks that will be analyzed.
n) Enter the number of laboratory Duplicates that will be analyzed.
o) Enter the number of matrix spikes that will be analyzed.
p) Enter the number of matrix spike duplicates that will be analyzed.
q) Describe any other laboratory QC samples and the total number that will be analyzed.

Section 4:

- Enter the approximate site dimensions with units.
- List all potentially contaminated matrices, regardless of whether or not they will be sampled during this sampling event.
- For well sampling, complete "Range of Depth to Groundwater" to ensure proper pump is utilized.
- For soil sampling, circle Surface or Subsurface or complete Other:_____.
- For sediment sampling, circle Stream, Pond, Estuary, Wetland, or complete Other:_____.
- For soil/sediment sampling, circle expected moisture content: High or Low. **Note: Analytical methods used for high moisture content samples should ensure that DQO-specified dry weight quantitation limits are achieved.**

Section 5:

When multiple matrices will be sampled during a sampling event, complete Sections 5-10 for each matrix and enter the Matrix Code.

- Identify the two-letter matrix code for which the information is provided in sections 5-10.
- Circle the potential uses for sample data such as, site investigation/assessment, PRP determination, removal actions, nature and extent of contamination, human and/or ecological risk assessment, remediation alternatives, engineering design, remedial action, post-remedial action, i.e., quarterly monitoring. A space is available for other potential uses of data.

Section 6:

- Briefly summarize the project DQOs. This section should describe the specific objectives of the sampling event, i.e., to identify health risks to children, ages 1-6, residing on the site who might be exposed to surface soils located in the area, or to characterize the extent of groundwater contamination. Identify the purpose of sampling, the decisions that will be made using the data, action level information, and any related information needed to identify that appropriate analytical and field sampling methods were chosen. Complete the table with the following information: contaminants of concern (COC), COC action levels and analytical method quantitation limits for each COC. **Note: Since this information will be used by data validators to identify potential data usability issues for the user, it is imperative that it is clear and concise.**

Section 7:

- Circle applicable sampling technique(s) used and/or complete "Other" to describe an innovative sampling technique or one that is not listed.
- Identify the SOPs that will be utilized for sample collection. Include SOP name, identification number and revision number and/or date.
- Record the discrete Background sample station location number(s) that will be sampled.
- Circle if samples will be "grab" or "composite".
- To indicate potential "Hot spots" on site, circle Yes or No.

Section 8:

- Identify the field data that will be collected including, ORP, pH, specific conductance, dissolved O_2, temperature, and turbidity. A space is available to indicate other field testing that will be performed.

Section 9:

- If an analytical method does not have a Parameter code (required information in Section 3), then the method title and/or SOP name, method and/or SOP identification number, and method and/or SOP revision date should be included. Attach a separate page if additional space is needed.
- Record the specific parameters required for analysis.

Section 10: **In accordance with Region I QA policy, all data must be validated in accordance with the most recent revision of Part I the "Data Validation Manual: The Data Quality System" of the Region I, EPA-NE Data Validation Functional Guidelines of Evaluating Environmental Analyses.**

- Circle the data validation criteria required by the QAPjP and/or SAP. In most cases, the QAPjP and/or SAP should cite the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines of Evaluating Environmental Analyses and identify the applicable Functional Guideline criteria procedures that will be used to validate the data: Part II-Volatile/Semivolatile Data Validation Functional Guidelines, Part III-Pesticide/PCB Data Validation Functional Guidelines, and Part IV-Inorganic Data Validation Functional Guidelines.
  If modified criteria or alternate data validation criteria will be utilized, the modified or alternate criteria must be documented in an approved QAPjP and/or SAP as stipulated in Part I, the "Data Validation Manual: The Data Quality System", December 1996 revision of the Region I, EPA-NE Data Validation Functional Guidelines of Evaluating Environmental Analyses, December 1996 revision.
- Circle the Region I Validation Tier that will be used.
- If a partial Tier III data validation is required, then the subset receiving a partial Tier III should be specified (e.g., benzene, VOA, etc).
- Identify the company performing the data validation. Circle either Prime or Subcontractor.

Section 11:

- Record the field sampling contractor company/organization name
- Contract number
- Name of contract
- Work assignment number
- Name and title of person completing Form
- Completion date of the DQO Summary Form

Attachment Q

"Region I ARCS Delivery of Analytical Services Pilot Program, Final Report Volume II. Appendices," March 15, 1994

For hardcopy of Attachment Q contact:

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PART II

Volatile/Semivolatile Data Validation Functional Guidelines

December 1996

VOLATILE/SEMIVOLATILE DATA VALIDATION FUNCTIONAL GUIDELINES - PART II

The requirements to be checked in validation are listed below. "CCS" indicates that the contractual requirements for these items will also be checked by Contract Compliance Screening (CCS). CCS requirements are not always the same as data validation criteria. "CADRE" indicates that CADRE checks for these items in CLP-Low/Medium Organic electronic data and provides a CADRE printout. Additional manual evaluation may be required. Refer to the Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, February 1995, or most recent revision (Attachment L of Part I, Data Validation Manual).

I.	Preservation and Technical Holding Times	(CCS)	(CADRE)	VOA/SV-I-1
II.	GC/MS Instrument Performance Check (Tuning)	(CCS)	(CADRE)	VOA/SV-II-1
III.	Initial Calibration	(CCS)	(CADRE)	VOA/SV-III-1
IV.	Continuing Calibration	(CCS)	(CADRE)	VOA/SV-IV-1
V.	Blanks (Method Blanks Only)	(CCS)	(CADRE)	VOA/SV-V-1
VI.	Surrogate Compounds	(CCS)	(CADRE)	VOA/SV-VI-1
VII.	Internal Standards	(CCS)	(CADRE)	VOA/SV-VII-1
VIII.	Matrix Spike/Matrix Spike Duplicate	(CCS)	(CADRE)	VOA/SV-VIII-1
IX.	Field Duplicates			VOA/SV-IX-1
X.	Sensitivity Check			VOA/SV-X-1
XI.	PE Samples/Accuracy Check			VOA/SV-XI-1
XII.	Target Compound Identification			VOA/SV-XII-1
XIII.	Compound Quantitation and Reported Quantitation Limits		(CADRE)	VOA/SV-XIII-1
XIV.	Tentatively Identified Compounds			VOA/SV-XIV-1
XV.	Semivolatile Cleanup			VOA/SV-XV-1
XVI.	System Performance			VOA/SV-XVI-1
XVII.	Overall Evaluation of Data			VOA/SV-XVII-1

Appendices

Appendix A	CLP SOW OLM03.2/Volatile Organic Analysis
Appendix B	CLP SOW OLM03.2/Semivolatile Organic Analysis
Appendix C	CLP SOW OLC02.0/Low Concentration Volatile Organic Analysis
Appendix D	CLP SOW OLC02.0/Low Concentration Semivolatile Organic Analysis
Appendix E	VOA/SV Functional Guidelines Action Tables

I. PRESERVATION AND TECHNICAL HOLDING TIMES

A. OBJECTIVE

The objective is to ascertain the validity of the analytical results based on the preservation techniques which were used and the holding time of the sample from time of collection to time of sample preparation and sample analysis, as appropriate.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. REGION I PRESERVATION CRITERIA

SAMPLE TYPE	PRESERVATION CODE
Volatile Aqueous [a]	1,2,3
Volatile Soil/Sediment [b]	1,3
Semivolatile Aqueous [a]	1,3
Semivolatile Soil/Sediment [b]	1,3
VOA/SV Sludge [b]	1,3
VOA/SV Oily Waste [b]	1,3
VOA/SV Biological Tissue [c]	3,4
VOA Air (Canister) [c]	3,5
VOA Air (Adsorbent Tubes) [c]	1,3
SV Air (PUF, Filters) [c]	1,3
SV Wipes [c]	1,3
SV Fly Ash [b]	1,3

Preservation Code:

1. Cool @ 4°C (± 2°)
2. Preserve with HCl to at least pH 2
3. Protect from light
4. Freeze
5. Room Temperature (Avoid excessive heat)

References:

a. 40 CFR, Part 136, Appendix A, 600 Series

b. SW-846, 8000 Series

c. Region I policy

2. REGION I TECHNICAL HOLDING TIME CRITERIA

SAMPLE TYPE	CRITERIA
Volatile Aqueous [a]	If the sample was not properly preserved with HCl but was protected from light and stored at 4°C (± 2°), aromatic volatiles must be analyzed within 7 days and non-aromatic volatiles within 14 days of sample collection. If the sample was properly preserved, then both aromatic and non-aromatic volatiles must be analyzed within 14 days of sample collection.
Volatile Soil/Sediment [b]	Properly preserved soil/sediment samples must be analyzed within 14 days of sample collection.
Semivolatile [a] Aqueous	Extraction of properly preserved aqueous samples by liquid-liquid procedures must be started within 7 days of sample collection. Extraction of properly preserved aqueous samples by separatory funnel or solid phase extraction (SPE) must be completed within 7 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Semivolatile Soil/Sediment [b]	Extraction of properly preserved soil/sediment samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
VOA/SV Sludge [b]	Purge and trap or extraction of properly preserved sludge samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction. . . .
VOA/SV Oily Waste [b]	Purge and trap or extraction of properly preserved oily waste samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction. . . .

SAMPLE TYPE	CRITERIA
VOA/SV Biological Tissue [c]	Extraction and analysis of frozen tissue must be completed within 60 days of sample collection. Tissue must remain frozen until homogenization is completed. Extraction and/or analysis must be initiated immediately after homogenization.
VOA Air [c]	Analyses of properly preserved VOA air samples must be completed within 14 days of sample collection. Pre-cleaned and certified volatile air collection devices, i.e., Tenax and charcoal cartridges and SUMMA canisters, must be utilized for sample collection within the method-specified time frame.
SV Air [c]	Analyses of properly preserved SV air samples must be completed within 14 days of sample collection. Pre-cleaned and certified semivolatile air collection devices, i.e., PUFS, and filters, must be utilized for sample collection within the method-specified time frame.
SV Wipes [c]	Extraction of properly preserved SV Wipe samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
SV Fly Ash [b]	Extraction of properly preserved SV fly ash samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from preservation and/or holding time anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Volatile Samples	1. Volatile Samples
a. Preservation	a. Preservation
Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form), laboratory tracking/storage forms, and the data package narrative to verify that samples were properly preserved by the sampler and the laboratory maintained preservation. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information.	If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume the samples were not preserved and should document on the holding time worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify or reject the sample data. If the samples were not preserved properly in the field and/or if the laboratory failed to properly maintain sample preservation, then the validator should take the following actions:
i. Verify that volatile samples were refrigerated or frozen (as required) and protected from light according to Region I preservation criteria.	i. If volatile samples for aqueous and soil/sediment matrices were not refrigerated and/or protected from light according to Region I preservation criteria, then the validator should estimate (J) positive detects and reject (R) non-detects for the affected samples, regardless of whether or not technical holding time criteria were met and regardless of whether or not the sample (aqueous) was acid preserved. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when temperature and light protection preservation criteria were not met. Professional judgment should be used when the laboratory has reported transportation cooler temperatures that slightly exceed the upper limits of the preservation criteria (> + 6°C). In this case, the laboratory procedure for monitoring cooler temperature may be in question. In this event, the validator should document all justifications for qualifying or not qualifying sample data in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
1. a. ii. Verify from the EPA Traffic Report and/or COC Form and the data package narrative that aqueous volatile samples were preserved with HCl according to Region I preservation criteria.	1. a. ii. If data package documentation does not list the pH of each aqueous VOA sample, then the validator should contact the laboratory to obtain any omitted information. If aqueous volatile samples were not preserved with HCl according to Region I preservation criteria, then the validator must evaluate holding times to determine if qualification of sample data is necessary for detected and non-detected aromatic and non-aromatic compounds.
iii. Review sample records (COC Forms, Sample Receipt and/or Login Forms, DC-1, etc.) to determine if excessive headspace in any aqueous sample was noted by the laboratory.	iii. If volatile aqueous samples contain excessive headspace (bubbles greater than 2 mm diameter should not be present), then the validator should estimate (J) positive detects and reject (R) non-detects.
b. Technical Holding Times	b. Technical Holding Times
i. Verify that volatile samples were analyzed within the technical holding time criteria. Establish technical holding times by comparing sampling dates reported on the EPA Traffic Report and/or COC Forms with dates of analysis on tabulated result forms.	i. If aqueous volatile samples were not preserved with HCl (but refrigeration and light protection criteria were met) and the samples were not analyzed within 7 days, then the validator should: - Estimate (J) aromatic positive detects analyzed within 14 days. - Reject (R) aromatic non-detects. - Accept non-aromatic positive detects analyzed within 14 days - Accept non-aromatic non-detects analyzed within 14 days. - Estimate (J) aromatic positive detects analyzed after 14 days. - Estimate (J) non-aromatic positive detects analyzed after 14 days. - Estimate (UJ) non-aromatic non-detects analyzed after 14 days. If volatile samples for aqueous and soil/sediment matrices were properly preserved, but the technical holding time criteria were exceeded yet samples were analyzed within 28 days, then the validator should estimate (J) positive detects and (UJ) non-detects. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when technical holding time criteria are exceeded. For all matrices, if technical holding times for volatile samples were grossly exceeded (> 28 days), then the validator should estimate (J) positive detects and reject (R) non-detects.

C. EVALUATION	D. ACTION
*1. b. ii. Check the raw data including instrument run and extraction logs to verify reported sample extraction and analysis dates.	1. b. ii. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Semivolatile Samples a. Preservation Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form), laboratory tracking/storage forms, and the data package narrative to verify that samples were properly preserved by the sampler and the laboratory maintained preservation. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information.	2. Semivolatile Samples a. Preservation If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume the samples were not preserved and should document on the holding time worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify or reject the sample data. If the samples were not preserved properly in the field and/or if the laboratory failed to properly maintain sample preservation, then the validator should take the following actions:
i. Verify that semivolatile samples were refrigerated or frozen (as required) and protected from light according to Region I preservation criteria.	i. If semivolatile samples for aqueous and soil/sediment matrices were not refrigerated and/or protected from light according to Region I preservation criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples, regardless of whether or not technical holding time criteria were met. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when temperature and light protection preservation criteria were not met. Professional judgment should be used when the laboratory has reported transportation cooler temperatures that slightly exceed the upper limits of the preservation criteria (> + 6°C). In this case, the laboratory procedure for monitoring cooler temperature may be in question. In this event, the validator should document all justifications for qualifying or not qualifying sample data in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
2. b. Technical Holding Times i. Verify that semivolatile samples were extracted within technical holding time criteria. Establish extraction holding times by comparing sampling dates reported on the EPA Traffic Report and/or COC Forms with dates of extraction reported on tabulated result forms. 1. Verify that liquid-liquid extractions for semivolatile aqueous samples were begun within 7 days of sample collection. 2. Verify that aqueous semivolatile extractions by separatory funnel were completed within 7 days of sample collection. (Note: OLM03.2 does not allow separatory funnel extraction of semivolatiles.) 3. Verify that aqueous semivolatile extractions by solid phase extraction (SPE) or other extraction technique were completed within 7 days of sample collection. 4. Verify that semivolatile soil/sediment sample extractions by sonication or soxhlet procedures were completed within 14 days of sample collection. 5. Verify that samples of other matrices, i.e., wipes, biological tissue, were extracted within the Region I holding time criteria. Verify that semivolatile samples and/or extracts (as required) were analyzed within technical holding time criteria for analysis. Establish analytical holding times by comparing collection and/or extraction dates (as required) and analysis dates reported on tabulated result forms.	2. b. Technical Holding Times i. If aqueous and soil/sediment semivolatile samples were properly preserved, but the technical extraction and/or analytical holding time criteria were exceeded, then the validator should estimate (J) positive detects and estimate (UJ) non-detects. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when technical holding time criteria are exceeded. For all matrices, if semivolatile extraction technical holding time criteria were grossly exceeded (> 28 days) and/or analytical technical holding time criteria were grossly exceeded (> 60 days), then the validator should estimate (J) positive detects and reject (R) non-detects.

C. EVALUATION	D. ACTION
*2. b. ii. Check the raw data including extraction and instrument run logs to verify reported sample extraction and analysis dates.	2. b. ii. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b.ii, C.2.b.ii

Table VOA/SV-I-1:

QUALIFICATION OF VOLATILE ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION			TECHNICAL HOLDING TIMES			
Matrix	Refrig. & Light Protected	Acid Preserved	≤ 7 Days	7 < HT ≤ 14 Days	14 < HT ≤ 28 Days	> 28 Days
AQ	No	Yes or No	J - detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects
AQ	Yes	Yes	A	A	J - detects UJ - non-detects	J - detects R - non-detects
AQ	Yes	No	A	**Aromatics** J - detects R - non-detects **Non-aromatics** A - detects A - non-detects	**Aromatics** J - detects R - non-detects **Non-aromatics** J - detects UJ - non-detects	J - detects R - non-detects
S/S	No	N/A	J- detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects
S/S	Yes	N/A	A	A	J - detects UJ - non-detects	J - detects R - non-detects

Note: AQ = Aqueous, S/S = Soil/Sediment

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify or reject non-detects when Region I preservation and/or technical holding time criteria are not met.

For VOA aqueous samples containing excessive headspace (bubbles greater than 2 mm diameter); J-detects, R-non-detects

Table VOA/SV-I-2:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION		TECHNICAL HOLDING TIMES		
Matrix	Refrig. & Light Protected	Extracted and/or Analyzed Within H.T.	Extracted and/or Analyzed Outside H.T.	If Extraction HT > 28 days and/or Analytical HT > 60 days
AQ and S/S	Yes	A	J - detects UJ - non-detects	J - detects R - non-detects
AQ and S/S	No	J - detects UJ - non-detects	J - detects UJ - non-detects	J - detects R - non-detects

Note: AQ = Aqueous, S/S = Soil/Sediment

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify or reject non-detects when Region I preservation and/or technical holding time criteria are not met.

REFERENCES

a - 40 CFR, Part 136, Appendix A, 600 Series

b - SW-846, 8000 Series

E. EXAMPLES

Example #1: (Improper preservation (without acid); Analysis holding time exceeded)

Aqueous volatile sample SAA99 was analyzed by routine analysis following CLP SOW OLM03.2. The validator examines the data package narrative and determines that the laboratory did not report the pH. The validator contacts the laboratory to determine whether the pH was checked by the laboratory and notes that it was not checked. The validator then examines the Traffic Report contained in the data package and notes that the sampler failed to record what, if any, preservation techniques were utilized. The validator attempts, but fails, to contact the sampler. It cannot be determined if the sample was preserved by the sampler with HCl.

The sampling date for SAA99 was 6/1/95 and the analysis date was 6/21/95, 20 days from sampling. The aqueous volatile samples exceeded the technical holding time criteria for aromatics and non-aromatics. The validator examines the Form I and notes that benzene, toluene, ethylbenzene, chlorobenzene, and xylenes (aromatics) are not detected and that acetone (non-aromatic) is reported at 30 ug/L. The validator reports the benzene, toluene, ethylbenzene, chlorobenzene, and xylenes non-detects as rejected (R), the non-aromatic non-detects as (UJ), and acetone as 30J on the Data Summary Table. The validator notes in the Data Validation Memorandum that the sample data are qualified based on improper preservation (without acid) and exceeded technical holding times.

Example #2: (Improper preservation (refrigeration); Holding times met)

Volatile air samples SAA11-SAA22 were analyzed by the most recent Region I analytical specification for Method TO-1. The laboratory noted in the data package narrative that the samples were received on a Friday afternoon and remained unrefrigerated in the shipping area for over 2 days. The laboratory further noted that this area has no climate control and that temperatures routinely exceed that of the sample storage area by 15-20°C. The validator uses professional judgment to estimate (J) positive detects and reject (R) non-detects in all samples on the Data Summary Table due to the exposure to excessive heat over the 2 day period and discusses this problem in the Data Validation Memorandum.

Example #3: (Proper preservation; Analysis holding time exceeded)

Volatile soil sample SAA33 was sampled on 8/1/95 and was received at the laboratory on 8/2/95. Upon examination of the Traffic Report and the laboratory sample receipt and tracking information, the validator determines that the sample was shipped and stored at 4°C and was light protected. As noted in the data package narrative, due to a laboratory tracking error, the laboratory analyzed the sample following CLP SOW OLM03.2 on 8/18/95, 17 days from the sampling date. The validator estimates (J) the positive detects of sample SAA33 and estimates (UJ) the non-detects on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

Example #4: (Proper preservation; Extraction holding time grossly exceeded)

Semivolatile soil sample SAA44 was sampled on 8/1/95 and received at the laboratory on 8/2/95. Upon examination of the Traffic Report, laboratory receipt information, and sample tracking records, the validator determines that the sample was properly preserved at 4°C and was light protected. The sample was not extracted until 9/1/95, 31 days from sampling date, due to a laboratory tracking error and extraction holding times were grossly exceeded. The validator estimates (J) the positive detects of sample SAA44 and rejects (R) the non-detects on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

II. GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING)

A. OBJECTIVE

Gas chromatograph/mass spectrometer (GC/MS) instrument performance (tuning) checks are performed to ensure proper mass calibration and resolution, identification and to some degree, sensitivity.

B. CRITERIA

GC/MS instrument performance (tuning) criteria are not sample specific. Since conformance is determined using standard materials, these criteria should be met under all circumstances. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from tuning anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify from the reported results that the mass scale is correct (amu assignments are accurate) and that the ion abundance QC acceptance criteria specified in the method were met for each 12-hour period that samples were analyzed.	1. a. If tabulated result forms are not present for each 12-hour period for which samples are analyzed, then the validator should contact the laboratory to obtain the tabulated forms. b. If the mass scale is incorrect and amu assignments are inaccurate, then the validator should reject (R) all data associated with that tune. The data should be returned to the laboratory and payment denied. c. If ion abundance QC acceptance criteria are not met, then professional judgment should be used to determine to what extent the data may be utilized. The most important factors to consider are the empirical results that are unrelated to retention time and type of instrumentation.

C. EVALUATION	D. ACTION
*2. Compare the reported tuning results on each GC/MS Tuning and Mass Calibration Form with each raw data mass listing and mass spectrum submitted. Verify that the laboratory has not made any transcription or calculation errors.	2. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*3. If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the spectra are obtained from chromatographic peaks that should be free from coelution problems, background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract or method specifications are contrary to quality assurance objectives and are, therefore, unacceptable.	3. If the validator has reason to believe that tuning/instrument performance checks were achieved using non-compliant techniques, then the performance and procedures of the laboratory merit further investigation.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.2, C.3

E. EXAMPLES

Example #1: (Ion abundance criteria not met for several ions)

The validator examines tabulated and raw tuning data generated under CLP SOW OLM03.2 to check for calculation and transcription errors. The validator compares the BFB mass spectrum and mass listing with Form V-A. The ion abundances have not been normalized to ion 95 as per the SOW and, when normalized by the validator, do not meet the SOW ion abundance criteria. The validator notes that the abundance criteria for ions 50, 75, 96, and 174 are exceeded by 25%. The validator uses professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects on the Data Summary Table for samples associated with that tune and discusses this problem in the Data Validation Memorandum.

Example #2: (Ion abundance criteria not met for one ion)

The validator examines tabulated and raw tuning data generated under CLP SOW OLM03.2 to check for calculation and transcription errors. The validator compares the DFTPP mass spectrum and mass listing with Form V-B. The % Relative Abundance for ion 275 is 35% of ion 198 (OLM03.2 criteria for ion 198 is 10.0 - 30.0% of mass 198). The validator uses professional judgment to accept the tune since only one ion abundance slightly exceeds criteria. The validator reviews the mass spectra for all positive hits in samples in accordance with Section XII, Target Compound Identification and determines that all ion abundance ratios are acceptable. The validator discusses the non-compliant tune and justifies the decision to accept the sample data in the Data Validation Memorandum.

Example #3: (Mass calibration error)

The validator examines tabulated and raw DFTPP tuning data generated following method 625 to check for calculation and transcription errors. The validator notes that the tabulated tuning results were acceptable, however, the raw data do not agree with the tabulated results. Upon further review of the raw data, the validator notes that the mass calibration is off by 1 amu. In addition, surrogate recoveries and internal standard areas were unacceptably low. The validator rejects (R) all associated data, returns the data package to the laboratory, and payment is denied. The EPA Site Manager is informed by letter and resampling is subsequently scheduled.

III. INITIAL CALIBRATION

A. OBJECTIVE

Compliance requirements for initial calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Initial calibration data demonstrate that the instrument is capable of satisfactory performance at the beginning of the analytical sequence by producing a linear calibration curve.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. Initial calibration standards containing volatile and semivolatile target and surrogate compounds at method-specific concentrations are analyzed prior to the analysis of any field samples, QC samples, and blanks, or as necessary if the continuing calibration method acceptance criteria are not met. The initial calibration and any associated field samples, QC samples, and blanks must be analyzed within 12 hours of the associated GC/MS instrument performance check.

2. Initial calibration standards must be analyzed using the same instrumental conditions that will be used to analyze field samples, QC samples, and blanks.

3. The mean Relative Response Factors ($\overline{RRF}$s) for all volatile and semivolatile target and surrogate compounds in each initial calibration must be greater than or equal to 0.05.

   The Percent Relative Standard Deviation (%RSD) for all volatile and semivolatile target and surrogate compound RRFs in each initial calibration must be less than or equal to 30.0 percent.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from initial calibration anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Verify that the initial calibration standards were analyzed at the method-required concentrations and frequency, and that the standards were analyzed within 12 hours of the associated GC/MS instrument performance check.	1. a. If the laboratory did not use the required concentrations and/or frequency when analyzing the initial calibration standards, or the standards were not analyzed within 12 hours of the associated GC/MS instrument performance check, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.
b. Verify that the method-required calibration standard(s) was used for calculating sample results if any sample results were calculated using an initial calibration.	b. If the correct method-required calibration standard(s) was not used to quantitate sample results, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*2. Verify that the same instrument parameters were used for sample and calibration analyses, and that the instrument parameters which were utilized met method requirements.	2. If correct instrument parameters (i.e., purge and trap conditions, etc.) were not used for the initial calibration standards and sample analyses, then the validator should contact the laboratory to obtain corrected data and forms. a. If the laboratory is unable to submit a correct initial calibration, then the validator should determine whether a qualitative analysis is of any benefit by reviewing the project Data Quality Objectives. b. If the data are deemed unusable, then the validator should reject (R) all associated data. The data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
3. Verify that the $\overline{\text{RRF}}$s for all volatile and semivolatile target and surrogate compounds are greater than or equal to 0.05 in the initial calibration. Verify that the %RSDs for all volatile and semivolatile target and surrogate compound RRFs do not exceed 30.0% in the initial calibration. Evaluate compounds that fail to meet both %RSD and RRF criteria. **Note:** **The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.**	3. **Situation 1:** If any target compound has a ___ %RSD less than or equal to 30.0% and an RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the initial calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the initial calibration. **Situation 2:** If any target compound has a %RSD greater than 30.0% **and** an RRF greater than or equal to 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound for all samples associated with the initial calibration. b. Estimate (UJ) non-detects for that affected compound for all samples associated with the initial calibration. c. See D.4, Situation 2 Expanded for additional guidance. **Situation 3:** If any target compound has a %RSD greater than 30.0% **and** an RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the initial calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the initial calibration. **Surrogates:** If any surrogate compound fails to meet minimum RRF criteria and/or %RSD criteria, then the validator should use professional judgment to assess the impact of surrogate compound calibration data on the sample results. **See Table VOA/SV-III-1**

C. EVALUATION	D. ACTION
4. Evaluate the cause of a non-linear calibration curve based on 5 or more concentration points.	4. **Situation 2 Expanded:** If the %RSD is greater than 30.0%, and all the initial calibration RRFs for a target compound are greater than or equal to 0.05, then the validator should use professional judgment to determine the need to check the calibration points for the cause of the non-linearity. This is checked by eliminating either the high or the low calibration points and recalculating the %RSD. At the validator's discretion, a more in-depth review to minimize data qualification can be accomplished by considering the following: a. If any target compound has a %RSD greater than 30.0%, and if eliminating either the high point or the low point of the curve does not restore the %RSD to less than or equal to 30.0%, then the validator should: - Estimate (J) positive detects for that affected compound for all samples associated with the initial calibration. - Estimate (UJ) non-detects for that affected compound for all samples associated with the initial calibration. b. If eliminating the high point of the curve restores the %RSD to less than 30.0%, then the validator should: - Accept (A) positive detects in the linear portion of the curve for that affected compound for all samples associated with the initial calibration. - Estimate (J) positive detects at the high end of curve outside of the linear portion for that affected compound for all samples associated with the initial calibration. - Accept (A) non-detects for that affected compound for all samples associated with the initial calibration.

C. EVALUATION	D. ACTION
4. Continued from above.	4. c. If eliminating the low point of the curve restores the %RSD to less than 30.0%, then the validator should: - Accept (A) positive detects in the linear portion of the curve for that affected compound for all samples associated with the initial calibration. - Estimate (J) positive detects at the low end of curve outside linear portion for that affected compound for all samples associated with the initial calibration. - Estimate (UJ) non-detects for that affected compound for all samples associated with the initial calibration. **See Table VOA/SV-III-2**
*5. Check and recalculate the RRF and $\overline{RRF}$ for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	5. If errors greater than 10% are detected in the RRF calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*6. Check and recalculate the %RSD for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	6. If errors greater than 10% are detected in the %RSD calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*7. a. Review Standard Preparation Logs (if provided in the data package) to ensure that primary and secondary initial calibration standard concentrations are accurate and traceable to NIST standards.	7. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to facilitate the validation of sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.
* b. Check and recalculate the initial calibration standard concentration for one volatile and one semivolatile target compound (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.2, C.5, C.6, C.7.a, C.7.b

Table VOA/SV-III-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON THE INITIAL CALIBRATION

Sample Results	QC Criterion $\overline{RRF} \geq 0.05$ %RSD $\leq$ 30.0%	Situation 1 $\overline{RRF} < 0.05$ %RSD $\leq$ 30.0%	Situation 2** $\overline{RRF} \geq 0.05$ %RSD > 30.0%	Situation 3 $\overline{RRF} < 0.05$ %RSD > 30.0%
Detects	A	J	J	J
Non-detects	A	R	UJ	R

** See Table VOA/SV-III-2 for additional guidance.

Table VOA/SV-III-2:

EXPANDED INITIAL CALIBRATION VOA/SV ANALYTE QUALIFICATIONS

Sample Results	Elimination of High or Low Calibration Points %RSD > 30.0%	Elimination of High Calibration Points %RSD $\leq$ 30.0% RRF $\geq$ 0.05	Elimination of Low Calibration Points %RSD $\leq$ 30.0% RRF $\geq$ 0.05
Detects	J	A: On linear portion of curve J: On high end of curve outside linear portion	A: On linear portion of curve J: On low end of curve outside linear portion
Non-detects	UJ	A	UJ

E. EXAMPLES

Example #1: Situation 1 (Low $\overline{RRF}$; Acceptable linearity)

The $\overline{RRF}$ of an initial calibration for benzene is 0.035 which does not meet the 0.05 acceptance criteria. The %RSD of the calibration points for benzene is 19.0%. Due to the low instrument response for benzene, the validator estimates (J) all the positive benzene detects and rejects (R) the benzene non-detects on the Data Summary Table and notes this problem in the Data Validation Memorandum.

Example #2: (Low $\overline{RRF}$; Acceptable linearity; Modified Region I $\overline{RRF}$ criteria)

The $\overline{RRF}$ of an initial calibration for acetone is 0.035 and the %RSD is 12.0%. The site-specific EPA-approved QAPjP documents that modified Region I minimum RRF criteria will be used to validate project data. The modified criteria are:

- The mean initial calibration $\overline{RRF}$ and the continuing calibration RRF for all volatile and semivolatile target and surrogate compounds must be greater than or equal to 0.05 except for the following compounds which must have an initial calibration RRF and a continuing calibration RRF greater than or equal to 0.01: chloromethane, chloroethane, methylene chloride, acetone, carbon disulfide, 1,2-dichloroethane (total), 2-butanone, 1,2-dichloropropane, 4-methyl-2-pentanone, 2-hexanone and surrogates, toluene-d8 and 1,2-dichloroethane-d4.

The validator accepts all acetone positive detects and non-detects in the samples associated with the initial calibration and reports the sample results unqualified on the Data Summary Table. The validator documents the modified data validation criteria in the Data Validation Memorandum.

Example #3: Situation 2 (Acceptable $\overline{RRF}$; High RSD - Elimination of high point)

The validator examines the initial calibration data and notes that the %RSD for tetrachloroethene was 60.0% and the $\overline{RRF}$ was 0.07. Elimination of the high calibration point restored the %RSD to 18.0%. Since linearity was verified for a portion of the tetrachloroethane curve, the validator accepts all positive tetrachloroethene detects on the linear portion of the curve and estimates (J) the positive tetrachloroethene detects on the non-linear portion of the curve. Tetrachloroethene non-detects are accepted. All results are reported on the Data Summary Table and the qualifications are discussed in the Data Validation Memorandum.

Example #4: Situation 2 (Acceptable $\overline{RRF}$; High RSD - Elimination of low point)

The validator examines the initial calibration data and notes that the %RSD for acetone was 70.0% and the RRF was 0.07. Elimination of the low calibration point restored the %RSD to 20.0%. Since linearity was verified for a portion of the acetone curve, the validator accepts all positive acetone detects on the linear portion of the curve and estimates (J) the positive acetone detects on the non-linear portion of the curve. Acetone non-detects are estimated (UJ). All results are reported on the Data Summary Table and the qualifications are discussed in the Data Validation Memorandum.

E. EXAMPLES

Example #5: Situation 3 (Low $\overline{RRF}$; High RSD)

The $\overline{RRF}$ for trichloroethene is 0.029 which is well below the 0.05 acceptance criteria and the %RSD for trichloroethene is 65.0% which is well above the acceptance criteria. Linearity cannot be achieved by eliminating the high or low points. Due to erratic instrument performance, the validator uses professional judgment to estimate (J) positive trichloroethene detects and reject (R) trichloroethene non-detects on the Data Summary Table and discusses sample qualifications in the Data Validation Memorandum.

IV. CONTINUING CALIBRATION

A. OBJECTIVE

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the daily relative response factors on which target compound quantitation is based and checks the stability of instrument response on a day-to-day basis.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. Continuing calibration standards containing volatile and semivolatile target and surrogate compounds at method-specified concentrations are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of the field samples, QC samples, and blanks.

2. Continuing calibration standards must be analyzed using the same instrumental conditions which were used to analyze the initial calibration and that will be used to analyze field samples, QC samples, and blanks.

3. The continuing calibration Relative Response Factors (RRFs) for all volatile and semivolatile target and surrogate compounds must be greater than or equal to 0.05.

   The Percent Difference (%D) between the most recent initial calibration RRF and the continuing calibration RRF for all volatile and semivolatile target compounds and surrogate compounds must not exceed ± 25.0 percent.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from continuing calibration anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Verify that the continuing calibration standard was analyzed at the required concentration and frequency, and that the standard was analyzed within 12 hours of the associated GC/MS instrument performance check.	1. a. If the laboratory did not use the required concentration and/or frequency when analyzing the continuing calibration standard or the standard was not analyzed within 12 hours of the associated GC/MS instrument performance check, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.
b. Verify that quantitation was performed using a continuing calibration analyzed within 12 hours of the field samples.	b. If the correct continuing calibration standard was not used to quantitate sample results, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*2. Verify that the same instrument parameters were used for sample and calibration analyses, and that instrument parameters which were utilized met method requirements.	2. If the same method-required instrument parameters (i.e., purge and trap conditions, etc.) were not used for the continuing calibration standards and field sample analyses, then the validator should contact the laboratory. a. If the laboratory is unable to submit a correct continuing calibration, then the validator should determine whether a qualitative analysis is of any benefit by reviewing the project Data Quality Objectives. b. If the data are deemed unusable, then the validator should reject (R) all associated data. The data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
3. Verify that the continuing calibration was compared to the most recent initial calibration. Verify that RRFs for all volatile and semivolatile target and surrogate compounds are greater than or equal to 0.05 in the continuing calibration. Verify that the %D between initial calibration RRF and continuing calibration RRF for all volatile and semivolatile target and surrogate compounds is less than or equal to ± 25.0%. Evaluate compounds that fail to meet both %D and RRF criteria. **Note:** **The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.**	3. If the continuing calibration was not compared to the most recent initial calibration, then the validator should have the laboratory recalculate %Ds based on the correct initial calibration and resubmit all affected data and forms. **Situation 1:** If any target compound has a %D between the initial calibration and the continuing calibration which is less than or equal to ± 25.0% **and** a continuing calibration RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the continuing calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the continuing calibration. **Situation 2:** If any target compound has a %D between the initial and continuing calibration of greater than ± 25.0% **and** a continuing calibration RRF greater than or equal to 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound for all samples associated with the continuing calibration. b. Estimate (UJ) non-detects for that affected compound for all samples associated with the continuing calibration. **Situation 3:** If any target compound has a %D between the initial and continuing calibration of greater than ± 25.0% **and** a continuing calibration RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the continuing calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the continuing calibration.

C. EVALUATION	D. ACTION
3. Continued from above.	3. Continued from above. **Surrogates:** If any surrogate compound fails to meet minimum RRF criteria and/or %D criteria, then the % surrogate recoveries in the samples, QC samples and blanks associated with the continuing calibration may be biased high or low resulting in unacceptable surrogate recoveries. In this case, the validator should use professional judgment to assess the impact of surrogate compound calibration data on the sample results. **See Table VOA/SV-IV-1**
*4. Check and recalculate the RRF for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	4. If errors greater than 10% are detected in the RRF calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*5. Check and recalculate the %D for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	5. If errors greater than 10% are detected in the %D calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*6. a. Review Standard Preparation Logs (if available in the data package) to ensure that primary and secondary continuing calibration concentrations are accurate and traceable to NIST standards.	6. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to validate sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.
* b. Check and recalculate the continuing calibration standard concentration for one volatile and one semivolatile target compound (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.2, C.4, C.5, C.6.a, C.6.b

Table VOA/SV-IV-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON THE CONTINUING CALIBRATION

Sample Results	**QC Criteria** RRF ≥ 0.05 %D ≤ ± 25.0%	**Situation 1** RRF < 0.05 %D ≤ ± 25.0%	**Situation 2** RRF ≥ 0.05 %D > ± 25.0%	**Situation 3** RRF < 0.05 %D > ± 25.0%
Detects	A	J	J	J
Non-Detects	A	R	UJ	R

E. EXAMPLES

Example #1: Situation 1 (Low RRF; Acceptable %D)

The RRF for 2-butanone in a continuing calibration is 0.035 and the %D is 10.0%. Due to the low response, the validator estimates (J) all 2-butanone positive detects and rejects (R) all 2-butanone non-detects that are associated with this continuing calibration on the Data Summary Table. The validator discusses the qualification of sample data in the Data Validation Memorandum.

Example #2: (Low RRF; Acceptable %D; Modified Region I RRF criteria)

The RRF for acetone in a continuing calibration is 0.025 and the %D is 12.0%. The site-specific EPA-approved QAPjP documents that modified Region I minimum RRF continuing calibration data validation criteria will be used to validate project data. The modified criteria are:

- The mean initial calibration $\overline{RRF}$ and the continuing calibration RRF for all volatile and semivolatile target and surrogate compounds must be greater than or equal to 0.05 except for the following compounds which must have an initial calibration RRF and a continuing calibration RRF greater than or equal to 0.01: chloromethane, chloroethane, methylene chloride, acetone, carbon disulfide, 1,2-dichloroethane (total), 2-butanone, 1,2-dichloropropane, 4-methyl-2-pentanone, 2-hexanone and surrogates, Toluene-d8 and 1,2-dichloroethane-d4.

The validator reviews the acetone mass spectra for positive detects in samples and determines that all mass spectral identification criteria are met. The validator accepts all acetone positive detects and non-detects in the samples associated with the continuing calibration and reports the sample results unqualified on the Data Summary Table. The validator documents the modified data validation criteria in the Data Validation Memorandum.

E. EXAMPLES

Example #3: Situation 2 (Acceptable RRF; High %D)

The RRF for methylene chloride in a continuing calibration is greater than 0.05 and the %D between the initial and continuing calibration for methylene chloride is 45.0%. The validator reviews the initial calibration, continuing calibration, and blank data, and determines that an intermittent methylene chloride contamination problem exists in the laboratory which may contribute to the high %D. The validator estimates (J) all methylene chloride positive detects and estimates (UJ) the methylene chloride non-detects in the associated samples on the Data Summary Table. The validator discusses this problem in the Data Validation Memorandum.

Example #4: Situation 3 (Low RRF; High %D)

The RRF for N-nitroso-di-n-propylamine in a continuing calibration is 0.001 and the %D is 110.0%. Due to low and unstable instrument response to N-nitroso-di-n-propylamine, the validator determines that both the quantitation limits and positive detects for N-nitroso-di-n-propylamine are unusable. Therefore, the validator rejects (R) all N-nitroso-di-n-propylamine results that are associated with this continuing calibration on the Data Summary Table. The validator discusses the qualification of sample data in the Data Validation Memorandum.

V. BLANKS

A. OBJECTIVE

The purpose of blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory and/or field activities and to subsequently assess their contribution to measurement error. The criteria for evaluation of laboratory blanks (method blanks and instrument blanks) may be applied to any blank associated with the samples. If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent measurement error associated with the entire data set, or if the problem is an isolated occurrence limited to specific samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. The frequency and types of blanks collected and analyzed must support the site-specific Data Quality Objectives (DQOs) as documented in the EPA approved QAPjP or SAP. Different types of blanks may be used to identify the source of potential contamination resulting in analytical and/or sampling measurement error. The following table lists types of blanks, the environment of those blanks, and the possible sources of contamination associated with those blanks:

BLANK	LABORATORY/FIELD	IDENTIFIES CONTAMINATION FROM
Method Blank	Laboratory	Laboratory and Reagents
Instrument Blank	Laboratory	Instrumentation
Storage Blank	Laboratory	Storage Environment
Trip Blank	Field	Transit Environment
Bottle Blank	Field	Sample Container
Equipment Blank (Rinsate)	Field	Sampling Equipment

Note: Aqueous equipment (rinsate) blank results, bottle blank results and trip blank results will be used to determine blank action levels for aqueous samples based on a volume of 1 liter of blank sample. Ideally soil/sediment blanks should be used to determine soil/sediment blank actions for soil/sediment samples based on a known weight of blank sample. However, often aqueous equipment blanks, bottle blanks and trip blanks are collected to evaluate contamination associated with soil/sediment sampling. **Aqueous equipment (rinsate) blank results, bottle blank results and trip blank results will not be used to determine blank action levels for non-aqueous samples.** Compounds that are present in both the non-aqueous sample and the associated aqueous equipment blank, bottle blank or trip blank will be flagged EB (Equipment Blank), BB (Bottle Blank) or TB (Trip Blank), respectively. The degree of "sampling error" that this flagged sample result represents will be left to the determination of the end user.

2. Method Blanks:

   a. A volatile method blank must be analyzed after the continuing calibration and before any samples, QC samples, or other types of blanks (i.e., storage blanks). The VOA method blank must be analyzed at least once during every 12 hour time period on each GC/MS system used to analyze samples.

   b. A semivolatile method blank must be extracted with each sample delivery group or each 20 samples of similar matrix in each sample delivery group or whenever a sample extract procedure is performed. The method blank must undergo all cleanup procedures performed on samples, i.e., GPC, Silica Gel, etc. used in sample preparation. The semivolatile method blank extract must be analyzed on each GC/MS system used to analyze samples.

3. Instrument Blanks:

   a. An instrument blank must be analyzed after any sample that exceeds the calibration range to check that the blank is free of interference and the system is not contaminated.

   b. For purge and trap volatile organic analysis, an instrument blank must be analyzed in the same purging position as a sample that exceeds the calibration range to check that the blank is free of interference and the purging position is not contaminated.

   c. Instrument blanks and apparatus blanks for each cleanup procedure, including GPC and Silica Gel, etc. used in sample preparation must be analyzed prior to sample analysis.

4. Storage Blanks:

   a. A volatile storage blank vial (in duplicate) must be prepared by the laboratory when the first samples of the sample delivery group are received. The storage blank is stored with the samples and analyzed after all the samples in the sample delivery group have been analyzed.

5. All blanks should be spiked with surrogate compounds and internal standards according to the method. Note: CLP OLM03.2 does not require that the GPC instrument blank be spiked with internal standards or surrogates.

   a. Blank internal standards must meet method internal standard QC acceptance criteria.

   b. Blank surrogate compounds must meet method surrogate compound QC acceptance criteria.

6. No contaminants should be present in the blanks.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from blank anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. **Qualification should be based upon a comparison of the sample concentration(s) with the highest blank concentration associated with the sample delivery group.** However, in cases of specific instrument, storage and/or method blank contamination, the validator should use professional judgment to qualify only those samples associated with that isolated blank contamination. Likewise, the validator may need to apply blank qualifications to a sample delivery group based on associated equipment, trip, or bottle blank data that exists in another sample group data package. **Sample results must not be corrected by subtracting any blank values.**
1. a. Verify that the correct number and type of blanks have been collected and analyzed in accordance with the EPA approved QAPjP or SAP.	1. a. If the correct number and type of blanks have not been collected and analyzed, then the validator should note this deviation from the EPA approved QAPjP or SAP in the Data Validation Memorandum. The validator should use professional judgment to qualify sample data when blank data are absent. When required trip, equipment (rinsate) or bottle blanks are not identified on the chain of custody, then the validator must contact the sampler or site project manager to obtain this information and note this contact on the Blank Analysis validation worksheet.
b. Ascertain if aqueous equipment (rinsate) blanks, aqueous bottle blanks or aqueous trip blanks have been collected with non-aqueous samples to identify sources of field contamination.	b. If positive results are detected in the aqueous equipment (rinsate) blanks, bottle blanks and/or trip blanks and the associated non-aqueous samples, then the validator should flag (EB, BB or TB) those detected compounds in the associated non-aqueous samples to indicate to the end user that an indeterminate amount of sampling error has potentially affected the sample results. (See example #4)

C.	EVALUATION	D.	ACTION
2. a.	Verify that a VOA method blank analysis has been reported per matrix, per concentration level, per extraction batch (for medium-level VOAs only) after each continuing calibration and for each 12-hour time period on each GC/MS system used to analyze samples.	2. a.	If VOA method blanks were not analyzed at the required frequency and for each matrix, concentration level, extraction batch (for medium-level VOAs only), and on each GC/MS system used to analyze samples, then the validator should use professional judgment to determine whether the associated sample data should be qualified.
b.	Verify that a semivolatile method blank analysis has been reported once per matrix, per concentration level, per extraction technique and SDG, and on each GC/MS system used to analyze sample extracts.	b.	If semivolatile method blanks were not analyzed at least once for each matrix, concentration level, extraction technique and batch, and on each GC/MS system used to analyze sample extracts, then the validator should use professional judgment to determine whether the associated data should be qualified.
* c.	Verify from the raw data that the extraction and/or analysis dates and times, sample IDs, file IDs, instrument IDs, etc. are accurately reported on the tabulated result forms.	c.	If review of the raw data reveals discrepancies and/or transcription errors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C.	EVALUATION	D.	ACTION
3. a.	Verify from the Blank Summary form and Form Is that a VOA instrument blank was analyzed after each sample that exceeded the instrument calibration range.	3. a.	If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), then sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification and/or quantitation, and to determine whether the affected sample data should be qualified or rejected. If cross-contamination is suggested, then this should be noted in the Data Validation Memorandum.
* b.	Verify from the raw data, the Blank Summary form, and Form Is that a VOA instrument blank was analyzed in the same purging/sparging vessel (i.e., same position in the autosampler) as the sample that exceeded the instrument calibration range.	b.	If an instrument blank was not analyzed in the same purging vessel used to analyze a sample that exceeded the instrument calibration range, then sample analysis results generated in that purging vessel after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification and/or quantitation, and to determine whether the affected sample data should be qualified or rejected. If cross-contamination is suggested, then this should be noted in the Data Validation Memorandum.
* c. i.	Verify from the raw GPC data that a GPC instrument blank was analyzed after the GPC calibration and prior to sample analysis.	c. i.	If a GPC instrument blank was not analyzed at the method-required frequency, then the validator should evaluate the method blank data and use professional judgment to qualify sample data associated with that GPC cleanup procedure.
* ii.	Verify from the raw Silica Gel data that a Silica Gel Column reagent blank was analyzed prior to sample analysis.	ii.	If a Silica Gel Column reagent blank was not analyzed at the method-required frequency, then the validator should evaluate the method blank data and use professional judgment to qualify sample data associated with that Silica Gel Column cleanup procedure.

C. EVALUATION	D. ACTION
4. Verify that a VOA storage blank was analyzed for each sample delivery group and that it was analyzed after all field samples were analyzed.	4. If a VOA storage blank was not analyzed at the correct frequency, then the validator should use professional judgment to accept or qualify sample data.
5. a. Verify that the blank internal standard areas and retention times and surrogate compound recoveries meet method QC acceptance criteria.	5. a. If blank internal standard areas and/or retention times and/or surrogate compound recoveries do not meet method QC acceptance criteria, then the validator should use professional judgment in applying blank actions. The possibility of false positives or false negatives being incorrectly reported for the blank should be evaluated.
* b. Check 10% of the raw data for each blank to verify that internal standard areas and retention time data, have been correctly transcribed to tabulated forms and that surrogate compound recovery data have been correctly calculated and transcribed to tabulated forms. Review the blank chromatograms, quantitation reports, and mass spectra to ensure that no false positives or false negatives have been reported.	b. If the laboratory has reported a false positive or a false negative and/or has incorrectly transcribed and/or calculated data, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. Review the reported results of all associated blanks on the tabulated forms.	6. If a contaminant is found in a blank but not in the sample, no action is taken. If a contaminant is found in both a blank and a sample, then the validator should note this problem in the Data Validation Memorandum and qualify the data according to the following guidance: **Note: If the blank action level for a compound is determined using the value from a bottle blank, equipment blank or trip blank, then the positive values in the bottle, equipment, or trip blank should be reported unqualified on the Data Summary Tables. However, if the blank action is determined using the value from a laboratory blank (e.g., method, storage, or instrument), then the positive values in the trip, bottle, or equipment blanks should be qualified. (See example #6)**
a. Determine if any target compounds are present at or above the quantitation limit/CRQL in any of the blanks.	a. **Target Compound Contaminants at or Above the Quantitation Limit/CRQL:** i. If positive sample results for a compound are greater than 5 times the concentration in any blank (with the exception of the common laboratory contaminants in Section V.C.6.b), then the compound's concentration should be reported as unqualified. ii. If positive sample results for a compound are less than or equal to 5 times the concentration of the compound in any blank (with the exception of the common laboratory contaminants in Section V.C.6.b) but are greater than the quantitation limit, then the sample quantitation limit for that compound should be elevated to the concentration found in the sample and reported as not detected (U). The validator should use professional judgment to determine if further elevation of the quantitation limit is required. (See example #1 - 5x rule)

C. EVALUATION	D. ACTION
6. Continued	6. Continued Note: The validator should note that blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" or "10x" criteria, such that a comparison of the total amount of contamination is actually made. (See example #5). Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample data is deemed necessary. If the validator determines that the contamination originates from a source other than the sample, the sample data should be qualified. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. In this case, the "5x" rule may not apply; the target compound should be reported as not detected (U), and an explanation of the data qualification rationale should be provided in the Data Validation Memorandum.
b. Determine if any common volatile laboratory contaminants (acetone, methylene chloride, 2-butanone) or any common semivolatile laboratory contaminants (phthalates) are present at or above the quantitation limit/CRQL in any of the blanks.	b. **Common Laboratory Contaminants at or Above the Quantitation Limit/CRQL:** i. If positive sample results for a common laboratory contaminant compound are greater than 10 times the concentration in any blank, then the compound's concentration should be reported as unqualified (See example #3 - 10x rule). ii. If positive sample results for a common laboratory contaminant compound are less than or equal to 10 times the concentration of the compound in the blank, then the sample quantitation limit should be elevated to the concentration found in the sample and reported as not detected (U). The validator should use professional judgment to determine if further elevation of the quantitation limit is required.

C. EVALUATION	D. ACTION
6. c. Determine if low level contamination below the quantitation limit exists in any of the blanks.	6. c. **Common Laboratory Contaminants and Target Compounds Below the Quantitation Limit/CRQL:** i. If a positive sample result is reported at less than the quantitation limit and is also less than the blank action level, then the sample quantitation limit should be reported on the Data Summary Tables (See example #2 - 5x rule). ii. If a positive sample result is reported at less than the quantitation limit but is greater than the blank action level, then the estimated sample result should be reported on the Data Summary Tables. iii. If several target compounds are found at low levels, below the quantitation limit, in the laboratory blank(s), it may indicate a systemic problem in the laboratory and should be noted in the Data Validation Memorandum. iv. If low level contamination exists solely in the trip, bottle or equipment (rinsate) blanks, then the validator should notify the sampler. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.
d. Determine if gross contamination, greater than 10x CRQL for any analyte, exists in any of the blanks.	d. **Gross Contamination** i. If gross contamination, greater than 10x CRQL for any analyte, exists in any blank, then the validator should reject (R) all affected compounds in samples associated with that blank due to the interference. This serious problem should be discussed in the Data Validation Memorandum. ii. If gross contamination exists solely in the trip, bottle or equipment (rinsate) blanks, then the validator should notify the sampler. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.
e. Determine if instrument contamination is isolated to specific sample runs.	e. If contamination is limited to a few samples due to instrument contamination, then the validator may use professional judgment in qualifying sample data from isolated sample runs.

C. EVALUATION	D. ACTION
*6. f. Review the raw data (chromatograms, mass spectra and quantitation reports) to confirm the presence of target and non-target compounds in the blanks and to evaluate the presence of additional contaminants.	6. f. If review of raw data suggests that additional contaminants are present or, conversely, the review indicates false positives have been reported, then the validator should contact the laboratory to obtain additional information and/or have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
7. Evaluate the overall contamination in each type of blank to ascertain probable source(s) of contamination. For example, a contaminated equipment blank might indicate decontamination problems if the method, storage, instrument, and bottle blanks were all clean.	7. If a review of the various types of blanks identifies a potential source of blank contamination, then the validator should discuss this problem in the Data Validation Memorandum. The validator should identify whether the measurement error is a result of either sampling or analytical error or both (see Data Validation Manual p.1).

* **Note: The following subsections are applicable only to a Tier III data validation. C.2.c, C.3.b, C.3.c.i, C.3.c.ii, C.5.b, C.6.f**

E. EXAMPLES

Example #1: (Bottle blank target compound contaminant ≥ CRQL, sample result < 5x blank action level)

Carbon disulfide is detected in a water sample at greater than the CRQL, but less than 5x the bottle blank concentration.

5x Rule

	ug/L
Bottle Blank Result	20
CRQL	10
Carbon disulfide Sample Result	80
Action Level	100
Qualified Sample Result	80 U

In this case, the laboratory sample result for carbon disulfide is less than 100 ug/L (5 x 20) and the validator reports the carbon disulfide result as non-detected at an elevated quantitation limit on the Data Summary Table. Carbon disulfide was not detected in the method blank but was detected at 12 ppb in the trip blank. The validator notes in the Data Validation Memorandum that the bottle blank was contaminated with carbon disulfide, documents the lot number of the sample bottle, and alerts the site project manager regarding a contaminated lot of bottles.

E. EXAMPLES

Example #2: (Instrument blank target compound contaminant < CRQL, sample result < 5x blank action level)

Ethylbenzene is detected in a water sample at less than the CRQL and also less than 5x the instrument blank concentration. The instrument blank contained the highest concentration of ethylbenzene of all blanks analyzed. In addition, all field samples analyzed were associated with the same contaminated instrument blank.

5x Rule

	ug/L
Instrument Blank Result	5
CRQL	10
Ethylbenzene Sample Result	8 J
Action Level	25
Qualified Sample Result	10 U

In this case, the ethylbenzene sample result is less than 25 ug/L (5 x 5) and is reported non-detected at the CRQL on the Data Summary Table. This problem is noted in the Data Validation Memorandum.

Example #3: (Common laboratory contaminant ≥ CRQL, sample result > 10x blank action level)

Bis(2-ethylhexyl)phthalate is detected in a water sample at greater than 10x the method blank concentration.

10x Rule

	ug/L
Blank Result	20
CRQL	10
Bis(2-ethylhexyl)phthalate Sample Result	220
Action Level	200
Qualified Sample Result	220

In this case, the bis(2-ethylhexyl)phthalate sample result exceeded the blank action level of 200 ug/L (10 x 20) and the bis(2-ethylhexyl)phthalate sample result is reported unqualified on the Data Summary Table.

Example #4: (Blank target compound contamination in aqueous equipment blank collected with soil samples)

An equipment blank (rinsate) was included in a sample delivery group of soil samples. The validator examines the data and finds that the equipment blank contains 40 ug/L of bis(2-ethyl-hexyl)phthalate. The validator then reviews all other blank data and finds no further bis(2-ethylhexyl)phthalate contamination. One soil sample contains 60 ug/kg of bis(2-ethylhexyl) phthalate. The validator reports the soil sample result on the Data Summary Table as 60 (EB) to indicate to the end user that sampling error has potentially affected the sample results and notes this information in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (Application of sample weights and volumes with 5x Rule)

Soil sample TAA35 was analyzed as a routine semivolatile soil sample under CLP SOW OLM03.2 and contained 70% solids. The validator reviewed the sample results and found naphthalene (560 ug/kg) and pyrene (460 ug/kg) in sample TAA35. The method blank was found to be contaminated with pyrene (420 ug/kg) and naphthalene (430 ug/kg). These blank results were reported by the laboratory on a dry weight basis and were the maximum levels of contamination found for these compounds in this sample delivery group. The validator determines the blank action level by applying the 5x rule. The method blank action level for pyrene was calculated to be 2100 ug/kg (420 x 5), and the action level for naphthalene was calculated to be 2150 ug/kg (430 x 5).

The validator calculates the sample quantitation limits for naphthalene and pyrene for 30.0 g extracted:

$$\text{naphthalene QL} = \frac{\text{CRQL}}{\text{\% solids}} = \frac{330\ \text{ug/kg}}{0.7} = 471\ \text{ug/kg}$$

$$\text{pyrene QL} = \frac{\text{CRQL}}{\text{\% solids}} = \frac{330\ \text{ug/kg}}{0.7} = 471\ \text{ug/kg}$$

The validator applies the following action to the naphthalene and pyrene results for sample TAA35:

Naphthalene		Pyrene	
5x Rule		5x Rule	
	ug/kg		ug/kg
Blank Result	430	Blank Result	420
CRQL	471	CRQL	471
Sample Result	560	Sample Result	460 J
Action Level	2150	Action Level	2100
Qualified Sample Result	560 U	Qualified Sample Result	471 U

- The sample quantitation limit for naphthalene is elevated to the sample concentration result on the Data Summary Table and is reported as 560U, since the result falls between the sample quantitation limit and the blank action level.

- The pyrene sample result on the Data Summary Table is replaced with the sample quantitation limit and is reported as 471U, since the positive sample detect of 460 ug/kg is below both the sample quantitation limit and the blank action level.

The validator notes all actions taken in the Data Validation Memorandum.

E. EXAMPLES

Example #6: (Application of laboratory blank action levels to trip blanks)

The method blank for an aqueous batch of volatile samples was contaminated with 25 ug/L of trichloroethene. The trip blank for this batch of samples was contaminated with 22 ug/L of trichloroethene and 15 ug/L of ethylbenzene. Since trichloroethene was detected in both the method blank and the trip blank, the highest detected concentration is used to determine the blank action level. The method blank concentration is, therefore, used to determine the blank action level for trichloroethene.

Trichloroethene	ug/L	Ethylbenzene	ug/L
Method Blank Result	25	Method Blank Result	10 U
Trip Blank Result	22	Trip Blank Result	15
CRQL	10	CRQL	10
Blank Action Level	125 (5x25)	Blank Action Level	75 (5x15)

The trichloroethene positive detect in the trip blank is qualified and reported as 22U ug/L on the Data Summary Table. The blank action level for ethylbenzene is determined using the value from the trip blank and, as a result, the ethylbenzene positive detect in the trip blank is reported unqualified as 15 ug/L on the Data Summary Table.

VI. SURROGATE COMPOUNDS

A. OBJECTIVE

Sample matrix effects and laboratory performance on individual samples are assessed by spiking the samples with surrogate compounds prior to extraction and/or analysis and determining their recoveries. Evaluation of surrogate recoveries is not necessarily straightforward. Interfering matrix effects, including high concentrations of target and/or non-target analytes, are frequently outside control of the laboratory and may present relatively unique problems. Therefore, the evaluation and review of the surrogate compound results are frequently subjective, demanding extensive analytical experience and professional judgment. Accordingly, this section consists primarily of guidance with several optional approaches suggested.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. The correct method-required surrogate compounds must be added to all samples, QC samples and blanks at the proper concentrations.

2. a. Recoveries for surrogate compounds in samples, QC samples and blanks must be within the QC acceptance criteria specified in the method.

   b. Recoveries for advisory surrogate compounds in samples, QC samples, and blanks must be greater than or equal to 10%.

3. Volatile samples must be reanalyzed in accordance with method requirements if surrogate compound recoveries are outside the method QC acceptance criteria.

4. Semivolatile samples must be reextracted and/or reanalyzed in accordance with method requirements if surrogate compound recoveries are outside the method QC acceptance criteria.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from surrogate compound anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct compounds were used as surrogate compounds and were added at the required concentrations and frequencies to all samples, QC samples and blanks.	1. a. If surrogate compounds were not added to all samples, QC samples and blanks, were added at the wrong concentration (for example a sample was "double" spiked) or an incorrect compound was used, then the validator should use professional judgment to qualify or reject sample data.
	b. If surrogate compounds were diluted out of a sample, then the validator should use professional judgment to qualify or reject sample data. Greater than five-fold dilutions result in surrogate recovery data that may be analytically unusable.

C. EVALUATION	D. ACTION
2. a., b., c. Verify that no surrogate compound recovery is outside the method QC acceptance criteria for volatile field and QC samples and verify that no more than one base/neutral surrogate or one acid surrogate is outside method QC acceptance criteria for semivolatile field and QC samples.	2. a. If one surrogate in the VOA fraction or two or more surrogates in the base/neutral or acid fractions have recoveries greater than the upper method QC acceptance limit, then the validator should: i. Estimate (J) all volatile, base/neutral or acid positive detects in the affected sample fraction. ii. Accept all volatile, base/neutral or acid non-detects in the affected sample fraction. b. If one surrogate in the VOA fraction or two or more surrogates in the base/neutral or acid fractions have recoveries greater than or equal to 10% but less than the lower method QC acceptance limit, then the validator should: i. Estimate (J) all volatile, base/neutral or acid positive detects in the affected sample fraction. ii. Estimate (UJ) all volatile, base/neutral or acid non-detects in the affected sample fraction. c. If any surrogate compound in a fraction recovers at less that 10%, then the validator should: i. Estimate (J) all volatile, base/neutral or acid positive detects in the affected sample fraction. ii. Reject (R) all volatile, base/neutral or acid non-detects in the affected sample fraction.

C. EVALUATION	D. ACTION
2. d. Verify that no advisory surrogate compound recovers at less than 10%.	2. d. If any advisory surrogate compound in a fraction recovers at less than 10%, then the validator should use professional judgment to qualify the sample data, taking into account the recoveries of all other surrogate compounds and the compounds of concern at the site.
e. Determine if blank surrogate recovery results meet method QC acceptance criteria.	e. In the special case of a blank analysis with surrogate compound recoveries outside the method QC acceptance criteria, the validator must give special consideration to the validity of the associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if most of the samples including other types of blanks in the batch show acceptable surrogate compound recoveries, then the validator may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems should be noted in the Data Validation Memorandum. All samples that were extracted with or analyzed after an out of control blank should be noted in the Data Validation Memorandum. Also, note in the Data Validation Memorandum if there are potential contractual problems associated with the failure to reextract and/or reanalyze blanks that were outside the method QC acceptance criteria.
3. For aqueous and low/medium soil volatile samples, verify that if surrogate compound recoveries are outside the method QC acceptance criteria, then the required reanalysis was performed to confirm that the non-compliance was due to sample matrix effects rather than poor laboratory performance.	3. If a laboratory fails to reanalyze a sample which is out of specification, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.
4. For semivolatile samples, verify that if surrogate compound recoveries are outside the method QC acceptance criteria, then the required reextraction/reanalysis was performed to confirm that the noncompliance was due to sample matrix effects rather than poor laboratory performance.	4. If a laboratory fails to reextract and reanalyze a sample which is out of specification, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*5. a. Check raw data (e.g., chromatograms and quantitation reports) to verify that surrogate recoveries were reported accurately on the Surrogate Recovery Forms.	5. a. If there are any transcription errors, then the validator should contact the laboratory to obtain corrected raw data and forms.
* b. Ten percent of the surrogate compound recovery data should be checked for calculation and/or transcription errors. If errors are detected in this ten percent, then an additional ten percent of the data should be checked. If errors are found in the additional ten percent, then all surrogate compound recovery calculations and transcriptions in the data package should be checked.	b. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note:** **The following subsections are applicable only to a Tier III data validation:**

C.5.a, C.5.b

Table VOA/SV-VI-1:

QUALIFICATION OF VOLATILE/SEMIVOLATILE ANALYTES BASED ON SURROGATE COMPOUND RECOVERIES

Surrogate Compound Recovery				
Sample Results	**one or more surrogates < 10%**	**one VOA, two B/N or two acid surrogates 10% ≤ %Rec < LL**	**all VOA, one B/N or one acid surrogate LL ≤ %Rec ≤ UL**	**one VOA, two B/N or two acid surrogates > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

E. EXAMPLES

Example #1: (Two low acid surrogate recoveries - one of which recovered at less than 10%)

Semivolatile aqueous sample SA125, analyzed by CLP SOW OLM03.2, recovered two acid surrogate compounds, phenol-d_5 and 2-fluorophenol, below the method QC acceptance criteria. In addition, the phenol-d_5 recovered at less than 10%. All other surrogate recoveries met QC criteria. The following table lists the surrogate spike recoveries and the method QC acceptance criteria:

Sample No.	Phenol-d_5 % Recovery	QC Acceptance Criteria (aqueous)	2-Fluorophenol % Recovery	QC Acceptance Criteria (aqueous)
SA125	8	10-110	15	21-110

The sample was reextracted and reanalyzed with similar results. The validator examines the PE sample results, and determines that the laboratory accurately prepared and analyzed the QC samples. Also, all internal standard areas were acceptable and the MS/MSD results for sample SA126 did not show a low bias for acid compounds. Therefore, the validator estimates (J) positive detects and rejects (R) non-detects for the acid fraction of sample SA125 on the Data Summary Table. The validator notes in the Data Validation Memorandum that the low recoveries may be due to matrix interferences specific to sample SA125.

E. EXAMPLES

Example #2: (One low volatile surrogate recovery)

Volatile drinking water sample SA925, analyzed by the Region I 524.2 method-Revision 8.0, had one surrogate compound, 1,2-dichlorobenzene-d_4, recover below the method QC acceptance criteria. The other surrogate compound (1,2-dichloroethane-d_4) recovered within the method QC acceptance criteria. The following table lists the surrogate spike recovery and the QC acceptance criteria:

Sample No.	1,2-Dichlorobenzene-d_4 % Recovery	QC Acceptance Criteria (drinking water)
SA925	45	80-120

The sample was reanalyzed 22 days past the holding time. 1,2-Dichlorobenzene-d_4 recovered at 52% in the reanalysis. The validator reports SA925 sample results from the initial analysis because the reanalysis results may be biased low due to the exceeded holding time. The validator reviews the MS/MSD results for sample SA928 and determines that there is no indication of matrix bias in this data set. The validator estimates (J) positive detects and estimates (UJ) non-detects in sample SA925 on the Data Summary Table and notes in the Data Validation Memorandum that the low recovery may be due to matrix interferences specific to SA925.

Example #3: (One slightly low acid and one slightly low base/neutral surrogate recovery)

Semivolatile soil sample SA225, analyzed by CLP SOW OLM03.2, had one acid surrogate compound, 2,4,6-tribromophenol, and one base/neutral surrogate compound, 2-fluorobiphenyl, recover below the method QC acceptance criteria but above 10%. The following table lists the surrogate spike recoveries and the method QC acceptance criteria:

Sample No.	2,4,6-Tribromophenol % Recovery	QC Acceptance Criteria (soil/sediment)	2-Fluorobiphenyl % Recovery	QC Acceptance Criteria (soil/sediment)
SA225	16	19-122	22	30-115

Reanalysis was not contractually required because only one acid surrogate and only one base/neutral surrogate exceeded method QC acceptance criteria. The validator reviews the MS/MSD results for sample SA228 and determines that there is no indication of matrix bias in this data set. The validator examines all surrogate recoveries, including the advisory surrogates in the sample, and determines that validation criteria were met. The validator reports the sample results unqualified on the Data Summary Table.

E. EXAMPLES

Example #4: (Two slightly low acid surrogate recoveries)

Semivolatile soil sample SA882, analyzed by CLP SOW OLM03.2, had two acid surrogate compounds, phenol-d_5 and 2-fluorophenol, recover below the method QC acceptance criteria. All other surrogate recoveries met method QC acceptance criteria. The following table lists the surrogate spike recoveries and the method QC acceptance criteria:

Sample No.	Phenol-d_5 % Recovery	QC Acceptance Criteria (soil/sediment)	2-Fluorophenol % Recovery	QC Acceptance Criteria (soil/sediment)
SA882	20	24-113	18	25-121

The sample was reextracted and reanalyzed with similar results. The validator reviews the MS/MSD results for sample SA880 and determines that there is no indication of matrix bias in this data set. The validator estimates (J) positive detects and estimates (UJ) non-detects for the acid fraction of sample SA882 on the Data Summary Table and notes in the Data Validation Memorandum that the low recovery may be due to matrix interferences specific to sample SA882.

Example #5: (One advisory base/neutral surrogate with 0% recovery)

1,2-dichlorobenzene is a contaminant of concern at Site XX. Semivolatile water sample SA335, analyzed by CLP SOW OLM03.2, had advisory surrogate compound, 1,2-dichlorobenzene-d_4, recover at 0%. All of the remaining surrogate compounds and advisory surrogate compounds had recoveries which were within method QC acceptance criteria. The validator reviews the MS/MSD results for sample SA336 and determines that there is no indication of matrix bias in this data set. The validator uses professional judgment to reject (R) the analyte of concern, 1,2-dichlorobenzene, and to reject (R) the other dichlorobenzene isomers in sample SA335, based upon their chemical similarity to the advisory surrogate. The validator reports the qualified results on the Data Summary Table and notes in the Data Validation Memorandum that the low recovery may be due to matrix interferences specific to sample SA335 or poor laboratory technique during the sample extraction and/or cleanup procedures.

E. EXAMPLES

Example #6: (One high volatile surrogate recovery)

Volatile soil sample SA966, analyzed using SW-846 Method 8260, recovered one surrogate above the method QC acceptance criteria. The following table lists the surrogate percent recoveries and method QC acceptance criteria:

Sample Number	Toluene-d_8 % Recovery	QC Acceptance Criteria
SA966	128	81 - 117

The sample was reanalyzed within holding time with similar results. The validator reviews the MS/MSD results for sample SA960 and determines that there is no indication of matrix bias in this data set. The validator estimates (J) positive detects and accepts (A) non-detects in the associated sample. The validator reports qualified data on the Data Summary Table and notes sample qualifications in the Data Validation Memorandum.

VII. INTERNAL STANDARDS

A. OBJECTIVE

Instrument performance and stability and laboratory precision throughout an analytical sequence are monitored by the addition of internal standard compounds. Internal standards (ISs) are added to every field sample, QC sample, standard and blank just prior to analysis. Evaluation of the behavior of internal standards is not necessarily straightforward. Interfering sample matrix effects, including high concentrations of target and non-target analytes, are frequently outside of the laboratory's control and may adversely affect the analysis of internal standards.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. The internal standard compounds specified in the method must be added to all samples, QC samples, standards and blanks at the required concentrations.

2. Internal standard area counts must be within the method QC acceptance criteria.

3. The retention time of the internal standard must be within the method QC acceptance criteria.

4. Samples must be reanalyzed and/or reextracted in accordance with method requirements if internal standard method QC acceptance criteria are not met.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from internal standard anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct compounds were added to all samples, QC samples, standards and blanks at the method-specified concentrations.	1. If the laboratory did not add the required internal standard compounds to all samples, QC samples, standards and blanks at the correct concentration, then the validator must use professional judgment to determine how the associated sample data should be qualified or rejected.
2. Verify that all IS area counts are within the method QC acceptance criteria.	2. If an IS area count for a sample, QC sample, or blank is outside the method QC acceptance criteria, then the validator should: a. Estimate (J) positive detects for compounds quantitated using an IS area count greater than the upper limit of the method QC acceptance criteria. b. Accept (A) non-detects for compounds quantitated using an IS area count greater than the upper limit of the method QC acceptance criteria. c. Estimate (J) positive detects for compounds quantitated using an IS area count less than the lower limit of the method QC acceptance criteria. d. Estimate (UJ) non-detects for compounds quantitated using an IS area count less than the lower limit of the method QC acceptance criteria but greater than or equal to 20% of the associated daily continuing calibration standard area. e. Reject (R) non-detects for compounds quantitated using an IS area count less than 20% of the associated daily continuing calibration standard area or if internal standard performance exhibits a major abrupt drop-off, indicating a severe loss of sensitivity. Alternatively, professional judgment may be used to assess signal to noise ratios to qualify or reject sample data.

C. EVALUATION	D. ACTION
3. Verify that all IS retention times are within method QC acceptance criteria.	3. If an IS retention time for a sample, QC sample, or blank is outside the method QC acceptance criteria, then the validator should examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the validator may consider partial or total rejection of the data for that sample fraction. The validator should use professional judgment to determine if positive detects can be reported based upon mass spectral identification criteria being met. The validator should consider, however, the possible presence of non-target compounds that are isomers of target compounds.
*4. Check raw data (e.g., chromatograms and quantitation reports) to verify that the internal standard retention times and areas are accurately reported on the tabulated forms.	4. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
5. a. Verify that if any internal standard compound area count or retention time is outside the method QC acceptance criteria, that the required reanalysis was performed to confirm that the non-compliance was due to sample matrix effects rather than poor laboratory performance.	5. a. If a laboratory fails to reanalyze a sample with an internal standard compound that is outside the method QC acceptance criteria, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this problem in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
5. b. If there are two analyses for a particular fraction, then the validator must determine which are the best data to report. Considerations should include but are not limited to: - Magnitude and direction of the IS area shift; - Magnitude and direction of the IS retention time shift; - Technical holding times; - Comparison of the values of the target compounds reported in each analysis; - Other relevant QC.	5. b. If a sample has been analyzed and reported more than once, then the validator should use professional judgment when considering which analysis or portion of an analysis to report. The validator must consider all relevant QC information in making a decision.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.4

Table VOA/SV-VII-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON INTERNAL STANDARD AREA COUNTS

Internal Standard Area Counts				
Sample Results	**Area Counts < 20% of associated calibration std. area**	**20% ≤ Area Counts < LL**	**LL ≤ Area Counts ≤ UL**	**Area Counts > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria based on associated calibration standard area
UL - Upper Limit of method QC acceptance criteria based on associated calibration standard area

E. EXAMPLES

Example #1: (Sequential instrument sensitivity loss for one volatile IS compound ending with sample IS area < LL of method QC acceptance criteria based on associated daily continuing calibration standard area)

IS = 1,4-difluorobenzene

12 Hour STD 27105
Upper Limit (+ 100%) 54210
Lower Limit (50%) 13553

	Sample AAA01	Sample AAA02	Sample AAA03	Sample AAA04
IS Area Count:	30000	22000	15000	10000
benzene concentration (ug/kg)	24	32	38	45

The validator reviews the IS area counts for samples analyzed by CLP SOW OLM03.2 and notes that the 1,4-difluorobenzene area counts decrease sequentially over time and the area counts for sample AAA04 are below the lower method QC acceptance limit but greater than 20% of the associated daily continuing calibration standard area. Upon review of the sample data, the validator ascertains that benzene was the only target compound detected in the samples. Therefore, the validator estimates (J) the benzene positive detects in sample AAA04 and estimates (UJ) quantitation limits for all other target analytes quantitated using 1,4-difluorobenzene in sample AAA04 on the Data Summary Table. The validator discusses the instrument's sensitivity loss and the sample qualifications in the Data Validation Memorandum.

Example #2: (One semivolatile IS compound with area counts < 20% of associated daily continuing calibration standard)

IS = 1,4-dichlorobenzene-d_4

12 Hour STD 76400
Upper Limit (+ 100%) 152800
Lower Limit (50%) 38200

	Sample AAA01	Sample AAA02	Sample AAA03	Sample AAA04
IS Area Count:	75000	73000	10000	76000
phenol concentration (ug/L)	35	10U	17	55

The validator reviews the IS area counts for samples analyzed by CLP SOW OLM03.2 and notes that the 1,4-dichlorobenzene-d_4 area count in sample AAA03 is less than 20% of the associated daily continuing calibration standard area (20% = 15280). Upon review of the sample data, the validator ascertains that phenol was the only target compound detected in the samples. Therefore, the validator estimates (J) the positive phenol detect in sample AAA03 and rejects (R) the quantitation limits for all other target analytes quantitated using 1,4-dichlorobenzene-d_4 in sample AAA03 on the Data Summary Table. The validator notes the sample qualifications in the Data Validation Memorandum.

E. EXAMPLES

Example #3: (One semivolatile IS compound with RT shift greater than method QC acceptance limit)

The validator reviews the IS data and determines that the retention time for chlorobenzene-d_5 has shifted by + 60 seconds which exceeds the ± 30 second QC acceptance limit allowable under CLP SOW OLM03.2. Upon inspection of the chromatographic profile, the validator determines that the mass spectral identification criteria have been met for positive detects associated with chlorobenzene-d_5. The validator accepts the positive detects associated with chlorobenzene-d_5 and rejects (R) the quantitation limits for all other target analytes quantitated using chlorobenzene-d_5 on the Data Summary Table. The validator discusses the possibility of false negatives in this sample in the Data Validation Memorandum.

VIII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. OBJECTIVE

Data for matrix spike/matrix spike duplicates (MS/MSDs) are generated to determine laboratory precision and method bias for specific sample matrices at the time of sample preparation and analysis. MS/MSD data can be used to determine long-term interlaboratory precision and bias of an analytical method for various matrices and are used in setting quality control acceptance criteria for spiking compounds. MS/MSD data should be used in conjunction with other QC data, such as field duplicate data and surrogate compound recoveries, to determine if a sample or an entire sample group should be qualified.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. In accordance with the SAP, QAPjP and/or method, a field sample of each matrix is spiked in duplicate with known concentrations of specific target compounds to generate an MS/MSD pair. Concurrently, the laboratory analyzes an unspiked aliquot and the MS/MSD pair of the field sample.

2. a. Field samples (not trip, equipment, or bottle blanks and not PE samples) must be spiked to assess matrix effects.

   b. Field samples chosen for MS/MSD analysis should not contain high levels of MS/MSD spiking compounds prior to spiking. Preferably, field samples chosen for MS/MSD analysis should contain low levels of the spiking compounds.

3. Spike recoveries must be within the QC acceptance criteria specified in the method, SAP or QAPjP.

4. Relative percent differences (RPDs) between MS and MSD recoveries must be within the QC acceptance criteria specified in the method.

5. The percent relative standard deviation (%RSD) between positively detected non-spike compounds in the unspiked sample, MS, and MSD must be less than or equal to 50%.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from matrix spike/matrix spike duplicate anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct compounds were added at the required concentrations; that MS/MSD samples were analyzed at the proper frequency; and that MS/MSD results are provided for each sample matrix.	1. If the laboratory did not use the required compounds at the concentration and frequency specified in the method for each sample matrix, then the validator must use professional judgment to determine whether the associated sample data should be qualified.
2. a. Verify that a field sample was chosen for the MS/MSD.	2. a. If a trip, equipment or bottle blank or a PE sample was used for the MS/MSD, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing laboratory precision, method bias, sample matrix effects and ultimately data usability.
b. Determine if an inappropriate sample containing high levels of the spiking compounds was chosen for the MS/MSD pair.	b. If the MS/MSD compounds were present in the field sample at high concentrations (e.g., 4x spike concentration) before spiking, then the validator must use professional judgment in assessing matrix spike recoveries and RPDs.
c. Ascertain if the MS/MSD analyses required dilutions.	c. If no MS/MSD data can be reported because of sample dilution, then the validator should note this problem in the Data Validation Memorandum and discuss the impact on assessing data usability in the case where laboratory precision and method bias information are absent.

C. EVALUATION	D. ACTION
3. Verify that all spike recoveries are within the QC acceptance criteria specified in the method.	3. a. If any spike recovery result is greater than the upper limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) the positive detect for that affected compound in the unspiked sample. ii. Accept the non-detect for that affected compound in the unspiked sample. b. If any recovery result is greater than or equal to 10%, but less than the lower limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) the positive detect for that affected compound in the unspiked sample. ii. Estimate (UJ) the non-detect for that affected compound in the unspiked sample. c. If any recovery result is less than 10%, then the validator should: i. Estimate (J) the positive detect for that affected compound in the unspiked sample. ii. Reject (R) the non-detect for that affected compound in the unspiked sample. d. If the majority of spike compound recoveries are outside the method QC acceptance criteria, then the validator may use professional judgment to estimate (J) or reject (R) <u>all</u> positive detects and estimate (UJ) or reject (R) <u>all</u> non-detects in the unspiked sample.

C. EVALUATION	D. ACTION
4. Verify that all the RPDs between the MS and MSD are within the QC acceptance criteria specified in the method.	4. If any RPD result is outside the method QC acceptance criteria, then the validator should: a. Estimate (J) the positive detect for that affected compound in the unspiked sample. b. Estimate (UJ) the non-detect for that affected compound in the unspiked sample. c. If the majority of the matrix spike RPDs are outside method QC acceptance criteria, then the validator should use professional judgment to estimate (J) all positive detects and estimate (UJ) or reject (R) all non-detects in the unspiked sample. Refer to Section VIII C. 8 and 9 for additional guidance.

C. EVALUATION	D. ACTION
5. a. Calculate the % RSD for the non-spiked target positive detects in the unspiked sample, the MS and the MSD.	5. a. If a non-detected result or a detect less than the quantitation limit is reported for a non-spiked target compound in one of the samples in the MS, MSD or unspiked sample set, then the validator should use the sample quantitation limit value for that compound to calculate the %RSD. If a non-detected result or a detect less than the quantitation limit is reported for a non-spiked target compound in two of the samples in the MS, MSD or unspiked sample set, then the validator should not calculate the %RSD but should use professional judgment to qualify sample data.
b. The unspiked sample, MS, and MSD may be considered a triplicate in determining the overall precision of the analytical method. Therefore, evaluate the %RSD data for positive detects in the triplicate set.	b. If any %RSD is greater than 50%, then the validator should: i. Estimate (J) the positive detect for that affected compound in the unspiked sample. ii. Use professional judgment to qualify or accept the non-detect for that affected compound in the unspiked sample. If overall laboratory precision for the unspiked field sample, MS, and MSD is poor, then the validator may use professional judgment to qualify <u>all</u> positive detects and non-detects in the unspiked sample. The Data Validation Memorandum should include a discussion of the potential impact of laboratory precision on representativeness and usability of the data in meeting the project DQOs.

C. EVALUATION	D. ACTION
*6. Check and recalculate the analytical concentrations and percent recovery for at least one spiked compound per MS/MSD fraction. Verify that the recalculated value agrees within ± 10% of the reported value.	6. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*7. Check and recalculate the RPD for at least one spiked compound per MS/MSD fraction. Verify that the recalculated value agrees within ± 10% of the reported value.	7. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
8. Evaluate the appropriateness of qualifying the entire data set based on MS/MSD laboratory precision and method/matrix bias results.	8. Generally, no action is taken based on the MS/MSD data alone to qualify an entire case. The qualification is limited to the unspiked sample associated with the MS/MSD. However, professional judgment may be used to qualify sample results across a particular aqueous matrix (i.e., all associated groundwater samples) or a homogeneous soil matrix.

C. EVALUATION	D. ACTION
9. Evaluate MS/MSD precision data to confirm the laboratory's ability to generate precise data and field duplicate precision data to assess overall precision. Surrogate recovery data can also be evaluated to identify laboratory precision issues and overall matrix precision issues.	9. If precision data for the laboratory MS/MSD pair, surrogate compound recoveries and field duplicate pair indicate a heterogenous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify all affected compounds and/or all field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting the project DQOs should be discussed. Refer to Section IX for additional guidance.

* **Note:** **The following subsections are applicable only to a Tier III data validation:**

C.6, C.7

Table VOA/SV-VIII-1:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MATRIX SPIKE RECOVERIES AND RPDs**

Sample Results	Recovery < 10%	10% ≤ Recovery < Lower QC Limit	Lower QC Limit ≤ Recovery ≤ Upper QC Limit	Recovery > Upper QC Limit	RPD > QC Limit
Detects	J	J	A	J	J
Non-detects	R	UJ	A	A	UJ

** Note that qualification and rejection generally are limited to the spiking compounds, however, the validator may use professional judgment to qualify or reject all positive detects or non-detects in the unspiked sample if the majority of spike compound recoveries and/or RPDs are outside the method QC acceptance criteria.

Table VOA/SV-VIII-2:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MS, MSD, AND UNSPIKED SAMPLE %RSD

Sample Results	%RSD ≤ 50%*	%RSD > 50%*	Two out of three sample results reported as non-detects
Detects	A	J	Professional Judgment
Non-detects	A	Professional Judgment	Professional Judgment

* If a non-detect is reported for a compound in only one of the samples in the MS, MSD or unspiked sample set, then the validator should use the sample quantitation limit value for that compound to calculate the %RSD.

E. EXAMPLES

Example #1: (High MS/MSD RPD for one compound)

Soil QC samples SAA99MS and SAA99MSD, analyzed as medium level soil samples under CLP SOW OLM03.2, have unacceptable RPD results for acenaphthene. Acenaphthene was detected in the unspiked sample, SAA99.

Sample No.	Compound	MS/MSD %Rec	MS/MSD %Rec Criteria	MS/MSD RPD	MS/MSD RPD Criteria
SAA99MS SAA99MSD	Acenaphthene	60/116	31-137	64	19

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator then concludes that the lack of laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) the positive detect for acenaphthene in the unspiked sample, SAA99, on the Data Summary Table. The validator discusses the lack of laboratory precision for one compound, acenaphthene, in the Data Validation Memorandum and notes that laboratory precision for the other semivolatile matrix spike compounds was acceptable.

E. EXAMPLES

Example #2: (Low MS/MSD recoveries for one compound)

Aqueous QC samples SAA22MS and SAA22MSD, analyzed under CLP SOW OLM03.2, have low toluene recovery results but acceptable RPD results. Toluene was detected in the unspiked sample, SAA22. Surrogate compound recoveries were acceptable for SAA22MS, SAA22MSD and the unspiked sample, SAA22.

Sample No.	Compound	MS/MSD %Rec	MS/MSD %Rec Criteria	MS/MSD RPD	MS/MSD RPD Criteria
SAA22MS SAA22MSD	Toluene	50/46	76-125	8	13

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 30%, indicating acceptable overall precision for this sampling event. The validator concludes that the sample matrix causes a reproducible negative bias for toluene in aqueous samples SAA22MS and SAA22MSD. The validator estimates (J) the positive detect for toluene in the unspiked sample, SAA22, on the Data Summary Table. The validator discusses the low matrix spike recoveries in the Data Validation Memorandum and notes that recoveries for the other volatile matrix spike compounds were acceptable.

Example #3: (High %RSD; High RPD, poor laboratory precision)

Soil samples SAA55, SAA55MS and SAA55MSD analyzed under CLP SOW OLM03.2, had high RPDs for two of the acid semivolatile matrix spike compounds in the MS/MSD, 2-chlorophenol (53%) and 4-nitrophenol (92%) and two base/neutral semivolatile spike compounds, 1,2,4-trichlorobenzene (65%) and acenaphthene (76%). 2-chlorophenol, 4-nitrophenol, 1,2,4-trichlorobenzene, and acenaphthene were not detected in the unspiked sample. The other remaining matrix spike compound RPDs were acceptable. The following non-spike target compound results were obtained for SAA55MS/MSD and the unspiked sample SAA55.

Sample No.	Compound	MS Conc. Dry Weight (ug/kg)	MSD Conc. Dry Weight (ug/kg)	Unspiked Sample Conc. Dry Weight (ug/kg)	% RSD
SAA55	2,4-Dimethylphenol	1200	350	600	61
SAA55	2,4,6-Trichlorophenol	380	1030	330U	67
SAA55	Hexachlorobenzene	920	330U	400	59

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator then concludes that the lack of precision is due to poor laboratory technique. The validator uses professional judgment to estimate (J) the positive detects for 2,4-dimethylphenol and hexachlorobenzene in SAA55 and estimate (UJ) all non-detects in sample SAA55 on the Data Summary Table. The validator discusses the poor laboratory precision and notes the sample qualifications in the Data Validation Memorandum.

E. EXAMPLES

Example #4: (Low MS/MSD recoveries for entire compound class)

Soil QC samples SAA01MS and SAA01MSD, analyzed under CLP SOW OLM03.2, have low spike recoveries for four of the five acid compounds in the matrix spike and the matrix spike duplicate (less than the specified QC acceptance criteria but greater than 10%); while base neutral matrix spike compounds meet QC acceptance criteria. The phenol-d_5 and 2-fluorophenol acid surrogate recoveries are at the low end of the QC acceptance criteria in SAA01MS and SAA01MSD.

Sample No.	Compound	MS % Recovery	MSD % Recovery	RPD	QC Acceptance Criteria	
					% Rec	RPD
SAA01MS/MSD	Phenol	21	21	0	26-90	35
	2-Chlorophenol	15	18	18	25-102	50
	4-Chloro-3-methylphenol	21	20	5	26-103	33
	Pentachlorophenol	14	14	0	17-109	47
	Phenol-d5 (surrogate)	26	28	NA	24-113	NA
	2-Fluorophenol (surrogate)	27	25	NA	25-121	NA

Upon review of the MS/MSD results and surrogate recoveries, the validator notes that the sample matrix causes a reproducible negative bias for acid compounds in soil QC samples SAA01MS and SAA01MSD. The validator reviews the surrogate recoveries for the unspiked sample and notes that the acid surrogate recoveries are within the QC acceptance criteria (at the low end of the QC acceptance range). The validator then reviews the surrogate recoveries for all samples associated with the sample delivery group to ascertain if acid surrogate recoveries are also low in the remaining samples.

Several samples, including the field duplicates, show low acid surrogate recoveries that were greater than 10%. The validator estimates (J) all positive acid detects in the unspiked MS/MSD sample and estimates (UJ) all acid non-detects in the unspiked MS/MSD sample. The validator uses professional judgment to estimate (J) the positive acid detects and estimate (UJ) the acid non-detects in all other samples associated with this sample delivery group in which acid surrogates recovered low. The validator reports qualified data on the Data Summary Table and discusses the low bias in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (High MS/MSD RPDs for multiple compounds)

Aqueous QC samples SAA08MS and SAA08MSD, analyzed under CLP SOW OLM03.2, have high RPDs for 2 acid and 3 base neutral compounds in the matrix spike/matrix spike duplicate pair. The matrix spike recoveries in the MS and MSD were all within QC acceptance criteria. All surrogate recoveries for SAA08MSD were acceptable except for the advisory surrogate, 2-chlorophenol-d_4. All surrogate recoveries for SAA08MS were acceptable except for nitrobenzene-d_5 and terphenyl-d_{14}. Hexachlorobenzene and dibenzofuran were the only positive detects in the unspiked sample, SAA08. The validator calculates the %RSD for hexachlorobenzene (59%) and dibenzofuran (70%).

Sample No.	Compound	MS % Recovery	MSD % Recovery	RPD	QC Acceptance Criteria	
					% Rec	RPD
SAA08MS/MSD	N-Nitroso-di-n-propylamine	43	80	60	41-116	38
	1,2,4-Trichlorobenzene	93	48	64	39-98	28
	2,4-Dinitrotoluene	87	33	90	24-96	38
	Pentachlorophenol	15	78	135	9-103	50
	2-Chlorophenol	96	40	82	27-123	40
	Nitrobenzene-d_5 (surrogate)	25	65	NA	35-114	NA
	2-Chlorophenol-d_4 (surrogate)	70	30	NA	33-110	NA
	Terphenyl-d_{14} (surrogate)	30	83	NA	33-141	NA

Sample No.	Compound	MS Conc. (ug/L)	MSD Conc. (ug/L)	Unspiked Sample Conc. (ug/L)	% RSD
SAA08	Hexachlorobenzene	20	80	85	59
	Dibenzofuran	57	22	110	70

Upon review of the MS/MSD results, surrogate recoveries, and the % RSDs, the validator notes the laboratory imprecision and suspects that problems occurred during extraction and/or analysis of the MS/MSD and/or unspiked sample. The validator then reviews the field duplicate data and surrogate recoveries for the remaining samples in the sample delivery group to assess other precision and bias data.

Surrogate recoveries in all other samples were acceptable. The field duplicate RPD data was also acceptable. Therefore, the validator determines that poor precision was limited to the MS/MSD pair. The validator uses professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects in the unspiked sample SAA08 on the Data Summary Table. The validator notes this problem in the Data Validation Memorandum.

IX. FIELD DUPLICATES

A. OBJECTIVE

Field duplicates measure the cumulative effects of both field and laboratory precision and hence provide an indication of overall precision. Therefore, field duplicates may have greater variability than laboratory duplicates which measure only laboratory precision. It is also expected that non-aqueous matrices will have a greater variance than aqueous matrices due to the heterogeneity of most non-aqueous samples (such as soil/sediment samples).

B. CRITERIA

1. The frequency of field duplicate analysis must support the site-specific Data Quality Objectives (DQOs) and be documented in the EPA approved QAPjP or SAP.

2. a. The RPD for all compounds detected at concentrations greater than the sample quantitation limit in aqueous matrices must be less than or equal to 30 percent.

   b. The RPD for all compounds detected at concentrations greater than the sample quantitation limit in non-aqueous matrices must be less than or equal to 50 percent.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from field duplicate anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Identify which samples are field duplicates from the Chain-of-Custody form and/or the Traffic Report.	1. a. If field duplicates are not listed on the Chain-of-Custody form or the Traffic Report, then the validator should contact the sampler to ascertain if field duplicates were collected. If the forms were completed incorrectly or if field duplicates were not collected, then the validator should document this on the Data Validation Worksheet and in the Data Validation Memorandum.
b. Verify that the appropriate number of field duplicates per matrix sampled were collected and analyzed to support project DQOs.	b. If field duplicates were not collected at the required frequency to support project DQOs, then the validator should note the absence of field precision data in the Data Validation Memorandum and discuss how the lack of field precision data might potentially increase uncertainty surrounding site decisions.

C. EVALUATION	D. ACTION
2. Calculate the RPD for all compounds detected at concentrations greater than or equal to the sample quantitation limit in the field duplicate sets.	2. a. If any compound is detected at concentrations greater than or equal to twice the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should estimate (J) the positive detects for that compound in both samples. If any compound is detected at concentrations greater than or equal to the sample quantitation limit but less than twice the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should use professional judgment to accept, qualify, or reject the positive detects for that compound in the field duplicate samples taking into consideration the increased variability of data near the sample quantitation limit and the site-specific DQOs. b. If any compound is detected at concentrations greater than or equal to twice the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should estimate (J) the positive detects for that compound in both samples. If any compound is detected at concentrations greater than or equal to the sample quantitation limit but less than twice the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should use professional judgment to accept, qualify, or reject the positive detects for that compound in the field duplicate samples taking into consideration the increased variability of data near the sample quantitation limit and the site-specific DQOs.

C. EVALUATION	D. ACTION
2. Continued from above.	2. c. If any compound in a field duplicate pair has one positive detect that is greater than or equal to twice the sample quantitation limit and a duplicate positive detect that is less than twice the sample quantitation limit, and the RPD exceeds field duplicate precision criteria for that matrix, then the validator should use professional judgment to qualify the positive detects for that compound in the field duplicate samples. d. If any compound in a field duplicate pair has one non-detect and a duplicate positive detect that is greater than or equal to twice the sample quantitation limit, then the validator should estimate (J) the positive detect and (UJ) the non-detect for that compound in the field duplicate samples. (RPDs should not be evaluated for those duplicate pairs.) e. If any compound in a field duplicate pair has one non-detect or a reported value below the sample quantitation limit and a duplicate positive detect that is less than twice the sample quantitation limit, then the validator should use professional judgment to qualify the positive detects and non-detects for that compound in the field duplicate samples taking into consideration the increased variability of data at the sample quantitation limit and the project DQOs. (RPDs should not be evaluated for those duplicate pairs.) f. If any compound in a field duplicate pair has one non-detect or a reported value below the sample quantitation limit and a duplicate positive detect that is less than the sample quantitation limit, then the validator should use professional judgment to qualify the positive detects and non-detects for that compound in the field duplicate sample pair taking into consideration the increased variability of data at the sample quantitation limit and the project DQOs (RPDs should not be evaluated for those duplicate pairs).

C. EVALUATION	D. ACTION
*3. Check and recalculate the analytical concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or soil sample) for each fraction, in every field duplicate sample, in accordance with Section VOA/SV-XIII, C.1 - C.3.	3. If calculation and/or transcription errors are detected, then the validator should follow the procedures outlined in Section VOA/SV XIII, D.1 - D.3.
4. Evaluate the appropriateness of qualifying the entire data set based on field duplicate results.	4. If field duplicate data indicate poor field precision and general sample heterogeneity and/or possible sampling error, then professional judgment may be used to qualify data for all samples of the same matrix.
5. Evaluate field duplicate precision data to assess overall precision and to verify the field sampler's ability to collect representative duplicate samples. MS/MSD precision data should be evaluated to verify the laboratory's ability to generate precise data. Surrogate recovery data can also be evaluated to identify laboratory precision issues and overall matrix precision issues.	5. If precision data for the field duplicate pair, surrogate compound recoveries and laboratory MS/MSD pair indicate a heterogeneous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify all affected compounds and/or all affected field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting project DQOs should be discussed. Refer to Section VIII for additional guidance.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.3

Table VOA/SV-IX-1:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES - SITUATION 1: POSITIVE DETECTS IN BOTH FIELD DUPLICATES

Relative Percent Difference	**Aqueous > 30% Non-Aqueous > 50%**	**Aqueous > 30% Non-Aqueous > 50%**	**Aqueous > 30 Non-Aqueous > 50%**
Sample Results	**Both duplicate sample concs. ≥ 2 X QL**	**QL ≤ both duplicate samples concs. < 2 X QL**	**One sample conc. ≥ 2 X QL QL ≤ Other sample conc. < 2 X QL**
Detects	J	Professional Judgment	Professional Judgment
Non-detects	NA	NA	NA

* QL = Sample Quantitation Limit

Note: Qualification refers to field duplicate sample results only. Professional judgment may be utilized to apply field duplicate actions to all samples of the same matrix.

Table VOA/SV-IX-2:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES - SITUATION 2: POSITIVE DETECT IN ONLY ONE FIELD DUPLICATE**

Aqueous and Non-Aqueous		
Sample Results	**One Sample Conc. = ND (or value reported as less than the QL) QL ≤ Other Sample Conc. < 2 X QL**	**One sample conc. = ND (or value reported as less than the QL) Other sample conc. ≥ 2 X QL**
Detects	Professional Judgment	J
Non-detects	Professional Judgment	UJ

* QL = Sample Quantitation Limit

** RPD should not be evaluated for these duplicate pairs

Note: Qualification refers to field duplicate sample results only. Professional judgment may be utilized to apply field duplicate actions to all samples of the same matrix.

E. EXAMPLES

Example #1: (Both field duplicate sample concentrations ≥ 2X QL; RPD > 50%; Acceptable laboratory precision)

Soil samples SAA11 and SAA12 are field duplicates, analyzed under CLP SOW OLM03.2, and they contain 89% and 85% solids, respectively. Sample SAA11 has a detected concentration of benzene of 100 ug/kg. Sample SAA12 has a detected concentration of benzene of 250 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 86%. The validator notes that both results are greater than twice the sample Quantitation Limit (QL). The QL for benzene in sample SAA11 is 11 ug/kg and for sample SAA12 is 12 ug/kg. The validator reviews the MS/MSD data and determines that laboratory precision was acceptable. As a result, the validator estimates (J) the positive benzene detects in the field duplicate samples only, on the Data Summary Table, and notes the qualification and justification in the Data Validation Memorandum. The validator also notes that poor field precision may be due to a heterogenous matrix or a result of sampling error.

Compound	SAA11		SAA12		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
benzene	100	11	250	12	86

E. EXAMPLES

Example #2: (QL ≤ both field duplicate sample concentrations < 2X QL; RPD > 50%; Acceptable laboratory precision)

Soil samples SAA21 and SAA22 are field duplicates, analyzed under CLP SOW OLM03.2, and they contain 50% and 52% solids, respectively. Sample SAA21 has a detected concentration of trichlorophenol of 690 ug/kg. Sample SAA22 has a detected concentration of trichlorophenol of 1220 ug/kg. The validator determines that the RPD equals 56%. The sample QL for trichlorophenol in sample SAA21 is 660 ug/kg based on 50% solids and the sample QL for sample SAA22 is 630 ug/kg based on 52% solids. The validator reviews the MS/MSD results and determines that laboratory precision is acceptable. The validator notes that both field duplicate results are between the sample QL and twice the sample QL. As a result, the validator uses professional judgment to accept the trichlorophenol results in the field duplicate samples taking into consideration the increased variability of data near the quantitation limit. The validator notes in the Data Validation Memorandum that field duplicate precision was acceptable.

Compound	SAA21		SAA22		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
trichlorophenol	690	660	1220	630	56

Example #3: (One sample concentration = ND; One sample concentration ≥ 2X QL; Acceptable laboratory precision)

Aqueous samples SAA31 and SAA32 are field duplicates, analyzed under CLP SOW OLM03.2. Sample SAA31 has a detected concentration of trichloroethene of 25 ug/L. Trichloroethene was not detected in sample SAA32. The validator notes that the positive trichloroethene detect in sample SAA31 is greater than twice the sample QL (10 ug/L). The validator reviews the MS/MSD data and determines that laboratory precision was acceptable. The validator estimates (J) the positive trichloroethene detect in sample SAA31 and estimates (UJ) the quantitation limit of the trichloroethene non-detect in sample SAA32 on the Data Summary Table based on poor field precision. The validator notes the qualification in the Data Validation Memorandum and also suggests that poor field precision may be due to sampling error.

Compound	SAA31		SAA32		RPD
	Sample Conc. (ug/L)	Sample QL (ug/L)	Sample Conc. (ug/L)	Sample QL (ug/L)	
trichloroethene	25	10	ND	10	NA

E. EXAMPLES

Example #4: (One sample concentration = ND; One sample concentration < 2X QL; Acceptable laboratory precision)

Soil samples SAA41 and SAA42 are field duplicates, analyzed under CLP SOW OLM03.2, and they contained 90% and 85% solids, respectively. Sample SAA41 has a detected concentration of chlorobenzene of 19 ug/kg. Chlorobenzene was not detected in sample SAA42. The validator notes that the positive chlorobenzene detect is between the sample QL and twice the sample QL. The sample QL for chlorobenzene in sample SAA41 is 11 ug/kg and in sample SAA42 is 12 ug/kg. The validator reviews the MS/MSD results and determines that RPD criteria were met for chlorobenzene, indicating acceptable laboratory precision. As a result, the validator uses professional judgment to accept the positive chlorobenzene detect in sample SAA41 and to accept the chlorobenzene non-detect in sample SAA42, taking into consideration the increased variability of data near the quantitation limit. The validator reports the results on the Data Summary Table and notes this in the Data Validation Memorandum.

Compound	SAA41		SAA42		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
chlorobenzene	19	11	ND	12	NA

Example #5: (Both duplicate concentrations ≥ 2X QL; Poor field and laboratory precision)

Soil samples SAA34 and SAA35 are field duplicates, analyzed under CLP SOW OLM03.2, and they contain 90% and 95% solids, respectively. Sample SAA34 has a detected concentration of pyrene of 1400 ug/kg. Sample SAA35 has a detected concentration of pyrene of 3500 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 86%. The validator notes that both results are greater than twice the sample QL. The sample QL for pyrene in sample SAA34 is 370 ug/kg and the sample QL for pyrene in sample SAA35 is 350 ug/kg. The validator reviews the MS/MSD data for samples SAA34 MS/MSD and determines that the RPD for pyrene equals 61%. The validator is unable to determine the source of the imprecision since both the lab and field precision were poor; therefore, the validator uses professional judgment to estimate (J) the positive pyrene detects in all samples associated with the sample delivery group and estimates (UJ) the quantitation limits for pyrene non-detects in all samples associated with the sample delivery group. The validator reports the qualified data on the Data Summary Table and justifies the qualification in the Data Validation Memorandum. The validator notes that the source of the imprecision cannot be determined.

Compound	SAA34		SAA35		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
pyrene	1400	370	3500	350	86

X. SENSITIVITY CHECK

A. OBJECTIVE

Although most CLP SOWs do not incorporate the analysis of sensitivity checks, many EPA methods do require that a Method Detection Limit (MDL) study be performed prior to sample analysis and/or that a Laboratory Fortified Blank (LFB) be analyzed at the time of sample analysis. The MDL study generates statistically-based detection limits and can be used to assess method sensitivity, laboratory precision and method bias for specific compounds within an analytical method on a specific instrument and column. An LFB, a type of Laboratory Control Sample, is a reagent blank spiked with several or all of the target compounds at or below their quantitation limits. LFB data can be used to assess laboratory sensitivity and bias for specific compounds at the quantitation limit within an analytical method on a specific instrument and column at the time of sample preparation and analysis. To determine sample qualification, the MDL study is evaluated prior to the LFB data.

Region I routinely uses MDL studies as a pre-qualification check to verify the laboratory's ability to meet the technical specification/method requirements prior to contract award and field sample receipt. Region I also routinely includes LFB analyses to document the method sensitivity and bias associated with the day-to-day preparation and analysis of field samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. Method Detection Limit (MDL) Study

a. The method detection limit (MDL) for each compound of interest must be established in accordance with the specified method and the Code of Federal Regulations (40 CFR Part 136, App. B). A minimum of seven replicates must be analyzed for each matrix of interest.

b. Surrogates and internal standards must be spiked into each MDL sample as specified in the method. Internal standard area counts and retention times must meet method QC acceptance criteria. Recoveries and %RSDs for surrogates and target compounds must meet the criteria specified in the method. If the method does not specify recovery and/or replicate %RSD criteria, then the %RSD for the seven replicates should be less than or equal to 25% and the mean recovery for target compounds and surrogates should be between 80-120%.

c. Samples must be analyzed on the same instrument under the same conditions (trap, column, temperature program, amount of sample purged, etc.) as was used for the MDL study.

1. d. The MDL study must be performed within one year prior to the start of the preparation and/or analysis of the samples.

e. The MDL for each compound must be less than or equal to that compound's method-required quantitation limit.

2. Laboratory Fortified Blank (LFB)

a. Verification of laboratory accuracy at the quantitation level requires the routine analysis of an LFB spiked with target compounds at the quantitation limit and, internal standard and surrogate compounds spiked at the concentrations specified in the method. The stock solution used for spiking the LFB must be prepared from a source other than the source used for preparing the initial and continuing calibration standards.

b. One LFB containing all the target compounds at the quantitation limit must be analyzed immediately

prior to sample analysis but after instrument tuning and calibration. Subsequently, an LFB must be analyzed every 12 hours. One LFB must be extracted with each sample delivery group of semivolatile samples, or whenever semivolatile samples are extracted, whichever is more frequent.

c. Method QC acceptance criteria must be met for surrogates, internal standards and target compounds. If the method does not specify recovery QC acceptance criteria for the LFB, then the recovery for target compounds should be between 60-140%. Surrogate compounds and internal standards for the LFB must meet validation criteria as per Sections VI and VII of this document.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
Qualification of data should be based on an combined evaluation of both the MDL study and LFB results. To determine appropriate sample qualification, the MDL study should be evaluated first and then the LFB results.	All potential impacts on the sample data resulting from LFB and/or MDL study anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Method Detection Limit (MDL) Study	**1. Method Detection Limit (MDL) Study**
a. Verify that the MDL study was generated in accordance with the method and 40 CFR Part 136 App. B, and that a minimum of seven replicates for each matrix of interest were prepared and analyzed.	a. If the required MDL study was not performed at all or was not performed according to the CFR criteria, then the validator should evaluate the LFB data, if available, to determine the action to be taken. See Tables VOA/SV-X-1, VOA/SV-X-2, and VOA/SV-X-3. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.
b. Verify that internal standard area counts and retention times meet method QC acceptance criteria.	b. If internal standard area counts and/or retention times do not meet method QC acceptance criteria, then the validator should follow the guidance provided in Section VOA/SV-VII.

C. EVALUATION	D. ACTION
1. c. Compare all seven replicates of the MDL study to verify that the %RSD for each surrogate and target compound is less than or equal to 20%.	1. c. If the MDL target and surrogate compound %RSD criteria are exceeded, then the validator should evaluate initial calibration %RSDs to assess instrument precision and linearity. The validator should use professional judgment to assess the impact of laboratory precision on analytical sensitivity and data quality.
d. Compare all seven replicates of the MDL study to verify that the mean recovery for each target and surrogate compound is within 80-120%.	d. If the mean percent recovery for a target or surrogate compound is greater than 120%, then the validator should: - Use professional judgment to estimate (J) positive detects for that compound in all samples associated with that MDL study, taking into consideration the LFB results. - Accept the non-detects. If the mean percent recovery for a target or surrogate compound is less than 80% but greater than or equal to 10%, then the validator should: - Use professional judgment to estimate (J) positive detects for that compound in all samples associated with that MDL study, taking into consideration the LFB results. - Use professional judgment to estimate (UJ) the non-detects for that compound in all samples associated with that MDL study, taking into consideration the LFB results. If the mean percent recovery for a target or surrogate compound is less than 10%, then the validator should estimate (J) positive detects for that compound and reject (R) the non-detects for that compound in all samples associated with that MDL study.

C.	EVALUATION	D.	ACTION
*1. e.	Check and recalculate the %RSDs and % recoveries for at least three compounds per MDL study. Verify that the recalculated values agree within ± 10% of the reported results.	1. e.	If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
f.	Verify that the samples were analyzed on the same instruments and under the same conditions (trap, column, temperature program, amount of sample purged, etc.) as was used for the MDL study.	f.	If the samples were not analyzed on the same instruments or under the same conditions as the MDL study, then the validator should contact the laboratory to obtain the correct MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.
g.	Compare the date of the MDL study to the dates of all associated sample analyses to verify that the MDL study was performed within one year of the start of the first sample prepared and/or analyzed in the sample delivery group.	g.	If the MDL study was not submitted or was not performed within one year of the start of preparation and/or analysis of the first sample in the SDG, then the validator should contact the laboratory to obtain a current MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should evaluate the instrument's response to the lowest standard of the initial calibration and use professional judgment to assess the impact of analytical sensitivity on data quality.

C. EVALUATION	D. ACTION
1. h. Verify that all MDLs are less than or equal to the method-required quantitation limits.	1. h. If the MDL study reveals that a target compound has a detection limit greater than the method-required quantitation limit, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should: i. Elevate the quantitation limit for that target compound in all samples associated with that MDL study to the lowest concentration calibration standard analyzed or to the laboratory-reported MDL, whichever is higher. ii. Estimate (J) positive detects which were below the elevated quantitation limit for that target compound in all samples associated with that MDL study.
If the LFB criteria are not met, then laboratory performance related to method bias and method/instrument sensitivity is questionable.	
2. Laboratory Fortified Blank (LFB)	**2. Laboratory Fortified Blank (LFB)**
* a. Check the standards preparation logs to verify that the stock standard used to prepare the LFB was from a source independent from the initial and continuing calibration standards.	a. If the LFB was not prepared from a source independent from the initial and continuing calibration standards, then the laboratory performance related to method bias and method/instrument sensitivity is questionable. The validator should review other calibration verification checks, i.e., PES analyses to ensure calibration accuracy. Professional judgment should be used to qualify sample quantitation limits.
b. Verify that an LFB was prepared and/or analyzed at the correct frequency and that it was spiked with the correct compounds at their quantitation limits.	b. If an LFB analysis was not performed or the LFB was not analyzed for the correct compounds at the proper frequency and concentration, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.
c. Verify that the reported recoveries for all LFB spike compounds are within the method QC acceptance criteria.	c. Sample data should be qualified based on the number and type of compounds that recover outside the method QC acceptance criteria and based on the degree that compound recoveries exceed the criteria.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. i. If any of the LFB compound recoveries are outside the method QC acceptance criteria, then the LFB results should be used to qualify sample data for the specific compounds that are included in the LFB solution. The validator should use professional judgment to qualify sample data for non-LFB compounds, taking into account the compound's chemical class, compound recovery efficiency, and any analytical problems historically associated with the compound or that were encountered by the laboratory. ii. If an LFB compound recovery is greater than 140%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that LFB to indicate potential high bias. - Accept the quantitation limit of the affected compound in any sample associated with that LFB. iii. If more than half of the LFB compound recoveries are greater than 140%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LFB to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that LFB. iv. If an LFB compound recovery is less than 60% but greater than or equal to 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that LFB to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected compound in any sample associated with that LFB to indicate potential low bias.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. v. If more than half of the LFB compound recoveries are less than 60% but greater than or equal to 10%, then the validator should: - Estimate (J) all positive detects in all samples associated with that LFB to indicate potential low bias. - Estimate (UJ) all quantitation limits for non-detects in all samples associated with that LFB to indicate potential low bias. vi. If an LFB compound recovery is less than 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that LFB to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives. vii. If more than half of the LFB compound recoveries are less than 10%, then the validator should: - Estimate (J) all positive detects in all samples associated with that LFB to indicate potential low bias. - Reject (R) the quantitation limits for all non-detects in all samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. viii. If more than half of the LFB compound recoveries are outside the method QC acceptance limits in one LFB, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular compound, class of compounds or the entire fraction for samples associated with that LFB. ix. Action on non-compliant surrogate recoveries should follow the guidance provided in Section VOA/SV-VI. Professional judgment should be used to evaluate the impact that a non-compliant LFB surrogate recovery has on the sample data. x. Action on non-compliant internal standard areas should follow the guidance provided in Section VII. Professional judgment should be used to evaluate the impact that non-compliant LFB internal standard areas have on the sample data.
* d. Check and recalculate the % recovery for at least one compound per LFB fraction. Verify that the recalculated value agrees within ± 10% of the reported result.	d. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.e, C.2.a, C.2.d

Table VOA/SV-X-1:

QUALIFICATION OF ORGANIC ANALYTES BASED ON MDL STUDY RESULTS

Sample Results	Mean % Recovery			
	%Rec < 10%	10% ≤ %Rec < 80%	80% ≤ %Rec ≤ 120%	%Rec > 120%
Detects	J	Professional Judgment*	A	Professional Judgment*
Non-Detects	R	Professional Judgment*	A	A

Sample Results	% RSD	
	> 25%	≤ 25%
Detects	Professional Judgment**	A
Non-detects	Professional Judgment**	A

* Taking into consideration LFB results.
** Taking into consideration initial calibration %RSDs.

Table VOA/SV-X-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE: ≤ ONE-HALF OF LFB COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	%Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target compounds at or below the quantitation limit.

Table VOA/SV-X-3:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE: > ONE-HALF OF LFB COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	%Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target compounds at or below the quantitation limit.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

E. EXAMPLES

Example #1: (Low LFB recoveries for several compounds)

Low concentration water samples were analyzed under CLP SOW OLC02.1 and, therefore, no MDL study was required. LFB compounds, benzene, carbon tetrachloride, and trichloroethene recovered below QC acceptance criteria but greater than 10%, (22%, 40%, and 38%, respectively). The validator estimates (J) the positive benzene, carbon tetrachloride, and trichloroethene detects in all the field samples associated with the LFB to indicate potential low bias and estimates (UJ) the quantitation limits for the benzene, carbon tetrachloride, and trichloroethene non-detects in all the field samples associated with the LFB to indicate a decrease in sensitivity and the possibility of false negatives. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #2: (High LFB recoveries for two compounds; Low internal standard area counts)

Low concentration water samples were analyzed under CLP SOW OLC02.1 and, therefore, no MDL study was required. LFB compounds 1,2-dichloropropane and tetrachloroethene recovered outside the upper QC acceptance criteria (160% and 200%, respectively). The IS area for chlorobenzene-d5, in the LFB sample and in all field samples associated with the LFB, was reported below the QC acceptance criteria but greater than 20% of the continuing calibration IS response. Since all analytes associated with the IS chlorobenzene-d5 were estimated (J or UJ indicating a potential high bias) previously in all affected samples due to the low IS area counts, the validator notes the high LFB recoveries in the Data Validation Memorandum but takes no additional action on the Data Summary Table.

Example #3: (Low MDL recoveries for LFB compounds; Acceptable LFB results)

The analytical method used for sample analysis did not specify QC acceptance criteria for the MDL study. The validator uses the default criteria for mean % recoveries (80-120%) and %RSDs to evaluate the MDL data. The MDL study submitted by the laboratory did not meet the default MDL recovery criteria for styrene and vinyl chloride (55% and 32%, respectively). The validator examines the LFB data submitted with the field sample results and determines that all LFB method QC acceptance criteria were met including styrene and vinyl chloride. The validator accepts the field sample data based on the acceptable LFB results and notes the low MDL recoveries in the Data Validation Memorandum.

E. EXAMPLES

Example #4: (High LFB recoveries for two compounds; High MDL %RSDs for two compounds)

The analytical method used for sample analysis did not specify QC acceptance criteria for the MDL study. The validator uses the default criteria for mean % recoveries (80-120%) and % RSDs to evaluate the MDL data. The MDL study submitted by the laboratory did not meet default (25%) %RSD criteria for benzene and ethylbenzene (34% and 36%, respectively). The validator reviews the initial calibration %RSDs and determines that benzene and ethylbenzene met the initial calibration %RSD acceptance criteria. In addition, the analytical method used did not specify QC acceptance criteria for the LFB. The validator uses the default recovery criteria of 60-140% to evaluate LFB results. The validator examines the LFB submitted with the analytical results and determines that benzene and ethylbenzene also exceeded the LFB % recovery criteria of 140% (164% and 170%, respectively). Since the initial calibration %RSDs were acceptable, the high MDL %RSDs were not utilized to qualify sample data. Based upon the LFB recoveries, the validator uses professional judgment to estimate (J) the positive benzene and ethylbenzene detects to indicate potential high bias for these two compounds and accept the quantitation limits for benzene and ethylbenzene non-detects in all field samples associated with the LFB. The validator reports the qualified results on the Data Summary Table and notes the sample qualifications in the Data Validation Memorandum.

XI. PERFORMANCE EVALUATION SAMPLES/ACCURACY CHECK

A. OBJECTIVE

Data for Performance Evaluation Samples (PESs) are generated to provide information on the overall accuracy and bias of the analytical method and on laboratory performance. PESs are evaluated for false negatives, false positives, and inaccurate target compound quantitation. In general, the most serious problem a PES can expose is the failure of the laboratory to properly detect and identify a PES compound. This failure is known as a false negative. False negatives significantly increase the "uncertainty" surrounding any site decisions made concerning the "cleanliness" or contamination present at a site. A second problem revealed by PES analysis is the laboratory's erroneous detection of target and non-target compounds that were not spiked into the PES, otherwise known as false positives. False positives should always be evaluated in conjunction with blank data to ascertain the probable source(s) of contamination.
Finally, the PES provides information on the magnitude and direction of quantitative bias for the entire laboratory method, including sample preparation (extraction and cleanup) and analysis (chromatography and calibration). Sample data that are biased high or low can potentially impact site decisions, especially when sample data have target compound concentrations at or near project action levels.

Ideally, a PES is comprised of the same matrix as the field samples being evaluated. However, for many matrices (i.e., soil) PESs are not available. In these situations, a PES of another matrix (i.e., water) may be analyzed with the field samples to assess laboratory performance on the "analysis" portion, even though laboratory performance on the "sample preparation" portion cannot be assessed. The validator should use professional judgment when evaluating samples of one matrix using PES data from another matrix.

B. CRITERIA

1. Zero Blind Performance Evaluation Samples

A Zero Blind PES is a quality control sample that is of a composition and concentration known to the laboratory.

A Laboratory Control Sample (LCS) is a Zero Blind PES which is often used by the laboratory as an internal quality control check of analytical accuracy and method bias.

An LCS containing several or all of the target compounds spiked at concentrations at or below their quantitation limits is called a Laboratory Fortified Blank (LFB). Refer to Section X for additional LFB guidance.

a. An LCS is required by some EPA methods and certain CLP SOWs. The frequency, concentration, acceptance criteria and corrective actions for LCS analysis should be stated in the method, Sampling and Analysis Plan (SAP) or the Quality Assurance Project Plan (QAPjP) and should support the DQOs of the project. The LCS should be prepared in the proper matrix for each parameter at the concentration level and frequency required in the EPA-approved project SAP, QAPjP, and/or method. The LCS must contain one or more target compounds. The LCS must be prepared and analyzed concurrently with field samples contained in the sample delivery group.

1. b. The percent recoveries for LCS compounds must be within the method QC acceptance criteria.

c. Surrogate compounds and internal standards for the LCS must meet validation criteria as per Sections VI and VII of this document.

2. Single Blind Performance Evaluation Samples

A Single Blind PES is a quality control sample that is of a composition and concentration not known to the laboratory, but the sample is identified to the laboratory as a PES.

A Single Blind PES may be submitted with a sample delivery group to assess method bias, laboratory performance and to evaluate data quality. A Single Blind PES may also be submitted for analysis prior to sample shipment to prequalify a laboratory for a specific matrix and/or parameter.

a. The latest revision of the EPA Region I Performance Evaluation Program Guidance, requires that a Single Blind or Double Blind PES be sent with each sample delivery group (20 samples or less) that is sent to a laboratory. A PES is required for each matrix, parameter, and concentration level unless an EPA or non-EPA PES does not currently exist for that particular matrix, parameter, or concentration level.

The PE Program applies to the Superfund program including EPA Fund-lead and PRP/Federal Facility Oversight Projects. In addition, the PE Program applies to Fund-lead projects performed by States under Cooperative Agreements and other Federal Agencies under Interagency Agreements. The PE Program also applies to Non-Fund-
lead Superfund projects undertaken by potentially responsible parties. The PE Program also applies to Non-Superfund Programs.

EPA-provided PE samples are available for certain categories of Superfund work as specified in the latest revision of the EPA Region I Performance Evaluation Program Guidance. The EPA Performance Evaluation Chemist provides the current list of EPA-provided PE samples upon request. For those categories of Superfund work that do not have access to EPA-provided PE samples and for all Non-Superfund program work scientifically defensible PE samples should be obtained from commercial vendors.

b. Acceptance criteria for EPA PESs are statistically-derived by the Analytical Operations Center under the QATS contract. Tabulated report forms for EPA PESs must be submitted to the Region I OEME-QA Unit for scoring at the time of data validation, in accordance with the latest revision of the EPA Region I Performance Evaluation Program Guidance.

c. True values and QC acceptance criteria for all non-EPA PESs should be provided by the manufacturer and these acceptance criteria must be fully documented and must be scientifically defensible.

d. Surrogate compounds and internal standards for EPA and non-EPA Single Blind PE samples must meet validation criteria as per Sections VI and VII of this document.

3. Double Blind Performance Evaluation Samples

A Double Blind PES is a quality control sample that is of a composition and concentration not known to the laboratory and the sample is **not** identifiable as a PES nor is it identified to the laboratory as a PES.

A Double Blind PES may be submitted with a sample delivery group, in lieu of a Single Blind PES, to assess method bias, laboratory performance and to evaluate data quality.

a. The use of Double Blind PESs is dictated by the project DQOs and should be documented in the EPA-approved SAP and/or QAPjP.

b. True values and acceptance criteria for Double Blind PESs must be fully documented and must be scientifically defensible.

c. Surrogate compounds and internal standards for EPA and non-EPA Double Blind PE samples must meet validation criteria as per Sections VI and VII of this document.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from performance evaluation sample anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Zero Blind PES - LCS	**1. Zero Blind PES - LCS**
a. Verify that an appropriate LCS sample (correct parameter, concentration level, target compounds and matrix) was prepared and analyzed at the required frequency for each sample delivery group in accordance with the EPA approved project SAP, QAPjP and/or method.	a. If an appropriate LCS was not analyzed at the required frequency for the correct parameters, concentration levels, target compounds or matrices, then the validator should use professional judgment to determine if the sample data should be qualified or rejected.
b. Verify that the required LCS results are provided for each sample delivery group.	b. If the required LCS results were not submitted for each sample delivery group, then the validator should contact the laboratory to obtain raw data and tabulated results.

C. EVALUATION	D. ACTION
1. c. Verify that the reported recoveries for all LCS spike compounds are within the method QC acceptance criteria.	1. c. Sample data should be qualified based on the number and type of compounds that recover outside the method QC acceptance criteria and based on the degree that compound recoveries exceed the criteria. i. If any of the LCS compound recoveries are outside the method QC acceptance criteria, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. Professional judgment should be used to qualify sample data for non-LCS compounds, taking into account the compound's chemical class, compound recovery efficiency, and any analytical problems historically associated with the compound or that were encountered by the laboratory. ii. If an LCS compound recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that LCS to indicate potential high bias. - Accept the quantitation limit of the affected compound in any sample associated with that LCS. iii. If more than half of the LCS compound recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LCS to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that LCS.

C. EVALUATION	D. ACTION
1. c. Continued from above.	1. c. iv. If an LCS compound recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that LCS to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected compound in any sample associated with that LCS to indicate potential low bias. v. If more than half of the LCS compound recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LCS to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that LCS to indicate potential low bias. vi. If an LCS compound recovery is less than 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that LCS to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that LCS to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
1. c. Continued from above.	1. c. vii. If more than half of the LCS compound recoveries are less than 10%, then the validator should: - Estimate (J) all positive detects in all samples associated with that LCS to indicate potential low bias. - Reject (R) the quantitation limits for all non-detects in all samples associated with that LCS to indicate that the data are unusable due to the possibility of false negatives. viii. If more than half of the LCS compound recoveries are outside the method QC acceptance limits in one LCS, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular compound, class of compounds or the entire fraction for samples associated with that LCS. ix. Based upon the number and type of compounds misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.
d. Evaluate surrogate compounds and internal standards for the LCS.	d. Action on non-compliant surrogate recoveries and internal standard area counts should follow the guidance provided in Sections VI and VII, respectively. Professional judgment should be used to evaluate the impact that non-compliant LCS surrogate recoveries and/or internal standard area counts have on the sample data.

C. EVALUATION	D. ACTION
*1. e. Check and recalculate the percent recovery for at least one compound per LCS fraction. Verify that the recalculated value agrees within ± 10% of the reported result.	1. e. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Single Blind <u>and</u> Double Blind PESs a. Verify that an appropriate Single Blind or Double Blind PES (correct parameter, concentration level, target compounds and matrix) was analyzed at the required frequency for each sample delivery group in accordance with Region I PE policy and/or the EPA approved SAP and/or QAPjP. b. Verify that Single Blind PES results are provided for each sample delivery group in accordance with Region I PE policy.	**2. Single Blind <u>and</u> Double Blind PESs** a. If a required Single Blind or Double Blind PES was not analyzed at the required frequency for the correct parameters, concentration levels, target compounds or matrices, then the validator should use professional judgment to determine if the sample data should be qualified or rejected. b. If the PES results were not submitted for each sample delivery group, then the validator should contact the laboratory to obtain raw data and/or tabulated results. If a PES was not submitted to the laboratory by the sampler, then the validator should contact the sampler to confirm the omission of a PES and document that fact on the worksheet and in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
2. c. **EPA PESs:** If the PES was supplied and scored by Region I OEME-QA, then the Region I PES Score Report must be evaluated to determine how many of the analytes met or exceeded PES acceptance criteria.	2. c. **Region I EPA PESs** Note: PES results should not be qualified based on QC sample data and should not be reported on the Data Summary Table. Rather, PES results should be discussed in the Data Validation Memorandum or Tier I Validation Cover Letter and PES Score Reports should be attached as supporting documentation.
• Evaluate the "TCL MISSES" to assess the potential for low bias and false negative sample results.	• Sample data should be qualified based on the number and type of "TCL MISSES" identified on the Region I PES Score Report. i. If a PES compound is not identified in the PES, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives. ii. Based upon the chemical class, the number of compounds that were not identified, and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.
• Evaluate the "TCL CONTAMINANTS" and "TIC CONTAMINANTS" in conjunction with blank data to assess the potential for high bias and false positive sample results.	• Sample data should not be qualified based on the number and type of "TCL CONTAMINANTS" and "TIC CONTAMINANTS" identified on the Region I PES Score Report alone. i. If a TCL or TIC contaminant is detected in the PES and is also found in a blank, then the validator should evaluate and qualify sample data based upon blank contamination in accordance with Section V. ii. If a TCL or TIC contaminant is detected in the PES but is not present in any blank, then that interference is specific to the PES and does not impact sample data.

C. EVALUATION	D. ACTION
2. c. Continued from above. ● Evaluate the "TCL HITS" that were misquantified to assess the potential for high and/or low bias in sample data.	2. c. Continued from above. ● Sample data should be qualified based on the number and type of misquantified compounds (Action High/Action Low "TCL HITS") identified on the Region I PES Score Report. **Sample data should not be qualified based on "Warning Low/Warning High" scores for "TCL HITS".** i. If any of the PES compounds do not meet PES acceptance criteria, then the PES results should be used to qualify sample data for the specific compounds that are included in the PES sample. Professional judgment should be used to qualify sample data for non-PES compounds taking into account the compound's chemical class, compound recovery efficiency, and analytical problems historically associated with the compound or that were encountered by the laboratory. ii. If a PES compound is scored in the "Action High" category, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that PES to indicate potential high bias. - Accept the quantitation limit of the affected compound in any sample associated with that PES. iii. If more than half of the PES compounds are scored in the "Action High" category, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that PES.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. iv. If a PES compound is scored in the "Action Low" category, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives. v. If more than half of the PES compounds are scored in the "Action Low" category, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives. vi. If more than half of the PES compounds are scored in the "Action" levels in one PES, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular compound, class of compounds or the entire fraction for samples associated with that PES. vii. Based upon the number and type of compounds misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. Continued from above.
● Evaluate "TIC HITS" and "TIC MISSES".	● Sample data should be qualified based on the number and type of "TIC HITS" and "TIC MISSES" identified on the Region I PES Score Report. i. If TIC identifications are required by the method, then the validator should use professional judgment to qualify the sample data based upon entries in the "TIC HITS" and "TIC MISSES" categories.
● Evaluate surrogate compounds and internal standards for the EPA PES.	● Action on non-compliant surrogate recoveries and internal standard area counts should follow the guidance provided in Sections VI and VII, respectively. Professional judgment should be used to evaluate the impact that non-compliant EPA PES surrogate recoveries and/or internal standard area counts have on the sample data.
d. **Non-EPA PESs** If the PES was obtained from a source other than Region I OEME-QA, then the validator should use the vendor's criteria to evaluate the PES results. Confirm that PES acceptance criteria are fully documented and scientifically defensible.	d. **Non-EPA PESs** If the non-EPA PES acceptance criteria are not fully documented and/or scientifically defensible, then the validator should use professional judgment to qualify or reject the sample data.
● Evaluate the "PES COMPOUND MISSES" to assess the potential for low bias and false negative sample results.	● Sample data should be qualified based on the number and type of "PES COMPOUND MISSES" identified from the vendor's acceptance criteria. i. If a PES compound is not identified in the PES, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. d. Continued from above.	2. d. Continued from above. ii. Based upon the chemical class, the number of compounds that were not identified, and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.
• Evaluate the "PES COMPOUND CONTAMINANTS" in conjunction with blank data to assess the potential for high bias and false positive sample results.	• Sample data should not be qualified based on the number and type of "PES COMPOUND CONTAMINANTS" identified from the vendor's acceptance criteria alone. i. If a PES COMPOUND CONTAMINANT is detected in the PES and is also found in a blank, then the validator should evaluate and qualify sample data based upon blank contamination in accordance with Section V. ii. If a PES COMPOUND CONTAMINANT is detected in the PES but is not present in any blank, then that interference is specific to the PES and does not impact sample data.
• Evaluate the "PES COMPOUND HITS" that were misquantified to assess the potential for high and/or low bias in sample results.	• Sample data should be qualified based on the number and type of misquantified "PES COMPOUND HITS" identified from the vendor's acceptance criteria. i. If any of the PES compounds do not meet acceptance criteria, then the validator should use the PES results to qualify sample data for the specific compounds that are included in the PES sample. Professional judgment should be used to qualify sample data for non-PES compounds, taking into account the compound's chemical class, compound recovery efficiency, and analytical problems associated with the compound either historically or that were encountered by the laboratory.

C. EVALUATION	D. ACTION
2. d. Continued from above.	2. d. Continued from above. ii. If a PES compound recovery is outside the upper limit of the vendor's documented acceptance limits (Note: The validator should confirm that the vendor's acceptance limits are calculated as plus and minus three standard deviations from the mean, similar to EPA-PES "Action Limits".), then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that PES to indicate potential high bias. - Accept the quantitation limit of the affected compound in any sample associated with that PES. iii. If more than half of the PES compound recoveries are outside the upper limit of the vendor's documented acceptance limits (See note above, Section 2.d.ii), then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that PES. iv. If a PES compound recovery is outside the lower limit of the vendor's documented acceptance limits (See note above, Section 2.d.ii), then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. d. Continued from above.	2. d. Continued from above. v. If more than half of the PES compound recoveries are outside the lower limit of the vendor's documented acceptance limits (See note above, Section 2.d.ii), then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives. vi. If more than half of the PES compound recoveries are outside the vendor's documented acceptance limits in one PES (See note above, Section 2.d.ii), where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular compound, class of compounds or the entire fraction for samples associated with that PES. vii. Based upon the number and type of compounds misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.
• Evaluate surrogate compounds and internal standards for the non-EPA PES.	• Action on non-compliant surrogate recoveries and internal standard area counts should follow the guidance provided in Sections VI and VII, respectively. Professional judgment should be used to evaluate the impact that non-compliant non-EPA PES surrogate recoveries and/or internal standard area counts have on the sample data.

C. EVALUATION	D. ACTION
*2. e. Determine what percentage of PES analytes were below or above PES acceptance criteria.	2. e. If more than half of the PES compounds are high or low, then the validator should check the raw data and/or contact the laboratory to verify that the PE sample was prepared according to the PE instructions (if applicable). Check also that the appropriate PE instructions (for that PE concentration level) were sent to the laboratory.
* f. Check and recalculate the analytical concentrations for at least one compound per PES fraction. Verify that the recalculated value agrees within ± 10% of the reported result.	f. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum. i. If corrected data reports affect the original results reported on the initial EPA PES score report, then the validator should resubmit the corrected PES results to Region I OEME-QA for a PES rescore. Sample data should be reevaluated and requalified based on the corrected PES data. ii. If corrected data reports affect the original results reported for the initial non-EPA PES, then the validator should reevaluate and requalify sample data based on the corrected PES data.

* **Note: The following subsections are applicable only to a Tier III data validation: C.1.e, C.2.e, C.2.f**

Table VOA/SV-XI-1:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON LCS RECOVERIES WHERE: ≤ ONE-HALF OF LCS COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table V/SV-XI-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LCS RECOVERIES WHERE: > ONE-HALF OF LCS COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS*

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table VOA/SV-XI-3:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON PES RESULTS WHERE:
≤ ONE-HALF OF PES COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	**•Single Blind •Double Blind PES < Lower Limit "Action Low"**	**•Single Blind •Double Blind PES "Within Warning Limits" "Warning High/Warning Low"**	**•Single Blind •Double Blind PES > Upper Limit "Action High"**
Detects	J	A	J
Non-Detects	R	A	A

Table VOA/SV-XI-4:

QUALIFICATION OF ORGANIC ANALYTES BASED ON PES RESULTS WHERE:
> ONE-HALF OF PES COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS *

Sample Results	**•Single Blind •Double Blind PES < Lower Limit "Action Low"**	**•Single Blind •Double Blind PES "Within Warning Limits" "Warning High/Warning Low"**	**•Single Blind •Double Blind PES > Upper Limit "Action High"**
All Detects	J	A	J
All Non-Detects	R	A	A

* Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

E. EXAMPLES

Example #1: (One LCS compound < lower limit; One LCS compound > upper PES acceptance limit)

A Laboratory Control Sample (LCS) containing 10 compounds spiked at three times the quantitation limit is found to have chlorobenzene with a % recovery of 150% and vinyl chloride with a % recovery of 50%. The method QC acceptance criteria for LCS compound recoveries are 60-140%. This amounts to less than one-half of the spike LCS compounds being outside the LCS acceptance criteria. The validator estimates (J) positive detects for chlorobenzene and vinyl chloride in all field samples associated with that LCS. The validator accepts the chlorobenzene non-detects and estimates (UJ) the vinyl chloride non-detects in all field samples associated with that LCS. The validator reports qualified data on the Data Summary Table and notes that the chlorobenzene positive detects are biased high, the vinyl chloride positive detects are biased low and the vinyl chloride non-detects contain possible false negatives in the Data Validation Memorandum.

Example #2: (One Single Blind PES compound < lower PES acceptance limit)

A Single Blind Performance Evaluation Sample (PES) is found to have a chloroethane positive result that scored below the lower PES acceptance limit. The validator determines that less than one-half of the spike PES compounds are outside the PES acceptance criteria. Therefore, the validator estimates (J) positive chloroethane detects and rejects (R) the quantitation limits for chloroethane non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and notes that the positive chloroethane detects are biased low and chloroethane non-detects are rejected due to the possibility of false negatives in the Data Validation Memorandum.

Example #3: (More than one-half of PES compounds greater than upper PES acceptance limits)

A Single Blind PES is found to have more than one-half of the spike volatile PES compounds with % recoveries above the upper PES acceptance limits. The validator estimates (J) all positive detects in all field samples associated with that PES and accepts (A) all quantitation limits for non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and notes the positive volatile results are biased high in the Data Validation Memorandum.

Example #4: (More than one-half of PES compounds "Action High" or "Action Low")

A Single Blind PES is found to have more than one-half of the spike semivolatile PES compounds with results that do not meet PES acceptance criteria. Some of the PES compounds are flagged "Action Low" and some flagged "Action High". The site DQOs are to determine whether cleanup levels were achieved. The validator determines that analytical error yields uncertainty in quantitative accuracy which may adversely affect site decisions. Therefore, the validator uses professional judgment to estimate (J) all positive detects in all field samples associated with that PES and reject (R) all quantitation limits in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and discusses the limited use of the data in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (One "TCL MISS")

A Single Blind PES is found to have one "TCL MISS" for vinyl chloride which is a contaminant of concern at the site. The validator estimates (J) all positive vinyl chloride detects and rejects (R) all vinyl chloride quantitation limits in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

Example #6: (One "TCL Contaminant", also in blank)

A Single Blind PES is found to have one "TCL Contaminant", 1,2-dichloroethane, at 45 ppb. The method blank contained 6 ppb of 1,2-dichloroethane, resulting in a Blank Action Level of 30 ppb. The validator uses the 1,2-dichloroethane Blank Action Level to evaluate the sample data and reports qualified data on the Data Summary Table. The validator suspects that the 1,2-dichloroethane false positive PES compound is a result of laboratory contamination and discusses this in the Data Validation Memorandum. PES results are not reported on the Data Summary Table.

Example #7: (One "TCL Contaminant", not in blank)

A Single Blind PES is found to have one "TCL Contaminant", 2-chlorophenol, which is not detected in any of the blanks but is detected in two samples. The validator determines that the 2-chlorophenol is an interference specific to the PES because it was not detected in any of the method, instrument, or storage blanks. The validator uses professional judgment to accept the positive 2-chlorophenol detects in the field samples. The validator reports the data unqualified on the Data Summary Table and discusses this in the Data Validation Memorandum.

XII. TARGET COMPOUND IDENTIFICATION

A. OBJECTIVE

Qualitative criteria for compound identification have been established to minimize the number of erroneous compound identifications. An erroneous identification can be either a false positive (reporting a compound that is not present) or a false negative (not reporting a compound that is present).

The identification criteria can be applied more easily in detecting false positives than false negatives (non-detects). More information is available for false positives due to the requirement for submittal of data supporting positive identifications. False negatives represent an absence of data and, therefore, are more difficult to assess. However, false negatives can be revealed when a compound is identified and reported to be a TIC when it should have been reported as a target compound.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been met. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. The relative retention time (RRT) for the sample compound must be within ± 0.06 RRT units of the daily standard RRT.

2. Mass spectra for the sample compound and a current laboratory-generated standard (i.e., the mass spectrum from the associated daily calibration standard) must match according to the following criteria:

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10 percent must be present in the sample spectrum.

   b. The relative intensities of these ions must agree within ± 20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance must be between 30 percent and 70 percent.)

   c. Ions present at greater than 10 percent in the sample mass spectrum but not present in the standard spectrum must be considered and accounted for.

3. All major chromatographic peaks (i.e., peaks present in the sample chromatogram at greater than 10 percent of the nearest internal standard) must be identified as either target compounds, TICs, surrogate compounds, or internal standards.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from target compound identification anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
*1. Check that the RRT of a reported compound is within ± 0.06 RRT units of the standard RRT.	1. a. If the RRT of a reported compound is outside of the retention time criteria, then the validator should use professional judgment to determine if mass spectral identification criteria have been met and if the compound has been correctly identified. b. If the reported compound does not meet mass spectral identification criteria and has been incorrectly reported, then the validator should report the compound as a non-detect and document the rationale for this decision in the Data Validation Memorandum. c. If instrument/analytical column malfunctions have severely affected retention times, making data suspect, then the validator should use professional judgment to reject (R) all associated sample data.

C. EVALUATION	D. ACTION
*2. Compare all sample compound spectra to the laboratory standard spectra and verify that the mass spectral identification criteria are met.	2. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is left to the validator's discretion to obtain additional information from the laboratory if it is deemed necessary. If it is determined that incorrect laboratory identifications were made, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which identification is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*3. Check the sample chromatogram to verify that all major peaks of interest are identified as either target compounds, TICs, surrogate compounds, or internal standards.	3. If a chromatographic peak is unaccounted for and is greater than 10% of the nearest internal standard, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms.
*4. The validator should be aware of situations (e.g., high concentration samples preceding low concentration samples or when VOA samples are purged in a contaminated sparge unit) when sample carryover is a possibility, and should use professional judgment to determine if instrument cross-contamination has affected any compound identification. An instrument blank should be run immediately after samples which cause detector saturation.	4. If cross-contamination has occurred, then the validator should use professional judgment to determine whether or not a reported target compound is native to the sample or an interferent from a previously analyzed sample. Additionally, the validator should use professional judgment to determine whether or not sample carryover has resulted in false negatives due to mass spectral identification criteria (pertaining to ions present and abundances) which cannot be met due to interfering ions from cross contaminants.

Note: **This section is applicable only to a Tier III validation - If a validator suspects compound misidentification while performing a Tier II validation, then the Site Manager must be contacted to approve the necessary full or partial Tier III validation.**

E. EXAMPLES

Example #1: (False negative-all major chromatographic peaks not identified)

The laboratory originally reported phenol as a TIC in the volatile fraction of soil sample SAA12. Phenol was reported as a non-detect in the semivolatile fraction. Upon review of the semivolatile chromatogram for sample SAA12, the validator notes that the laboratory failed to identify a peak that eluted within the phenol retention time window. The laboratory was contacted and requested to requantitate the false negative semivolatile phenol result and report phenol as a positive detect in the semivolatile fraction and deletes it from the VOA TIC list. The laboratory complied and the validator reports phenol as a positive detect in the semivolatile fraction on the Data Summary Table.

Example #2: (False positive; False negative-mass spectral identification criteria not met)

In aqueous sample SAA04, the validator notes that naphthalene and 2-chlorophenol have the same retention time on the quantitation report. The sample mass spectrum contains the molecular ion 128 and the laboratory reported naphthalene as a positive detect. Review of the mass spectrum shows a chlorine isotope ion at m/z 130 and fragmentation ions consistent with 2-chlorophenol, therefore, the validator determines that 2-chlorophenol is a more accurate identification of this peak. The laboratory was contacted and requested to requantitate the false positive naphthalene and false negative 2-chlorophenol. The validator reports 2-chlorophenol as a positive detect and naphthalene as a non-detect on the Data Summary Table.

Example #3: (False positive-sample compound RRT not within ±0.06 RRT units of the standard compound RRT)

The laboratory originally identified a peak as acetone and reported acetone as a positive detect in sample SAA67. The mass spectrum contained low area counts for ion 58 and the validator suspects a false positive. Upon review of the retention time data, the validator discovers that the RRT for the reported acetone peak was not within the standard ± 0.06 retention time window. The validator uses professional judgment to determine that acetone was misidentified. This unknown compound is less than 10% of the area of the nearest IS and, therefore, it is not reported as a TIC. The validator reports acetone as a non-detect on the Data Summary Table and documents this problem in the Data Validation Memorandum.

XIII. COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMITS

A. OBJECTIVE

The objective for the evaluation of compound quantitation and reported quantitation limits is to ensure that reported quantitative results and quantitation limits are accurate. To this end, laboratory calculations from raw data to the final reported concentrations are checked for accuracy.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. Reported quantitation limits must meet project-required DQOs.

2. a. Reported concentrations for positive detects and compound quantitation limits for non-detects and adjustments of those concentrations/compound quantitation limits must be calculated according to the appropriate method requirements.

   b. Reported concentrations for positive detects and compound quantitation limits for non-detects must be adjusted for percent solids, dilutions, concentrations and cleanup procedures that are not accounted for in the method.

3. a. Target compound quantitation must be based on the internal standard (IS) specified in the method.

   b. Target compound quantitation must be based on the quantitation ion (m/z) specified in the method for both the IS and target compound.

   c. Target compound quantitation must be calculated using the RRF from the appropriate daily standard.

4. Target compound quantitation must be within the initial calibration range.

5. All soil/sediment/solid sample results must be adjusted for percent solids, and must have percent solids greater than 30 percent. [1]

   Sediment samples are collected at CERCLA sites to establish whether or not the presence of hazardous chemicals has impacted the resident organisms and their natural environment. The data quality objectives for ecological risk assessment generally require that the analytical method used for sediment analysis achieve, at a minimum, the dry weight CLP SOW quantitation limits.

   [1]U.S. EPA Office of Water Regulations and Standards Industrial Technology Division - Method 1620, p. 29, Section 14.16, Draft September 1989.

   Most analytical methods that deal with soil-type matrices are applicable to both soils and sediments with no difference in how those two matrices are prepared and analyzed. Since a definition for soil and sediment matrices is not provided in the analytical methodology, Region I has adopted the definition for soil samples used by the Office of Water Regulations and Standards Industrial Technology Division (ITD). This definition states that soil samples are "soils, sediments, and sludge samples containing more than 30% solids".

   High moisture sediments cannot be successfully analyzed by routine CLP analytical methods. Additional sampling and analytical preparation steps, which are outside of the scope of a CLP method, should be employed. For example, standing water may first be decanted, and then the

sample may be centrifuged or filtered to remove excess water (except in the case of samples to be analyzed for volatile organics). To achieve the dry weight quantitation limits, the laboratory must perform a percent solids analysis prior to extraction and the initial volume of sample extracted must be increased accordingly. This presumes that the samplers have collected sufficient volume, above and beyond normal volume requirements, so that additional sample can be extracted. As a last resort, the laboratory can decrease the final extract volume to a minimum of 0.5 milliliters.

Certain solid matrices, such as peat, are unusual in both their reactive chemistry as well as their associated data quality objectives. Peat is a natural sink for organic compounds. It is composed of both a solid spongy matrix (which tightly binds organic compounds) and the interstitial pore water present therein.

Routine analytical methods underestimate the concentrations of organic compounds in peat matrices because the typical organic preparation and extraction techniques do not breach the matrix. In order for peat to be successfully analyzed, the matrix itself must be "sheared" into small pieces to increase surface area so that the extraction solvent can interact to partition the target organic compounds.

Sampling and analytical methodologies must be determined during project scoping processes and must be based on the project data quality objectives. For more information, see Attachment A of the Data Validation Manual.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from compound quantitation anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the reported quantitation limits meet project-required DQOs.	1. If reported quantitation limits do not meet the project-required DQOs, then the validator must investigate and document the cause of the deficiency and use professional judgment to assess sample data.
*2. a. Recalculate, from the raw data, the concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or a soil sample) for each fraction, in every field sample to verify that laboratory reported sample results were accurately calculated according to the method.	2. a. If incorrect values, equations or factors have been used to calculate sample results and/or sample quantitation limits, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*2. b. Verify that the concentrations for positive detects and sample quantitation limits have been adjusted to reflect sample dilutions, concentrations, cleanup methods and dry weight factors that are not accounted for in the method.	2. b. If the concentrations for positive detects and/or sample quantitation limits were not correctly adjusted for sample dilutions, concentrations, cleanup methods, or dry weight factors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*3. Verify that the correct internal standard, quantitation ion and standard RRF were used to quantitate sample results for at least one positive detect in each fraction in every field sample.	3. If the laboratory utilized an incorrect IS, quantitation ion, or RRF to quantitate a target compound, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
4. Verify that the concentrations for positive detects are within the initial calibration range.	4. a. If the concentrations for positive detects exceed the upper limit of the initial calibration range and no dilutions were reported, then the validator should estimate (J) those positive detects that exceed the initial calibration range. b. If the concentrations for positive detects fall below the lower limit of the initial calibration range, then the validator should estimate (J) those positive detects.

C. EVALUATION	D. ACTION
5. Ascertain if any soil/sediment/solid sample has less than or equal to 30 percent solids.	5. a. If a soil/sediment/solid sample has greater than 30 percent solids, then the validator should accept all sample data. b. If a soil/sediment/solid sample has percent solids of greater than or equal to 10% but less than or equal to 30%, then the validator should: • Estimate (J) positive detects. • Reject (R) non-detects. c. If a soil/sediment/solid sample has less than 10 percent solids, then the validator should reject (R) positive and non-detect sample results as unusable. d. The validator should include a discussion of the sample matrices having low percent solids in the Data Validation Memorandum. The validator may need to contact the field sampler to determine whether sampling techniques were appropriate for the sample matrix.

Note: **The following subsections are applicable only to a Tier III data validation:**

C.2.a, C.2.b, C.3

Table VOA/SV-XIII-1:

QUALIFICATION OF VOLATILE/SEMIVOLATILE ORGANIC ANALYTES BASED ON SAMPLE PERCENT SOLIDS

Sample Result	% Solids > 30%	10% ≤ % Solids ≤ 30%	% Solids < 10%
Detects	A	J	R
Non-detects	A	R	R

E. EXAMPLES

Example #1: (10% ≤ % Solids ≤ 30%)

DQOs for the Oak Street site specify that soil samples be analyzed for low level PAHs and other semivolatile compounds to assess human health risk posed by the site contamination. Semivolatile soil sample SAA58 had 15% solids and positive detects for chrysene, naphthalene, and benzo(a)pyrene. Due to the low percent solids, the chrysene, naphthalene, and benzo(a)pyrene detects are estimated (J) and all semivolatile non-detects are rejected (R) as unusable because the elevated sample quantitation limits do not meet project DQOs. The validator reports the qualified data on the Data Summary Table and notes this problem in the Data Validation Memorandum.

Example #2: (% Solids < 10%)

Volatile sediment sample SAA89 had 8% solids and positive detects for chlorobenzene, benzene, and trichloroethene. As a result of the extremely low percent solids (< 10%), the validator rejects (R) as unusable all positive detects and non-detects for this sample. The validator contacts the field sampler to determine if sampling techniques were inappropriate for the sample matrix resulting in high moisture content. The validator reports the qualified data on the Data Summary Table and discusses the high moisture content of the sample and the inappropriateness of the sampling and/or analytical methods in the Data Validation Memorandum.

XIV. TENTATIVELY IDENTIFIED COMPOUNDS

A. OBJECTIVE

Chromatographic peaks that are not target analytes, surrogate compounds, or internal standards are potential tentatively identified compounds (TICs). TICs must be qualitatively identified by a mass spectral library search, followed with interpretation by the laboratory's mass spectral interpretation specialist for potential compound identification. Laboratory-reported TICs are also assessed by the data validator.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. In accordance with the method, the laboratory must conduct mass spectral library searches for each sample and blank to report the possible identity of a specified number of volatile and semivolatile chromatographic peaks which are not surrogate compounds, internal standards, or target compounds, but which have an area count or peak height greater than 10 percent of the area count or peak height of the nearest internal standard. All GC/MS library searched mass spectra for every sample and blank must be examined by the laboratory for tentative compound identification.

NOTE: The laboratory should not report, as a tentatively identified compound, any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

2. TIC concentrations should be qualified by the laboratory as estimated (J). TIC concentrations should be calculated by the laboratory assuming an RRF of 1.0 and using the closest eluting IS that is free of interferences.

3. Chromatograms for blanks should not contain any TIC peaks.

4. Guidelines for making tentative identifications are as follows:

   a. Major ions (greater than 10 percent relative intensity) in the reference spectrum should be present in the sample spectrum.

   b. The relative intensities of the major ions should agree within ± 20 percent between the sample and reference spectra.

   c. Molecular ions present in the reference spectrum should be present in the sample spectrum.

4. d. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or coelution of additional TIC or target compound(s).

   e. Since library searches often yield several candidate compounds having closely matching scores, all reasonable choices must be considered and the most reasonable candidate chosen.

   f. When the above criteria are not met, but in the technical judgment of the validator or mass spectral interpretation specialist the identification is correct, the validator may report the identification.

   g. If in the validator's judgment the identification is uncertain or there are extenuating factors affecting compound identifications, the TIC result may be reported as "unknown".

5. The following common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants) should not be reported as TICs.

   Examples:

   a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluoro-trichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

   b. Solvent preservatives such as cyclohexene - a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

   c. Aldol condensation reaction products include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from tentatively identified compound anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
* 1. a. Verify that the laboratory has generated a library search for all required peaks in the sample and blank chromatograms.	1. a. If the laboratory has neglected to generate a library search for all required peaks, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms should be resubmitted. If a discrepancy remains unresolved, the validator must use professional judgment to decide which identification is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
b. Verify that reported TIC peaks were not surrogate compounds or internal standards.	b. If the laboratory performed a library search on a surrogate compound or internal standard, the validator should not report that compound as a TIC on the Tentatively Identified Compounds Table-Table III.
c. Verify that a target compound from another organic fraction was not reported as a TIC.	c. If the laboratory reported a target compound from another organic fraction as a TIC, then the validator should check that fraction to determine if the laboratory correctly identified the target compound in that organic fraction. If the laboratory did not correctly identify the target compound in that fraction, then the laboratory should be contacted to requantitate the false negative result, report that compound with the proper fraction, and remove that compound from the TIC form.

C. EVALUATION	D. ACTION
*1. d. Verify that a target compound was not missed by the target compound search procedure and erroneously reported as a TIC in the proper analytical fraction. The validator should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether data from the entire case may be affected.	1. d. If the laboratory reported a target compound from the proper fraction as a TIC, then the validator should contact the laboratory to requantitate the false negative result, report that compound on the correct form, and remove that compound from the TIC form.
*2. Verify that all TICs are reported with estimated (J) concentrations by the laboratory. Verify that TIC concentrations were calculated correctly, assuming a RRF of 1.0 and using the closest eluting IS that is free of interferences.	2. Qualify all TIC concentrations as estimated (J) if the laboratory has not already done so. If the laboratory did not quantitate the TIC assuming an RRF of 1.0 and using the appropriate IS, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*3. Verify that the blanks do not contain any TIC peaks. When a low level non-target compound is detected in a sample, a thorough check of blank chromatograms may be required. Look for peaks which are less than 10% of the area/height of the nearest, interference-free IS, and which are present in the blank chromatogram at a similar relative retention time.	3. a. If any TIC is found in a sample at a concentration greater than 10 times the level detected in an associated blank, then the TIC should be reported. b. If any TIC is found in a sample at a concentration less than or equal to 10 times the level detected in an associated blank, then the TIC should not be reported.

C. EVALUATION	D. ACTION
*4. a. Examine all TIC mass spectra in every sample and blank. Compare sample TIC spectra with all library search spectra to confirm that the most reasonable candidate was chosen according to the criteria set forth in Section XIV, B.4.	4. a. The validator must use professional judgment to determine if the criteria in Section XIV, B.4 were met and a reasonable identification was made. If there is more than one possible match, then the result may be reported as "either compound X or compound Y". If there is a lack of isomer specificity, the TIC result may be changed to a non-specific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to substituted aromatic compound). The validator may elect to quantitatively report all similar isomers as the sum of the individual isomers. For example, all alkanes may be quantitatively summed and reported as total hydrocarbons. The validator must summarize any changes made to the laboratory data and must document the rationale used to justify those changes in the Data Validation Memorandum.
* b. Verify that TICs were reported as unknowns if the TIC spectra presented do not meet the criteria set forth in Section XIV, B.4 and thus no reasonable choices could be determined.	b. If it is determined that a tentative identification of a non-TCL compound is unacceptable, then the tentative identification should be changed to unknown or to an appropriate identification. c. Other case factors may influence TIC judgments. If a sample TIC match is poor but other samples have a TIC with a good library match, similar relative retention time, and the same ions, then identification information may be inferred from the other sample TIC results.
* 5. Review blank and sample TIC spectra to ensure that common laboratory artifacts/contaminants are not reported as TICs. (See Section XIV, B.5 for examples of common laboratory artifacts/contaminants.)	5. If a common laboratory artifact and/or contaminant is reported as a TIC in a blank or sample, then the validator should not report the TIC on Table III TICs.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.a, C.1.d, C.2, C.3, C.4.a, C.4.b, C.5

E. EXAMPLES

Example #1: (Target analyte improperly reported as TIC in another fraction)

The laboratory originally reported 1,2-dichlorobenzene as a TIC in the volatile fraction of soil sample SAA12. 1,2-dichlorobenzene, however, was reported as a non-detect in the semivolatile fraction. Upon review of the semivolatile chromatogram for sample SAA12, the validator notes that the laboratory failed to identify a peak that eluted within the 1,2-dichlorobenzene retention time window. The laboratory was contacted and requested to requantitate the false negative semivolatile 1,2-dichlorobenzene result and report 1,2-dichlorobenzene as a positive detect in the semivolatile fraction, as well as remove the result from the VOA TIC form. The laboratory complied and the validator reports 1,2-dichlorobenzene as a positive detect in the semivolatile fraction on the Data Summary Table.

Example #2: (TIC not reported, lack of spectral confirmation)

Dichloronaphthalene is reported as a TIC in semivolatile sample SAA35. The reference dichloronaphthalene mass spectrum has a molecular ion of 196 and a 198, m+ 2, ion, with a relative intensity of 66.0%. The sample dichloronaphthalene mass spectrum has a molecular ion of 196 but the 198 ion has a 10.0% relative intensity. Because the sample spectrum's chlorine isotope (m+ 2 ion) relative intensity is not within ± 20.0% of the reference spectrum's relative intensity, the presence of dichloronaphthalene is not confirmed in the field sample. The validator uses professional judgment to determine that dichloronaphthalene is not present in the field sample, changes the TIC designation to "unknown", and justifies this in the Data Validation Memorandum. The validator does not report that TIC on the "Tentatively Identified Compound-Table III" since "unknowns" are not included on that table.

Example #3: (Unreported peak with relative intensity greater than 10% of the nearest IS)

The validator verifies that all peaks greater than 10% of the nearest IS for sample SAA01 are accounted for in the chromatogram and quantitation report for sample SAA01. To do this, the validator identifies target compound, internal standard, and surrogate peaks on the chromatogram quantitation report, and the Form I. The remaining peaks (greater than 10% of the nearest IS) should be listed as TICs. The validator notes that one peak (greater than 10% of the nearest IS) is unaccounted for and contacts the laboratory to obtain sample and reference mass spectra and to request revision of the Form I TIC. The laboratory complies and the validator reports that TIC on the "Tentatively Identified Compound-Table III" in the Data Validation Memorandum.

XV. SEMIVOLATILE CLEANUP

A. OBJECTIVE

Semivolatile cleanup procedures are utilized to remove matrix interferences from sample extracts prior to analysis. If not removed from the sample extracts, matrix interferences can inhibit accurate compound identification and quantitation resulting in highly suspect data. Semivolatile cleanup procedures are checked by spiking the cleanup columns or cartridges with target compounds, and evaluating the recovery of semivolatiles through the cleanup procedure.

Several types of semivolatile cleanup procedures exist, including but not limited to:

1. **Gel Permeation Chromatography (GPC)** - separates compounds based on molecular size and can be used to remove high molecular weight interferents.

   GPC is a size exclusion procedure that utilizes organic solvents and hydrophobic gels to separate macromolecules. The packing gel is porous and is characterized by the exclusion range (range of uniformity) of that pore size. The exclusion range must be greater than those of the molecules to be separated.

   General applications of GPC as a cleanup procedure for semivolatile organic fractions include the removal of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids and dispersed high molecular-weight compounds from the sample extract.

   Under CLP SOW OLM03.2, the GPC column is packed with bead-like packing and connected to a UV detector. After the GPC is calibrated and a blank analyzed, sample extracts are loaded into sample loops and an automated sequence is started. The target compounds are eluted with methylene chloride and collected during the pre-determined retention times. The high molecular weight interferences, those outside the exclusion range, elute earlier than the TCL semivolatile compounds during the "dump" phase, while the smaller interferents such as sulfur elute with a later volume of solvent during the "wash" phase.

2. **Silica Gel Cleanup** - separates interferents of different polarity.

   Silica gel is a regenerative adsorbent of amorphous silica with weakly acidic properties and is used for separating compounds of differing chemical polarity. Silica gel can be used for the cleanup of sample extracts containing polynuclear aromatic hydrocarbons (PAHs) and derivatized phenolic compounds.

   The silica gel column is packed with the required amounts of adsorbent, topped with a water adsorbent, and then loaded with a sample extract. The analytes are eluted with solvents of increasing polarity, to achieve desired separation, leaving the interfering compounds on the column.

Note: The CLP SOW OLM03.2 semivolatile method uses only GPC cleanup.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. **Gel Permeation Chromatography**

   a. Semivolatile sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

   b. The GPC system must be calibrated initially in accordance with the method prior to the analysis of field samples, QC samples or blanks to ensure acceptable solid phase activation, peak shape, and resolution of target compounds and interferents.

   c. i. GPC calibration must be checked on a continuing basis at the frequency specified in the method.

      ii. The method-required GPC calibration check solution must contain target and surrogate compounds and interferents at the method-required concentrations and must be analyzed according to the analytical method.

      iii. Target compound recoveries must meet method QC acceptance criteria.

      iv. Surrogate compound and internal standard area counts and/or retention times must meet method QC acceptance criteria.

      v. Peak shapes must be symmetrical and resolution must meet method QC acceptance criteria.

1. c. vi. Retention time shifts between GPC calibration checks must not exceed ±5% between calibrations.

   d. i. A GPC instrument blank spiked with surrogate compounds must be analyzed after each GPC calibration and calibration check and prior to sample analysis.

      ii. Target compounds must not be present at greater than or equal to the quantitation limit for any target compound in the GPC instrument blank.

      iii. Surrogate compound recoveries and internal standard area counts and/or retention times (if added) in GPC instrument blanks must meet method QC acceptance criteria after GPC cleanup. Note: CLP SOW OLM03.2 does not require the addition of surrogate compounds or internal standards to the GPC instrument blank.

2. **Silica Gel Cleanup**

   a. Semivolatile sample extracts, QC sample extracts and method blank extracts must undergo all cleanup procedures required by the method.

   b. Each lot number of solid phase adsorbent must be checked in accordance with the method prior to use to ensure acceptable solid phase activation, recovery of target analytes, and elimination of interferents.

   c. i. A Silica Gel Check solution must be prepared with each cleanup batch and must be analyzed prior to the Silica Gel column reagent blank. For each batch of samples undergoing Silica Gel column cleanup, the column performance must be checked with a Silica Gel Check solution to demonstrate that the compounds of interest are being quantitatively recovered.

      ii. The method-required Silica Gel Check solution must contain target and surrogate compounds and interferents at method-required concentrations and must be prepared and analyzed according to the analytical method.

      iii. Target compound recoveries must meet method QC acceptance criteria.

      iv. Surrogate compound and internal standard area counts and/or retention times must meet method QC acceptance criteria.

d. i. A Silica Gel column reagent blank spiked with surrogate compounds must be prepared with each cleanup batch. The Silica Gel column reagent blank must be analyzed after the Silica Gel Check solution and prior to field samples.

ii. Target compounds must not be present at greater than or equal to the quantitation limit for any target compound in the Silica Gel column reagent blank.

iii. Surrogate compound recoveries and internal standard area counts and/or retention times (if added) in Silica Gel column reagent blanks must meet method QC acceptance criteria after Silica Gel column cleanup.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from sample cleanup anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Gel Permeation Chromatography (GPC)	**1. Gel Permeation Chromatography (GPC)**
a. Verify from result forms, if available, that GPC cleanup was performed according to the analytical method on all method-required sample extracts, QC sample extracts, and method blank extracts.	a. If GPC was not performed according to the analytical method on all method-required extracts, then the raw data should be reviewed for the presence of high molecular weight contaminants and professional judgment should be used to qualify or reject sample data. The validator should request sample cleanup and reanalysis if GPC was required by the method.
* b. Verify that the GPC system was calibrated initially in accordance with the method requirements and that peak shape and resolution criteria were met.	b. If the GPC system was not calibrated initially in accordance with the method (prior to the analysis of field samples, QC samples or blanks) or fails to meet peak shape and/or resolution criteria or the initial calibration data are not available for review, then the validator should evaluate the last calibration check analyzed just prior to sample analysis.
* c. i. Confirm from the raw data that the GPC calibration check was performed at the method-required frequency.	c. i. If GPC calibration checks have not been performed at the method-required frequency, then the quality of the GPC operation may be suspect and the validator should use professional judgment to qualify or reject sample data.
* ii. Verify that a GPC calibration check solution was analyzed in accordance with the method and that the correct target and surrogate compounds, interferents and concentrations were used.	ii. If a GPC calibration check solution was not analyzed in accordance with the method or the correct compounds and/or concentrations were not used, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.

C. EVALUATION	D. ACTION
1. c. iii. Check the reported data from the GPC calibration check solution analyses to verify that target compound recoveries meet method QC acceptance criteria.	1. c. iii. If GPC calibration check method QC acceptance criteria are not met, then the GPC calibration check solution results should be used to qualify sample data for specific compounds included in the check solution. Professional judgment should be used to qualify or reject sample data for non-check solution compounds, taking into consideration the compound's chemical class. The validator should discuss the impact of unacceptable recoveries on the sample data in terms of high or low bias and note this in the Data Validation Memorandum. If a GPC calibration check compound recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that GPC calibration check to indicate potential high bias. - Accept the quantitation limit of the affected compound in any sample associated with that GPC calibration check. If more than half of the GPC calibration check compound recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that GPC calibration check to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that GPC calibration check. If a GPC calibration check compound recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that GPC calibration check to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected compound in any sample associated with that GPC calibration check to indicate potential low bias.

C. EVALUATION	D. ACTION
1. c. Continued from above.	1. c. iii. Continued from above. If more than half of the GPC calibration check compound recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) all positive detects in all samples associated with that GPC calibration check to indicate potential low bias. - Estimate (UJ) all quantitation limits for non-detects in all samples associated with that GPC calibration check to indicate potential low bias. If a GPC calibration check compound recovery is less than 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that GPC calibration check to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that GPC calibration check to indicate that the data are unusable due to the possibility of false negatives. If more than half of the GPC calibration check compound recoveries are less than 10%, then the validator should: - Estimate (J) all positive detects in all samples associated with that GPC calibration check to indicate potential low bias. - Reject (R) the quantitation limits for all non-detects in all samples associated with that GPC calibration check to indicate that the data are unusable due to the possibility of false negatives. If more than half of the GPC calibration check compound recoveries are outside the method QC acceptance limits in one GPC calibration check, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular compound, class of compounds or the entire fraction for samples associated with that GPC calibration check.

C. EVALUATION	D. ACTION
*1. c. iv. Verify that surrogate compound recoveries and internal standard area counts and/or retention times in the GPC calibration check meet method QC acceptance criteria.	1. c. iv. If surrogate compound recoveries and/or internal standard area counts or retention times in the GPC calibration check do not meet method QC acceptance criteria, then the validator should qualify the sample data in accordance with Sections VI and VII.
* v. Review the raw GPC calibration check data to verify that peaks are symmetrical and resolution meets method QC acceptance criteria for target and surrogate compounds and interferents in the GPC calibration check solution.	v. If the GPC calibration check method QC acceptance criteria do not meet peak shape and compound resolution, then the raw sample data should be examined for the presence of high molecular-weight interferences or the loss of late eluting target compounds and professional judgment should be used to qualify or reject sample data. The validator should discuss the impact of unacceptable peak shape and resolution on the sample data in terms of high or low bias and/or the possibility of false negatives and note this in the Data Validation Memorandum.
* vi. Check the raw GPC calibration check data to verify that retention times for any compounds or interferents in the GPC calibration solution did not vary more than ± 5% between calibrations.	vi. Retention time shifts indicate instrument performance problems that require laboratory corrective actions. If retention time shifts are excessive, the GPC cleanup procedure may be the cause of analyte losses and false negatives, and the validator should evaluate the sample data carefully and document all deficiencies in the Data Validation Memorandum.
* d. i. Verify that a GPC instrument blank was analyzed after each GPC calibration and calibration check and prior to sample analysis.	d. i. If a GPC instrument blank was not analyzed at the correct frequency and in the proper sequence, then the validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
* ii. Verify that there are no target compounds present at greater than or equal to the quantitation limit in the GPC instrument blank.	ii. If any target compounds are detected in the GPC instrument blank at greater than or equal to the quantitation limit, then the quality of the GPC operation is suspect. The validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
* iii. Verify that surrogate compound recoveries and internal standard area counts and/or retention times (if added) in the GPC instrument blank meet method QC acceptance criteria.	iii. If surrogate compound recoveries and/or internal standard area counts or retention times in the GPC instrument blank do not meet method QC acceptance criteria, then the validator should qualify the sample data in accordance with Sections V, VI, and VII.

C. EVALUATION	D. ACTION
*1. e. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred. If result forms are not available, then the validator must review the cleanup logs to confirm that method required cleanups were performed.	1. e. If the laboratory made any calculation and/or transcription errors, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is most accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
f. Review MS/MSD, surrogate, and PES data to evaluate the efficiency of the GPC cleanup.	f. If any compound or compound class has zero recovery indicating the possibility of false negatives and/or recovers low indicating a potential low bias, then the validator should discuss the possible false negatives and/or potential low bias in the Data Validation Memorandum and qualify and/or reject sample results according to the guidance provided in Sections VI, VIII and XI.
2. Silica Gel Cleanup	**2. Silica Gel Cleanup**
a. Verify from result forms, if available, that Silica Gel cleanup was performed according to the analytical method on all method-required sample extracts, QC sample extracts, and method blank extracts.	a. If Silica Gel cleanup was not performed according to the analytical method on all method-required extracts, then the data should be reviewed for the presence of interferents and professional judgment should be used to qualify or reject sample data. The validator should request sample cleanup and reanalysis if Silica Gel cleanup was required by the method.
b. Verify that each lot of Silica Gel used to cleanup samples was checked prior to use in accordance with method requirements.	b. If each lot of Silica Gel was not checked, then the solid phase may not be properly activated potentially resulting in unacceptable target compound recoveries, the presence of interferents and possibly the loss of target compounds (false negatives). The validator should review the Silica Gel Check solution data associated with each batch of Silica Gel column cleanups to ascertain if any target compounds should be qualified or rejected using the guidance provided in Section XV, D.2.c.iii.
c. i. Verify from result forms, if available, that a Silica Gel Check solution was prepared with each batch of samples undergoing Silica Gel cleanup and analyzed prior to the Silica Gel column reagent blank in accordance with the analytical method.	c. i. If the laboratory did not prepare and analyze the Silica Gel check solution at the correct frequency and sequence, according to the method, then the validator should use professional judgment to qualify or reject sample data.

C. EVALUATION	D. ACTION
*2. c. ii. Verify that a Silica Gel Check solution was prepared and analyzed in accordance with the method and that the correct target and surrogate compounds, interferents and concentrations were used.	2. c. ii. If a Silica Gel Check solution was not prepared and analyzed in accordance with the method or the correct compounds and/or concentrations were not used, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.
iii. Check the reported data from the Silica Gel Check solution analyses to verify that target compound recoveries meet method QC acceptance criteria.	iii. If Silica Gel cleanup method QC acceptance criteria are not met, then the Silica Gel Check solution results should be used to qualify sample data for specific compounds included in the check solution. Professional judgment should be used to qualify or reject sample data for non-check solution compounds, taking into consideration the compound's chemical class. The validator should discuss the impact of unacceptable recoveries on the sample data in terms of high or low bias and note this in the Data Validation Memorandum. If a Silica Gel Check solution compound recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that Silica Gel Check solution to indicate potential high bias. - Accept the quantitation limit of the affected compound in any sample associated with that Silica Gel Check solution. If more than half of the Silica Gel Check solution compound recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Silica Gel Check solution to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that Silica Gel Check solution.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. iii. Continued from above If a Silica Gel Check solution compound recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that Silica Gel Check solution to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected compound in any sample associated with that Silica Gel Check solution to indicate potential low bias. If more than half of the Silica Gel Check solution compound recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Silica Gel Check solution to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that Silica Gel Check solution to indicate potential low bias. If a Silica Gel Check solution compound recovery is less than 10%, then the validator should: - Estimate (J) the affected compound when detected in any sample associated with that Silica Gel Check solution to indicate potential low bias. - Reject (R) the quantitation limit of the affected compound in any sample associated with that Silica Gel Check solution to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. c. iii. Continued from above.	2. c. iii. Continued from above. If more than half of the Silica Gel Check solution compound recoveries are less than 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Silica Gel Check solution to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that Silica Gel Check solution to indicate that the data are unusable due to the possibility of false negatives. If more than half of the Silica Gel Check solution compound recoveries are outside the method QC acceptance limits in one Silica Gel Check solution, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular compound, class of compounds or the entire fraction for samples associated with that Silica Gel Check solution.
* iv. Verify that surrogate compound recoveries and internal standard area counts and/or retention times in the Silica Gel Check solution meet method QC acceptance criteria.	iv. If surrogate compound recoveries and/or internal standard area counts or retention times in the Silica Gel Check solution do not meet method QC acceptance criteria, then the validator should qualify the sample data in accordance with Sections VI and VII.
* d. i. Verify that a Silica Gel column reagent blank was prepared with each cleanup batch and was analyzed after the Silica Gel Check solution but prior to field samples.	d. i. If a Silica Gel column reagent blank was not prepared and analyzed at the correct frequency and in the proper sequence, then the validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
* ii. Verify that there are no target compounds present at greater than or equal to the quantitation limit in the Silica Gel column reagent blank.	ii. If any target compounds are detected in the Silica Gel column reagent blank at greater than or equal to the quantitation limit, then the Silica Gel may be contaminated. The validator should evaluate the method blank data and use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject data.

C. EVALUATION	D. ACTION
*2. d. iii. Verify that surrogate compound recoveries and internal standard area counts and/or retention times (if added) in the Silica Gel column reagent blank meet method QC acceptance criteria.	2. d. iii. If surrogate compound recoveries and/or internal standard area counts or retention times in the Silica Gel column reagent blank do not meet method QC acceptance criteria, then the validator should qualify the sample data in accordance with Sections V, VI, and VII.
* e. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred. If result forms are not available, then the validator must review the cleanup logs to confirm that method required cleanups were performed.	e. If the laboratory made any calculation and/or transcription errors, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is most accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
f. Review MS/MSD, surrogate, and PES data to evaluate the efficiency of the Silica Gel cleanup.	f. If any compound or compound class has zero recovery indicating the possibility of false negatives and/or recovers low indicating a potential low bias, then the validator should discuss the possible false negatives and/or potential low bias in the Data Validation Memorandum and qualify and/or reject sample results according to the guidance provided Sections VI, VIII and XI.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.1.c.i, C.1.c.ii, C.1.c.iv, C.1.c.v, C.1.vi, C.1.d.i, C.d.1.ii, C.1.d.iii, C.1.e, C.2.c.ii, C.2.c.iv, C.2.d.i, C.2.d.ii, C.2.d.iii, C.2.e

Table SV-XV-1:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON GPC CALIBRATION QUALITY CONTROL

	Criteria	Action
Peak Resolution	As per method QC acceptance criteria.	Professional Judgment
Peak Shape	Peak shapes must be symmetrical.	Professional Judgment
Retention Time Shift	Retention time shifts between GPC calibration checks must not exceed ± 5%.	Professional Judgment
GPC Instrument Blank	Target analytes must be < QL and surrogate compound recoveries and IS area counts and/or RTs (if added) must meet method QC acceptance criteria. (Note: CLP SOW OLM03.2 does not require the addition of surrogate compounds to the GPC instrument blank)	Refer to Section V for Blank Actions

Table VOA/SV-XI-2:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON GPC CLEANUP QUALITY CONTROL WHERE: ≤ ONE-HALF OF GPC CALIBRATION CHECK COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec < LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table V/SV-XI-3:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON GPC CLEANUP QUALITY CONTROL WHERE: > ONE-HALF OF GPC CALIBRATION CHECK COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

Note: Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table SV-XV-4:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON SILICA GEL CLEANUP QUALITY CONTROL WHERE: ≤ ONE HALF OF SILICA GEL CHECK SOLUTION COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE CRITERIA

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec ≤ LL	LL ≤ %Rec ≤ UL	%Rec > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A
Silica Gel Column Blank	Target analytes must be < QL and surrogate compound recoveries and IS area counts and/or RTs (if added) must meet method QC acceptance criteria.			Refer to Section V for Blank Actions

Note: Professional judgment should be used in applying the guidance above to qualify or reject sample data.

LL - Lower Limit of method QC acceptance criteria.
UL - Upper Limit of method QC acceptance criteria.

Table V/SV-XI-5:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON SILICA GEL CLEANUP QUALITY CONTROL WHERE: > ONE-HALF OF SILICA GEL CHECK SOLUTION COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

Note: Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

E. EXAMPLES

Example #1: (Unacceptable GPC peak resolution and retention time shift)

The validator compares the raw GPC calibration data with CLP SOW OLM03.2 criteria to verify that the proper collection and dump cycles were utilized to ensure that all interferences were removed without loss of target compounds. To do this, the validator reviews the peak shape, resolution, and retention time shift data for the GPC calibration. The validator notes that the calibration retention time shift exceeded the ± 5% criteria. The validator also notes that the baseline resolution between perylene and sulfur is less than 90%. The validator uses professional judgment to estimate (J) the positive detects and reject (R) the quantitation limits for non-detects for all samples associated with the non-compliant GPC calibration. The validator reports the qualified data on the Data Summary Table and discusses the low bias and potential false negatives due to insufficient column resolution and incorrect collect and dump cycles.

Example #2: (Silica Gel Check % recovery > upper limit for one compound)

The validator examines the raw Silica Gel cleanup data to verify that the percent recoveries from the Silica Gel Check meet method-specific QC acceptance criteria of 80-110%. The check solution contains several PAHs at 3 times the method quantitation limit. The validator notes that one of the check solution compounds, phenanthrene, was recovered at 150%. The validator uses professional judgment to estimate (J) the positive phenanthrene detects and accepts (A) the quantitation limits for phenanthrene non-detects on the Data Summary Table. The validator notes in the Data Validation Memorandum that a high bias exists for phenanthrene and that positive results of phenanthrene may actually be lower than the reported results.

XVI. SYSTEM PERFORMANCE

A. OBJECTIVE

The objective of assessing overall system performance is to determine if any method preparatory and/or analytical procedures result in qualitative and/or quantitative system error or bias. All sample, QC sample, and blank results are reviewed for accuracy, chromatography, precision, sensitivity, and contamination to ascertain if there are any general trends in data quality.

B. CRITERIA

Since there are no specific criteria for system performance, professional judgment should be used to assess the overall performance.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
*1. The results of Zero, Single and Double Blind PESs, MDL study, LFB, calibration standards, MS/MSD, and surrogate spike compound analyses may be used to assess the overall system accuracy including purge and extraction efficiency and instrument response. * a. Evaluate all PES and other relevant QC data to determine if any analytical trends exist over the sample analysis period. * b. The validator should ascertain from the PES and other relevant QC data if there is a high or low quantitative bias for a particular compound or group of compounds. * c. The validator should also ascertain from the PES and other relevant QC data if there is a potential for false negatives and/or false positives to be reported. * d. The validator should ascertain from the MS/MSD and surrogate spike compound analyses if the sample matrix effects impact compound recovery, thus indicating a method bias outside the control of the laboratory.	1. The validator should refer to the previous sections for specific guidance on evaluating accuracy using PES, MDL study, LFB, calibration standard, MS/MSD and surrogate data. If the validator determines that analytical trends indicate a qualitative and/or quantitative systematic bias, then the validator should use professional judgment to determine whether or not to qualify or reject the sample data based on the extent of the impact. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should differentiate between sample matrix-related preparatory and analysis problems that are outside the laboratory's control and those preparatory and analysis problems that are within the laboratory's control.

C. EVALUATION	D. ACTION
*2. The results of the PES, LFB and calibration standard analyses as well as field samples may be used to assess the overall system chromatography. * a. Evaluate sample and QC sample reconstructed ion chromatograms analyzed on all columns to determine if the column chromatography, peak shape, resolution, and baseline drift has either deteriorated or improved over the sample analysis period. * b. The validator should ascertain from the raw data if unacceptable chromatography may contribute to a high or a low quantitative bias for a particular compound or group of compounds. * c. The validator should also ascertain from the raw data if unacceptable chromatography may result in a potential for false negative and/or false positive identifications. * d. The validator should determine if chromatography problems are a result of the sample matrix or are unique to the instrument. To that end, the validator should review the data package narrative for a discussion of possible matrix problems that the laboratory may have encountered. * e. The validator should determine if significant retention time shifts have occurred between initial and continuing calibration.	2. The validator should refer to the previous sections for specific guidance on evaluating compound identification and quantitation. If the validator determines that chromatographic trends indicate a qualitative and/or quantitative systematic bias, then professional judgment should be used to determine whether or not to qualify or reject the sample data based on the extent of the impact. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should especially note when chromatography problems and column degradation are caused by severe matrix interferences. The validator should recommend additional cleanup procedures and/or alternate analytical methods for future site work.

C. EVALUATION	D. ACTION
*3. The results of the calibration standard, MDL study, internal standard, surrogate spike compound, MS/MSD, and field duplicate analyses may be used to assess overall system precision. * a. Compare the daily standard calibration area counts to ascertain if the instrument generated consistent detector responses over the sample analysis period. * b. Review the area counts of the internal standards and surrogate compounds for each sample to ascertain if there is a change in detector response. * c. The validator should evaluate the MS/MSD RPDs in conjunction with field duplicate RPDs to identify any analytical trends, ascertain if sample matrices were homogeneous or heterogeneous, and determine if sampling error may have contributed to field imprecision.	3. The validator should refer to the previous sections for specific guidance on evaluating laboratory and field precision and internal standard and surrogate compound analyses. If the validator determines that an instrument produces erratic detector responses, then they should use professional judgment to qualify or reject sample data. If MS/MSD RPDs indicate laboratory imprecision, then the validator should suspect laboratory technique and take into consideration the results of the field duplicate RPDs when using professional judgment to qualify sample data. If field duplicate RPDs indicate field imprecision resulting from heterogeneous sample matrices or field sampling error, then the validator should use professional judgment to qualify sample data based on the extent of impact. The validator should differentiate between lack of precision due to instrument performance problems and that caused by matrix effects or sampling error.

C. EVALUATION	D. ACTION
*4. The results of the LFB, PES, calibration and internal standard analyses may be used to assess the overall system sensitivity. (Note: VOA surrogates may also be used because they are equivalent to internal standards.) * a. Review all daily LFBs, low level calibration standards, and PES data to evaluate sensitivity for each instrument to verify that no instrument has lost its ability to accurately quantitate and identify compounds at the quantitation limit over the sample analysis period, which could potentially result in false negatives and low biased results. * b. Check the area counts of the individual sample, QC sample, calibration and blank internal standards and calibration standards to monitor instrument sensitivity changes. * c. Review the sample chromatograms for abrupt, discrete shifts in the chromatographic baseline which may indicate a change in the instrument's sensitivity or the zero setting. A baseline "decline" could indicate a decrease in sensitivity in the instrument or an increase in the instrument zero, possibly causing target compounds, at or near the detection limit, to miss detection (false negatives). Additionally, a decline in the baseline may result in incorrect peak integration and subsequent misquantitation. A sudden baseline shift could indicate problems such as a change in the instrument zero, a leak, degradation of the column or the formation of matrix degradation products. The validator should check for any abrupt shift in the zero setting which may cause a false positive to be reported. Additionally, a rise in the baseline may result in incorrect peak integration and subsequent misquantitation. * d. The validator may determine that instrument sensitivity is adequate but sample matrix effects may preclude obtaining the quantitation limits required by the project DQOs using the analytical method employed.	4. The validator should refer to the previous sections for specific guidance on evaluating sensitivity, accuracy, compound identification, and quantitation. If the validator determines that instrument sensitivity is unacceptable, then the validator should use professional judgment to qualify or reject the affected sample data. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should also note if sample matrix interferences did not allow quantitation limits to be achieved and should recommend additional cleanup procedures and/or alternate analytical methods for future site work.

C. EVALUATION	D. ACTION
*5. The results of the PES and method, instrument, cleanup, equipment/rinsate, trip, storage and bottle blank analyses may be used to assess overall system contamination. * a. Review all blank and sample results to evaluate the possibility of sample contamination introduced via either cross-contamination from a previously run sample or from general lab contamination. * b. Compare blank analysis on two different instruments to determine if the contamination is instrument related or the interferents are present in the blank from sample processing activities. * c. Assess whether problematic blank results are reproducible when replicate aliquots are analyzed or are sporadic interferences. Sporadic interferences, such as methylene chloride, acetone or phthalates, may indicate that the interferent is introduced from the laboratory environment. The validator should review sample chromatograms for suspected outlier interferents.	5. The validator should refer to the previous sections for specific guidance on evaluating blank contamination. If the validator determines that there is a systematic blank error introduced during sample collection or processing (extraction or analysis), then the data should be qualified according to Section V. However, if the validator suspects intermittent or sporadic introduction of interferents during analysis, then the validator should use professional judgment to qualify or reject sample data and document and justify all technical decisions in the Data Validation Memorandum.

* **Note:** **This section is only applicable to a Tier III data validation - If a validator suspects system performance has degraded to the degree that data are affected and a Tier II validation has been requested, then the validator should contact the Site Manager to approve the necessary Tier III validation.**

E. EXAMPLES

Example #1: (Abrupt decrease in baseline)

The validator notices a significant abrupt decrease in the baseline during the analysis of aqueous sample SAP55. The validator examines the IS area counts and observes that a decrease in the area counts for the last two internal standards has occurred. The validator notes that the VOA surrogate compound areas for the last two surrogates also decreased. There were no PE samples associated with these samples available for review. The validator uses professional judgment to estimate (J) all positive detects associated with the two problematic internal standards and rejects (R) all non-detects associated with the two problematic internal standards. The validator reports the qualified data on the Data Summary Table. The validator notes the sensitivity loss of the GC/MS instrument and justifies the decision to qualify sample data in the Data Validation Memorandum.

Example #2: (Peak broadening and tailing for volatile gases; PES quantitation low for 1 volatile gas)

The validator reexamines the Reconstructed Ion Chromatograms from packed column analysis and notices peak broadening and tailing of the following volatile gases: vinyl chloride, chloromethane, bromoethane, and chloroethane. The PE sample results were reviewed and found to have an "Action Low" qualification for vinyl chloride which was the only volatile gas included in the PES. The validator uses professional judgment to estimate (J) all positive volatile gas detects in all samples associated with that PES, and to estimate (UJ) the quantitation limits for all volatile gas non-detects in all samples associated with that PES. The validator reports the qualified data on the Data Summary Table. The validator notes the GC/MS chromatography problem and justifies the decision to qualify sample data in the Data Validation Memorandum.

XVII. OVERALL EVALUATION OF DATA

A. OBJECTIVE

The objective of the final evaluation of a data package is to identify the "analytical error" and any "sampling error" associated with the data. The sum of the "analytical error" and the "sampling error" equals the "measurement error". "Measurement error" will then be used by the end user in conjunction with sampling variability (spatial variations in pollutant concentrations) to determine "total error" (total uncertainty) associated with the data. Ultimately, the end data user will assess data usability in the context of the pre-determined Data Quality Objectives (DQOs) and resultant "total error" of the data.

B. CRITERIA

The Sampling and Analysis Plan (SAP) or Quality Assurance Project Plan (QAPjP) and DQO Summary Form should specify the site specific DQOs and acceptable levels of uncertainty or "total error".

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
1. Obtain the SAP, QAPjP or DQO Summary Form to review the DQOs for the sampling event.	1. Synopsize in the first section of the Data Validation Memorandum, Overall Evaluation of Data, in bullet format, the appropriate project DQOs for the data package.
2. Evaluate the appropriateness of the analytical method chosen. For example, was the method capable of achieving quantitation limits sufficiently low to meet DQOs for risk assessment? Was the method capable of successfully analyzing each particular matrix sampled?	2. If an inappropriate method was chosen for sample analysis, then the validator should discuss the method deficiencies and identify more appropriate methods or modifications for use in subsequent sampling rounds. The validator should include this discussion in the Overall Evaluation of Data Section of the Data Validation Memorandum.
3. Evaluate any analytical problems that were identified.	3. Estimate and describe the "analytical error" that contributes to the "measurement error" associated with the data package in the Overall Evaluation of Data Section of the Data Validation Memorandum. a. If "analytical error" causes the data to be unusable, then the validator should reject the data and return it to the laboratory and deny payment. b. If "analytical error" causes the data to be of reduced worth to the Region, then the validator should recommend that the laboratory's payment be reduced.

C. EVALUATION	D. ACTION
4. Evaluate any sampling issues that were identified. Note: The validator is only responsible for evaluating those "sampling errors" that are identified during the routine data validation process. Other "sampling errors" may have occurred and they should be assessed by the end user prior to data use.	4. Estimate and describe the "sampling error" that contributes to the "measurement error" associated with the data package in the Overall Evaluation of Data Section of the Data Validation Memorandum. Examples of "sampling error" for which the validator would have information include highly contaminated trip or equipment blanks as well as delayed sample shipment that caused holding time violations. a. If "sampling error" severely impacts potential data usability, then the validator should note this in the Data Validation Memorandum. b. The end user should review the results of the sampler's field notes/trip report to determine additional "sampling error" issues with which to fully assess "measurement error".
5. Evaluate data quality in terms of "measurement error" as a combination of "analytical error" and "sampling error".	5. Discuss data quality in terms of "measurement error" as the sum of "analytical error" and "sampling error". All discussions should be included in the Overall Evaluation of Data Section of the Data Validation Memorandum.
6. Identify potential usability issues raised by an unacceptable degree of "measurement error".	6. If data usability is potentially compromised by a high degree of "measurement error", then the validator should note this in the Overall Evaluation of Data section of the Data Validation Memorandum. If data quality impacts the use of those data by the end user, then the validator should detail in the Overall Evaluation of Data Section of the Data Validation Memorandum how data use will be limited and for which end user, i.e., risk assessor, hydrogeologist, etc..
7. Sampling variability is not assessed during data validation, and therefore, should be assessed by the end user prior to data use.	7. The end user should review the results of the Data Validation Memorandum in conjunction with the sampler's field notes/trip report to assess the impact of sampling variability issues on data usability.

APPENDICES

The following appendices were developed to assist the validator in volatile and semivolatile data validation. The appendices include method-specific Quality Control requirements for the following QC parameters: preservation and technical holding times, GC/MS instrument performance check (tuning), initial and continuing calibrations, blanks, surrogate compounds, internal standards, matrix spike/matrix spike duplicates, field duplicates, sensitivity check, PE samples/accuracy check, target compound identification, compound quantitation and reported quantitation limits, tentatively identified compounds, semivolatile cleanup, system performance, and overall evaluation of data.

Appendices are included for the following methods:

Appendix A:	CLP SOW OLM03.2/Volatile Organic Analysis
Appendix B:	CLP SOW OLM03.2/Semivolatile Organic Analysis
Appendix C:	CLP SOW OLC02.1/Low Concentration Volatile Organic Analysis
Appendix D:	CLP SOW OLC02.1/Low Concentration Semivolatile Organic Analysis

Additional appendices for other methods may be added as required.

Appendix E: VOA/SV Functional Guidelines Action Tables

Appendix A

CLP/SOW OLM03.2/Volatile Organic Analysis
Method QC criteria, Equations, and Definitions

APPENDIX A

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM03.2, Exhibit D Volatiles**.

SECTION I: PRESERVATION & TECHNICAL HOLDING TIME CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-I-B for preservation and technical holding time data validation criteria.

SECTION II: GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING) CRITERIA

Refer to the following method GC/MS instrument performance (tuning) QC criteria for data validation:

The analysis of the instrument performance (tuning) check solution (50 ng BFB on column) must be performed at the beginning of each **12-hour** period during which samples or standards are analyzed. The tuning check, bromofluorobenzene (BFB), for volatile analysis must meet the ion abundance criteria given below:

m/z	ION ABUNDANCE CRITERIA
50	8.0 - 40.0% of m/z 95
75	30.0 - 66.0% of m/z 95
95	Base Peak, 100% Relative Abundance
96	5.0 - 9.0% of m/z 95 (see note)
173	Less than 2.0% of m/z 174
174	50.0 - 120.0% of m/z 95
175	4.0 - 9.0% of mass 174
176	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120.0% that of m/z 95.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Part of the BFB peak must not be background subtracted.

SECTION III: INITIAL CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

The initial calibration standards must be analyzed upon contract award, whenever corrective action is taken which may change or affect the initial calibration criteria or if the continuing calibration acceptance criteria have not been met. Initial calibrations must be analyzed after the analysis of a compliant instrument performance check.

The initial calibration standards must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

All initial calibration standards must be analyzed at the following concentration levels: 10, 20, 50, 100, 200 ug/L (unheated for aqueous and medium level soils/heated for low level soils).

Note: The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.

RELATIVE RESPONSE FACTOR (RRF) - A measure of the relative mass spectral response of an analyte compared to its' internal standard. The RRF is calculated using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of primary quantitation ion response (EICP) for the internal standard
C_{is} = Concentration of the internal standard
C_x = Concentration of the compound to be measured

AVERAGE (MEAN) RELATIVE RESPONSE FACTOR (RRF) - The average or mean RRF is determined by the analysis of five different standard concentrations and is used in calculating compound concentrations in samples. The RRF is calculated using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{RRF_i}{n}$$

Where,

RRF_i = The individual RRFs for various concentration levels
n = The number of RRFs

PERCENT RELATIVE STANDARD DEVIATION (%RSD) - The % RSD for each compound is a measure of the linearity of the calibration curve. The % RSD is calculated using the following equation:

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

Where,

$$Standard\ Deviation = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \overline{x})^2}{(n-1)}}$$

$\overline{x}$ = Mean
n = total number of values
x_i= each individual value used to calculate the mean

SECTION IV: CONTINUING CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IV-B for continuing calibration data validation criteria and the following method continuing calibration QC criteria:

The continuing calibration standard must be analyzed once every 12 hours, following the analysis of a compliant instrument performance check and initial calibration, and prior to the analysis of field samples, QC samples and blanks.

The continuing calibration standard must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

Continuing calibration standards must be analyzed at a concentration level of 50 ug/L (unheated for aqueous and medium level soils/heated for low level soils).

Note: The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.

PERCENT DIFFERENCE (%D) - The % D is used to compare the initial calibration RRF with the continuing calibration RRF50. The % Difference indicates both the direction and the magnitude of the comparison, i.e., the % Difference may be either negative, positive or zero.

$$\%\ Difference = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

RRF_c = Relative response factor from continuing calibration standard

SECTION V: BLANK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-V-B for blank validation criteria and the following method QC criteria:

Method Required Blanks

1. Method Blank - A 5.0 mL aliquot of reagent water or purified solid matrix approximate in weight or volume to the samples that is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. The method blank must be analyzed at least once during every 12 hour time period on each GC/MS system used for volatile analysis.

2. Storage Blank - Consists of two 40 mL VOA vials filled with reagent water prepared by the laboratory when the first samples in an SDG are received. The vials are stored, under the same conditions, with the field samples. After all of the samples in the SDG are analyzed, a 5.0 mL aliquot of the storage blank is analyzed to determine whether contamination was introduced during storage of the samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. A minimum of one storage blank must be analyzed per SDG after all samples for that SDG have been analyzed.

3. Instrument Blank - A 5.0 mL aliquot of reagent water that is carried through the entire analytical procedure and is analyzed following highly contaminated samples containing target compounds that exceed the initial calibration range. The instrument blanks are used to determine if contamination is introduced by a previous sample and the level associated with the analytical instrument. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. An instrument blank must be analyzed after a sample that exceeds the calibration range. Until an instrument blank meets the technical acceptance criteria, the

system is considered contaminated.

SECTION VI: SURROGATE COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VI-B for surrogate compound data validation criteria and the following method surrogate compound QC criteria:

The proper surrogate compounds must be quantified using correctly assigned internal standards and the correct primary quantitation ions.

10 uL of a 25 ug/mL solution of surrogate compounds Toluene-d_8, 4-Bromofluorobenzene, 1,2-Dichloroethane-d_4 are added to 5 mL/5 g of sample, standard, QC sample, or blank for a final concentration of 50 ug/L or 50 ug/kg. 10 uL of a 25 ug/mL solution of internal standards Chlorobenzene-d_5 and Bromochloromethane are added to 5 mL/5 g of sample for a final concentration of 50 ug/L or 50 ug/kg. 10 uL of a 25 ug/mL solution of surrogate compounds Toluene-d_8, 4-Bromofluorobenzene, 1,2-Dichloroethane-d_4 are added to 5 mLs of reagent water containing an aliquot of the methanol medium level soil/sediment extract.

Table App.A.VI-1 - CHARACTERISTIC IONS FOR SURROGATE COMPOUNDS

Surrogate	**Characteristic Ions**		**Internal Standard**
	Primary Quantitation Ion	**Secondary Ion(s)**	
Toluene-d_8	98	70, 100	Chlorobenzene-d_5
4-Bromofluorobenzene	95	174, 176	Chlorobenzene-d_5
1,2-Dichloroethane-d_4	65	102	Bromochloromethane

The surrogate % recovery is calculated using the following equation:

$$\text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of surrogate determined by analysis

Q_a = Quantity of surrogate added to sample/blank

Table App.A.VI-2 - SURROGATE RECOVERY LIMITS

Surrogate	**Method QC Criteria**	
	Percent Recovery (Water)	**Percent Recovery (Soil/Sediment)**
Toluene-d_8	88-110	84-138
Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d_4	76-114	70-121

If one or more sample surrogate recovery does not meet the acceptance criteria, the sample must be reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Reanalysis is not required if the sample is a QC sample and both the matrix spike and matrix spike duplicate surrogate recoveries failed to meet the acceptance criteria. If the sample was reanalyzed and the surrogate recovery(ies) was acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the reanalysis also recovers the surrogate(s) outside of the acceptance limits, then both analyses should have been submitted.

SECTION VII: INTERNAL STANDARDS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VII-B for internal standard data validation criteria and the following method internal standard QC criteria:

The correct internal standard must be used for sample compound quantitation and the correct internal standard primary quantitation ion must be used for quantitation.

10 uL of a 25 ug/mL solution of internal standards Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d_5 are added to 5 mL/5 g of sample, standard, QC sample, or blank for a final concentration of 50 ug/L or 50 ug/kg.

Table App.A.VII-1 - VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

IS Bromochloromethane	IS 1,4)Difluorobenzene	IS Chlorobenzene)d5
Chloromethane	1,1,1)Trichloroethane	2)Hexanone
Bromomethane	Carbon Tetrachloride	4)Methyl)2)Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2)Dichloropropane	1,1,2,2)Tetrachloroethane
Methylene Chloride	trans)1,3)Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1)Dichloroethene	1,1,2)Trichloroethane	Styrene
1,1)Dichloroethane	Benzene	Xylene (total)
1,2)Dichloroethene(tot.)	cis)1,3)Dichloropropene	4-Bromofluorobenzene (surr)
Chloroform	Bromoform	Toluene-d8 (surr)
1,2)Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d4 (surr)		

Table App.A.VII-2 - CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR VOLATILE COMPOUNDS

Internal Standard	Characteristic Ions	
	Primary Quantitation Ion	**Secondary Ion(s)**
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d5	117	82, 119

Internal standard area counts for each of the internal standards must be within the inclusive range of -50.0% and +100.0% of the response of internal standards in the associated daily continuing calibration standard.

The retention time of the internal standard must not vary by more than ± 30 seconds from the retention time of the associated daily continuing calibration standard.

If one or more internal standard area count and/or retention time does not meet the acceptance criteria, then the sample must be reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Reanalysis is not required if the sample is a QC sample and both the matrix spike and matrix spike duplicate failed to meet the internal standard acceptance criteria. If the sample was reanalyzed and the internal standard area counts and/or retention times were acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the reanalysis also recovers the internal standard outside of the area count and/or retention time acceptance criteria, then both analyses should have been submitted.

SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VIII-B for MS/MSD data validation criteria and the following method MS/MSD QC criteria:

The MS/MSD spike compounds listed below are spiked at 10 uL in a 25 ug/mL solution into the 5 mL or 5 g sample for a final concentration of 50 ug/L or 50 ug/kg.

A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix for each SDG, or each matrix within an SDG or each group of samples of a similar concentration level (soils only) whichever is most frequent. The following advisory matrix spike compound recoveries and RPDs are listed below:

Table App.A.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	Method QC Criteria			
	Water		Soil/Sediment	
	% Recovery*	RPD**	% Recovery	RPD
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

*The MS/MSD % recovery is calculated using the following equation:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

**The MS/MSD relative percent difference (RPD) is calculated using the following equation:

$$\text{Relative Percent Difference} = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)} \times 100$$

Where,

MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

Note: The vertical bars in the formula indicate the absolute value of the difference, hence RPD is always positive.

SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IX-B for field duplicate data validation criteria.

SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-X-B for sensitivity check data validation criteria.

SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XI-B for accuracy check data validation criteria.

SECTION XII: TARGET COMPOUND IDENTIFICATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XII-B for target compound identification data validation criteria.

SECTION XIII: COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIII-B for compound quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Volatile target compounds must be quantitated using the internal standard method with the internal standards assigned in Appendix A, Section VII. The daily RRF50 must be used for sample quantitation. The sample target compounds must be quantified using the following primary quantitation ions and must be reported to the CRQLs listed below.

Table App.A.XIII-1 - TARGET COMPOUND LIST (TCL), PRIMARY QUANTITATION AND SECONDARY IONS, AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR OLMO3.1 SOW VOLATILE ORGANIC COMPOUNDS

		Quantitation Limits				Characteristic Ions	
Volatiles	CAS Number	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)	Primary	Secondary
Chloromethane	74)87)3	10	10	1200	(50)	50	52
Bromomethane	74)83)9	10	10	1200	(50)	94	96
Vinyl Chloride	75)01)4	10	10	1200	(50)	62	64
Chloroethane	75)00)3	10	10	1200	(50)	64	66
Methylene Chloride	75)09)2	10	10	1200	(50)	84	49,51,86
Acetone	67)64)1	10	10	1200	(50)	43	58
Carbon Disulfide	75)15)0	10	10	1200	(50)	76	78
1,1)Dichloroethene	75)35)4	10	10	1200	(50)	96	61,98
1,1)Dichloroethane	75)34)3	10	10	1200	(50)	63	65,83,85,98,100
1,2)Dichloroethene (total)	540)59)0	10	10	1200	(50)	96	61,98
Chloroform	67)66)3	10	10	1200	(50)	83	85
1,2)Dichloroethane	107)06)2	10	10	1200	(50)	62	64,100,98
2)Butanone	78)93)3	10	10	1200	(50)	43*	57
1,1,1)Trichloroethane	71)55)6	10	10	1200	(50)	97	99,117,119
Carbon Tetrachloride	56)23)5	10	10	1200	(50)	117	119,121
Bromodichloromethane	75)27)4	10	10	1200	(50)	83	85
1,2)Dichloropropane	78)87)5	10	10	1200	(50)	63	65,114
cis)1,3)Dichloropropene	10061)01)5	10	10	1200	(50)	75	77

* - m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

Table App.A.XIII-1 - TARGET COMPOUND LIST (TCL), PRIMARY QUANTITATION AND SECONDARY IONS, AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR OLMO3.1 SOW VOLATILE ORGANIC COMPOUNDS (CONT.)

		Quantitation Limits					
Volatiles	CAS Number	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)	Characteristic Ions Primary	Secondary
Trichloroethene	79)01)6	10	10	1200	(50)	130	95,97,132
Dibromochloromethane	124)48)1	10	10	1200	(50)	129	208,206
1,1,2)Trichloroethane	79)00)5	10	10	1200	(50)	97	83,85,99,132,134
Benzene	71)43)2	10	10	1200	(50)	78	--
trans)1,3)Dichloropropene	10061)02)6	10	10	1200	(50)	75	77
Bromoform	75)25)2	10	10	1200	(50)	173	171,175,250,252,254,256
4)Methyl)2)pentanone	108)10)1	10	10	1200	(50)	43	58,100
2)Hexanone	591)78)6	10	10	1200	(50)	43	58,57,100
Tetrachloroethene	127)18)4	10	10	1200	(50)	164	129,131,166
Toluene	108)88)3	10	10	1200	(50)	91	92
1,1,2,2)Tetrachloroethane	79)34)5	10	10	1200	(50)	83	85,131,133,166
Chlorobenzene	108)90)7	10	10	1200	(50)	112	114
Ethylbenzene	100)41)4	10	10	1200	(50)	106	91
Styrene	100)42)5	10	10	1200	(50)	104	78,103
Xylenes (Total)	1330)20)7	10	10	1200	(50)	106	91

SAMPLE CONCENTRATION - The amount of analyte present in a sample is calculated using the RRF50 of the continuing calibration standard in the following equations:

Sample concentration for water:

$$ug/L = \frac{(A_x)(IS)(Df)}{(A_{is})(RRF)(V_o)}$$

Sample concentration for low level soil/sediment:

$$ug/Kg\ (Dry\ weight\ basis)' \frac{(A_x)(IS)}{(A_{is})(RRF)(W_s)(D)}$$

Sample concentration for medium level soil/sediment:

$$ug/Kg\ (Dry\ weight\ basis)' \frac{(A_x)(IS)(V_t)(1000)(Df)}{(A_{is})(RRF)(V_a)(W_z)(D)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor from the (Ambient temperature purge for water and medium level soil/sediment and heated purge for low level soil/sediment) calibration standard
V_o = Volume of water purged in milliliters (mL)
Df = Dilution Factor - The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. If no dilution is performed , Df=1.
The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

W_s = Weight of the sample added to the purge tube in grams (g)
D = $\frac{100 - \%\ Moisture}{100}$
V_t = Total volume of the methanol extract in milliliters (mL)
V_a = Volume of aliquot of the methanol extract in microliters (uL) added to reagent water for purging
W_z = Weight of soil/sediment extracted in grams (g)

CRQL CALCULATIONS

Water:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(V_x)}{(V_o)}\ x\ (Df)$$

Where,

V_o and Df are defined in the sample concentration equation above
V_x = Contract sample volume (5 mL)

Soil/Sediment-Low:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(W_x)}{(W_s)(D)}$$

Where,

W_s and D are defined in the sample concentration equation above
W_x = Contract sample weight (5 g)

Soil/Sediment-Med:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(W_x)(V_t)(V_y)(Df)}{(W_s)(V_c)(V_a)(D)}$$

Where,

V_t, Df, W_s, V_a, and D are defined in the sample concentration equation above
W_x = Contract sample weight (4 g)
V_y = Contract soil aliquot volume from soil methanol extract (100 uL)
V_c = Contract soil methanol extract volume (10,000 uL)

SECTION XIV: TENTATIVELY IDENTIFIED COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIV-B for tentatively identified compound (TIC) data validation criteria and the following method

TIC QC criteria:

The validator is required to report up to 30 TICs in the Data Validation Memorandum.

TENTATIVELY IDENTIFIED COMPOUND CONCENTRATION - The estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method using the following equations:

Sample concentration for water:

$$ug/L = \frac{(A_x)(IS)(Df)}{(A_{is})(RRF)(V_o)}$$

Sample concentration for low level soil/sediment:

$$ug/Kg = \frac{(A_x)(IS)}{(A_{is})(RRF)(W_s)(D)}$$

Sample concentration for medium level soil/sediment:

$$ug/Kg = \frac{(A_x)(IS)(V_t)(1000)(Df)}{(A_{is})(RRF)(V_a)(W_z)(D)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the non-target compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor assumed to be 1
V_o = Volume of water purged in milliliters (mL)
Df = Dilution Factor - The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. If no dilution is performed , Df=1. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

W_s = Weight of sample added to the purge tube in grams (g)
D = $\frac{100 - \%\ \text{Moisture}}{100}$
V_t = Total volume of the methanol extract in milliliters (mL)
V_a = Volume of aliquot of the methanol extract in microliters (uL) added to reagent water for purging
W_z = Weight of soil/sediment extracted in grams (g)

SECTION XV: SEMIVOLATILE CLEANUP CRITERIA

Not applicable to volatile analysis.

SECTION XVI: SYSTEM PERFORMANCE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVI-B for system performance data validation criteria.

SECTION XVII: OVERALL ASSESSMENT CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVII-B for overall assessment data validation criteria.

Appendix B

CLP SOW OLM03.2/ Semivolatile Organic Analysis Method QC criteria, Equations, and Definitions

APPENDIX B

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM03.2, Exhibit D Semivolatiles**.

SECTION I: PRESERVATION & TECHNICAL HOLDING TIME CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-I-B for preservation and technical holding time data validation criteria.

SECTION II: GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING) CRITERIA

Refer to the following method GC/MS instrument performance (tuning) QC criteria for data validation:

The analysis of the instrument performance (tuning) check solution (50 ng DFTPP on column) must be performed at the beginning of each **12-hour** period during which samples or standards are analyzed. The tuning check, decafluorotriphenylphosphine (DFTPP), for semivolatile analysis must meet the ion abundance criteria given below:

m/z	ION ABUNDANCE CRITERIA
51	30.0 - 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 - 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base Peak, 100% Relative Abundance (see note)
199	5.0 - 9.0% of m/z 198
275	10.0 - 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 - 110.0% of m/z 198
443	15.0 - 24.0% of m/z 442

Note: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110% that of m/z 198.

The mass spectrum of DFTPP must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Part of the DFTPP peak must not be background subtracted.

SECTION III: INITIAL CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

The initial calibration standards must be analyzed upon contract award, whenever corrective action is taken which may change or affect the initial calibration criteria or if the continuing acceptance criteria have not been met. Initial calibrations must be analyzed after the analysis of a compliant instrument performance check.

The initial calibration standards must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

2 uL of the initial calibration standard must be injected and all initial calibration compounds and system monitoring compounds must be analyzed at the following concentration levels; 10, 20, 40, 60, 80 ng/uL. However, the following eight compounds only require a four point calibration and are injected at concentration levels of 50, 80, 120, and 160 ng/uL: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol since detection at less than 50 ng per injection is difficult.

Note: The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.

RELATIVE RESPONSE FACTOR (RRF) - A measure of the relative mass spectral response of an analyte compared to its' internal standard. The RRF is calculated using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of primary quantitation ion response (EICP) for the internal standard
C_{is} = Amount of the internal standard injected (ng)
C_x = Amount of the compound to be measured injected (ng)

AVERAGE (MEAN) RELATIVE RESPONSE FACTOR ($\overline{RRF}$) - The average or mean RRF is determined by the analysis of five different standard concentrations and is used in calculating a compound concentration in samples. The RRF is calculated using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{RRF_i}{n}$$

Where,

RRF_i = The individual RRFs for various concentration levels
n = The number of RRFs

PERCENT RELATIVE STANDARD DEVIATION (%RSD) - The % RSD for each compound is a measure of the linearity of the calibration curve. The % RSD is calculated using the following equation:

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

Where,

$$Standard\ Deviation = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \overline{x})^2}{(n-1)}}$$

$\overline{x}$ = Mean
n = total number of values
x_i = each individual value used to calculate the mean

SECTION IV: CONTINUING CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IV-B for continuing calibration data validation criteria and the following method continuing calibration QC criteria:

The continuing calibration standard must be analyzed once every 12 hours, following the analysis of a compliant instrument performance check and initial calibration, and prior to the analysis of field samples, QC samples and blanks.

The continuing calibration standard must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

Continuing calibrations must be analyzed at a concentration level of 50 ng/2 uL except for the following eight compounds which are analyzed at a concentration level of 100 ng/2 uL: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol since detection at less than 50 ng per injection is difficult.

Note: The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.

PERCENT DIFFERENCE (%D) - The % D is used to compare the initial calibration $\overline{RRF}$ with the continuing calibration RRF50. The % Difference indicates both the direction and the magnitude of the comparison, i.e., the % Difference may be either negative, positive or zero.

$$\% \, Difference = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where,

$\overline{RRF}_i$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

RRF_c = Relative response factor from continuing calibration standard

SECTION V: BLANK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-V-B for blank data validation criteria and the following method QC criteria:

Method Required Blank

Method Blank - A volume of reagent water or purified solid matrix approximate in weight or volume to the samples which is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of the samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. Method blank extraction and analysis must be performed once per each SDG, or each 20 samples in an SDG, or whenever samples are extracted by the same procedure, whichever is most frequent, and analyzed on each GC/MS system used to analyze associated samples.

SECTION VI: SURROGATE COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VI-B for surrogate compound data validation criteria and the following method surrogate compound QC criteria:

The proper surrogate compounds must be quantified using correctly assigned internal standards and the correct primary quantitation ions.

Surrogate compounds Nitrobenzene-d_5, 2-Fluorobiphenyl, Terphenyl-d_{14}, and 1,2-Dichlorobenzene-d_4 (advisory) are added to all samples, standards, QC samples, and blanks at a concentration of 100 ug/mL and surrogate compounds Phenol-d_5, 2-Fluorophenol, 2,4,6-Tribromophenol, and 2-Chlorophenol-d_4 (advisory) are added to all samples, standards, QC samples, and blanks at a concentration of 150 ug/mL.

Table App.B.VI-1 - CHARACTERISTIC IONS FOR SURROGATE COMPOUNDS

Surrogate	Characteristic Ions		Internal Standard
	Primary Quantitation Ion	Secondary Ion(s)	
Nitrobenzene-d_5	82	128, 54	Naphthalene-d_8
2-Fluorobiphenyl	172	171	Acenaphthene-d_{10}
Terphenyl-d_{14}	244	122, 212	Chrysene-d_{12}
Phenol-d_5	99	42, 71	1,4-Dichlorobenzene-d_4
2-Fluorophenol	112	64	1,4-Dichlorobenzene-d_4
2,4,6-Tribromophenol	330	332, 141	Phenanthrene-d_{10}
2-Chlorophenol-d_4 (Recovery limits advisory)	132	68, 134	1,4-Dichlorobenzene-d_4
1,2-Dichlorobenzene-d_4 (Recovery limits advisory)	152	115, 150	1,4-Dichlorobenzene-d_4

The % surrogate recovery is calculated using the following equation:

$$\textit{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of surrogate determined by analysis

Q_a = Quantity of surrogate added to sample/blank

Table App.B.VI-2 - SURROGATE RECOVERY LIMITS

Surrogate	Method QC Criteria	
	Percent Recovery (Water)	**Percent Recovery (Soil/Sediment)**
Nitrobenzene-d_5 (B/N)	35-114	23-120
2-Fluorobiphenyl (B/N)	43-116	30-115
Terphenyl-d_{14} (B/N)	33-141	18-137
Phenol-d_5 (Acid)	10-110	24-113
2-Fluorophenol (Acid)	21-110	25-121
2,4,6-Tribromophenol (Acid)	10-123	19-122
2-Chlorophenol-d_4 (Acid)	33-110 (advisory)	20-130 (advisory)
1,2-Dichlorobenzene-d_4 (B/N)	16-110 (advisory)	20-130 (advisory)

If two or more acid or base neutral surrogate compounds fail to meet their recovery acceptance criteria, the laboratory should check calculations, sample preparation logs, the surrogate compound spiking solutions, and the instrument operation. If sample surrogate recoveries do not meet the acceptance criteria, as a result of the above mentioned problems or other unknown problems, the sample should be re-extracted and reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Re-extraction and reanalysis are not required if the sample is a QC sample and both the matrix spike and matrix spike duplicate surrogate recoveries failed to meet the acceptance criteria. Reanalysis is required if the failed surrogate recoveries are the result of instrument malfunction. If the sample was re-extracted and reanalyzed and the surrogate recoveries were acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the re-extracted/reanalyzed sample also recovers the surrogates outside of the acceptance limits, then both analyses should have been submitted.

SECTION VII: INTERNAL STANDARDS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VII-B for internal standard data validation criteria and the following method internal standard QC criteria:

The correct internal standard and the correct internal standard primary quantitation ion must be used for sample compound quantitation.

The internal standard compounds listed below are added into all samples, standards, QC samples, and blanks at a concentration of 40 ng/2 uL.

Table App.B.VII-1 - SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

IS 1,4–Dichlorobenzene–d4	IS Naphthalene–d8	IS Acenaphthene–d10	IS Phenanthrene–d10	IS Chrysene–d12	IS Perylene–d12
Phenol	Nitrobenzene	Hexachlorocyclo–pentadiene	4,6–Dinitro–2–methylphenol	Pyrene	Di–n–octyl-phthalate
bis(2–Chloroethyl) ether	Isophorone	2,4,6–Trichloro–phenol	N–nitroso-di–phenylamine	Butylbenzyl-phthalate	Benzo(b)fluor–anthene
2–Chlorophenol	2–Nitrophenol	2,4,5–Trichloro–phenol	4–Bromophenyl-phenolether	3,3'–Dichloro–benzidine	Benzo(k)fluor–anthene
1,3–Dichlorobenzene	2,4–Dimethyl–phenol	2–Chloronaphthalene	Hexachloro–benzene	Benzo(a)–anthracene	Benzo(a)pyrene
1,4–Dichlorobenzene	bis(2–Chloro–ethoxy)methane	2–Nitroaniline	Pentachloro–phenol	bis(2–ethyl-hexyl)phthalate	Indeno(1,2,3–cd)-pyrene
1,2–Dichlorobenzene	2,4–Dichloro–phenol	Dimethylphthalate	Phenanthrene	Chrysene	Dibenz(a,h)-anthracene
2–Methylphenol	1,2,4–Trichloro–benzene	Acenaphthylene	Carbazole	Terphenyl–d14 (surr)	Benzo(g,h,i)-perylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3–Nitroaniline	Anthracene		
4–Methylphenol	4–Chloroaniline	Acenaphthene	Di–n–butyl-phthalate		
N–Nitroso–Di–n–propylamine	Hexachloro–butadiene	2,4–Dinitrophenol	Fluoranthene		
Hexachloroethane	4–Chloro–3–methylphenol	4–Nitrophenol	2,4,6-Tribromo-phenol (surr)		
2–Fluorophenol (surr)	2–Methylnaph-thalene	Dibenzofuran			
Phenol–d5 (surr)	Nitrobenzene–d5 (surr)	2,4–Dinitrotoluene			
2-Chlorophenol-d4 (surr)		2,6–Dinitrotoluene			
1,2-Dichlorobenzene-d4 (surr)		Diethylphthalate			
		4–Chlorophenyl phenylether			
		Fluorene			
		4–Nitroaniline			
		2–Fluorobiphenyl (surr)			

(surr) = surrogate compound

Table App.B.VII-2 - CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR SEMIVOLATILE COMPOUNDS

Internal Standard	Characteristic Ions	
	Primary Quantitation Ion	Secondary Ion
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

Internal standard area counts for each of the internal standards must be within the inclusive range of -50.0% and + 100.0% of the response of internal standards in the associated daily continuing calibration standard.

The retention time of the internal standard must not vary by more than ± 30 seconds from the retention time of the associated daily continuing calibration standard.

If one or more internal standard area count and/or retention time does not meet the acceptance criteria, then the sample must be reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Reanalysis is not required if the sample is a QC sample and both the matrix spike and matrix spike duplicate failed to meet the internal standard acceptance criteria. If the sample was reanalyzed and the internal standard area counts and/or retention times were acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the reanalysis also recovers the internal standard outside of the area count and/or retention time acceptance criteria, then both analyses should have been submitted.

SECTION VIII: MATRIX SPIKE/ MATRIX SPIKE DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VIII-B for MS/MSD data validation criteria and the following method MS/MSD QC criteria:

A matrix spike and matrix spike duplicate must be extracted and analyzed for each group of samples of a similar matrix for each SDG, or each matrix within an SDG or each group of samples of a similar concentration level. The following advisory matrix spike compound recoveries and RPDs are listed below:

MS/MSD base neutral compounds 1,4-dichlorobenzene, N-nitroso-di-n-propylamine, 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, and pyrene are spiked at a concentration of 100 ug/mL and MS/MSD acid compounds phenol, 2-chlorophenol, 4-chloro-3-methylphenol, 4-nitrophenol, and pentachlorophenol are spiked at a concentration of 150 ug/mL.

Table App.B.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	Method QC Criteria			
	Water		Soil/Sediment	
	% Recovery*	RPD**	% Recovery	RPD
Phenol	12-110	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-97	28	28-104	27
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39-98	28	38-107	23
4-Chloro-3-methylphenol	23-97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10-80	50	11-114	50
2,4-Dinitrotoluene	24-96	38	28-89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

*The MS/MSD % recovery is calculated using the following equation:

$$Matrix\ Spike\ Recovery = \frac{SSR - SR}{SA} X\ 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

**The MS/MSD relative percent difference (RPD) is calculated using the following equation:

$$Relative\ Percent\ Difference = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)}\ x\ 100$$

Where,

MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

Note: The vertical bars in the formula indicate the absolute value of the difference, hence the RPD is always positive.

SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IX-B for field duplicate data validation criteria.

SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-X-B for sensitivity check data validation criteria.

SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XI-B for accuracy check data validation criteria.

SECTION XII: TARGET COMPOUND IDENTIFICATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XII-B for target compound identification data validation criteria.

SECTION XIII: COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIII-B for compound quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Semivolatile target compounds must be quantitated using the internal standard method with the internal standards assigned in Appendix B, Section VII. The daily RRF50 must be used for sample quantitation. The sample target compounds must be quantified using the following primary quantitation ions and must be reported to the CRQLs listed below:

Table App.B.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLMO3.1 SOW SEMIVOLATILE ORGANIC COMPOUNDS

		Quantitation Limits					
		Low Water	Med Soil	Soil	Column	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)	Primary	Secondary
Phenol	108-95-2	10	330	10000	(20)	94	65,66
bis(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)	93	63,95
2-Chlorophenol	95-57-8	10	330	10000	(20)	128	64,130
1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)	146	148,113
1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)	146	148,113
1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)	146	148,113
2-Methylphenol	95-48-7	10	330	10000	(20)	108	107
2,2'-oxybis (1-Chloropropane)#	108-60-1	10	330	10000	(20)	45	77,79
4-Methylphenol	106-44-5	10	330	10000	(20)	108	107
N-Nitroso-di-n-propylamine	621-64-7	10	330	10000	(20)	70	42,101,130
Hexachloroethane	67-72-1	10	330	10000	(20)	117	201,199
Nitrobenzene	98-95-3	10	330	10000	(20)	77	123,65
Isophorone	78-59-1	10	330	10000	(20)	82	95,138
2-Nitrophenol	88-75-5	10	330	10000	(20)	139	65,109
2,4-Dimethylphenol	105-67-9	10	330	10000	(20)	107	121,122
bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)	93	95,123
2,4-Dichlorophenol	120-83-2	10	330	10000	(20)	162	164,98
1,2,4-Trichlorobenzene	120-82-1	10	330	10000	(20)	180	182,145
Naphthalene	91-20-3	10	330	10000	(20)	128	129,127
4-Chloroaniline	106-47-8	10	330	10000	(20)	127	129
Hexachlorobutadiene	87-68-3	10	330	10000	(20)	225	223,227

Previously known by the name bis(2-Chloroisopropyl) ether

Table App.B.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLM03.1 SOW SEMIVOLATILE ORGANIC COMPOUNDS (CONT.)

		Quantitation Limits					
		Low Water	Med Soil	Soil	Column	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)	Primary	Secondary
4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)	107	144,142
2-Methylnaphthalene	91-57-6	10	330	10000	(20)	142	141
Hexachlorocyclopentadiene	77-47-4	10	330	10000	(20)	237	235,272
2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)	196	198,200
2,4,5-Trichlorophenol	95-95-4	25	830	25000	(50)	196	198,200
2-Chloronaphthalene	91-58-7	10	330	10000	(20)	162	164,127
2-Nitroaniline	88-74-4	25	830	25000	(50)	65	92,138
Dimethylphthalate	131-11-3	10	330	10000	(20)	163	194,164
Acenaphthylene	208-96-8	10	330	10000	(20)	152	151,153
2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)	165	89, 121
3-Nitroaniline	99-09-2	25	830	25000	(50)	138	108,92
Acenaphthene	83-32-9	10	330	10000	(20)	153	152,154
2,4-Dinitrophenol	51-28-5	25	830	25000	(50)	184	63,154
4-Nitrophenol	100-02-7	25	830	25000	(50)	109	139,65
Dibenzofuran	132-64-9	10	330	10000	(20)	168	139
2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)	165	63,182
Diethylphthalate	84-66-2	10	330	10000	(20)	149	177,150
4-Chlorophenyl-phenylether	7005-72-3	10	330	10000	(20)	204	206,141
Fluorene	86-73-7	10	330	10000	(20)	166	165,167
4-Nitroaniline	100-01-6	25	830	25000	(50)	138	92,108
4,6-Dinitro-2-methylphenol	534-52-1	25	830	25000	(50)	198	182,77
N-nitroso-di-phenylamine	86-30-6	10	330	10000	(20)	169	168,167
4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)	248	250,141
Hexachlorobenzene	118-74-1	10	330	10000	(20)	284	142,149
Pentachlorophenol	87-86-5	25	830	25000	(50)	266	264,268
Phenanthrene	85-01-8	10	330	10000	(20)	178	179,176
Anthracene	120-12-7	10	330	10000	(20)	178	179,176
Carbazole	86-74-8	10	330	10000	(20)	167	166,139
Di-n-butylphthalate	84-74-2	10	330	10000	(20)	149	150,104
Fluoranthene	206-44-0	10	330	10000	(20)	202	101,100
Pyrene	129-00-0	10	330	10000	(20)	202	101,100
Butylbenzylphthalate	85-68-7	10	330	10000	(20)	149	91, 206

Table App.B.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLM03.1 SOW SEMIVOLATILE ORGANIC COMPOUNDS (CONT.)

		Quantitation Limits					
		Low	Med				
		Water	Soil	Soil	Column	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)	Primary	Secondary
3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)	252	254,126
Benzo(a)anthracene	56-55-3	10	330	10000	(20)	228	229,226
Chrysene	218-01-9	10	330	10000	(20)	228	226,229
bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	(20)	149	167,279
Di-n-octylphthalate	117-84-0	10	330	10000	(20)	149	--
Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)	252	253,125
Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)	252	253,125
Benzo(a)pyrene	50-32-8	10	330	10000	(20)	252	253,125
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	(20)	276	138,227
Dibenz(a,h)anthracene	53-70-3	10	330	10000	(20)	278	139,279
Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)	276	138,277

SAMPLE CONCENTRATION - the amount of analyte present in a sample is calculated using RRF50 of the continuing calibration standard in the following equations:

Sample concentration for water:

$$ug/L = \frac{(A_x)\ (IS)\ (V_t)\ (Df)\ (GPC)}{(A_{is})\ (RRF)\ (V_i)\ (V_o)}$$

Sample concentration for low and medium level soil/sediment:

$$ug/Kg\ (Dry\ weight\ basis) = \frac{(A_x)\ (IS)\ (V_t)\ (Df)\ (GPC)}{(A_{is})\ (RRF)\ (V_i)\ (W_s)\ (D)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor from the most recent continuing calibration standard
Df = Dilution Factor - The dilution factor for analysis of water and soil/sediment samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.
W_s = Weight of sample extracted in grams (g)
D = $\frac{100 - \%\ Moisture}{100}$
V_t = Volume of the concentrated extract in microliters (uL)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (uL)
GPC = GPC factor (If no GPC is performed, GPC = 1; if GPC is performed, then GPC = 2.0)

CRQL CALCULATIONS

Water:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(V_x)\ (V_t)\ (V_y)\ (Df)}{(V_o)\ (V_c)\ (V_i)}$$

Where,

V_t, V_o, V_i and Df are defined in the sample concentration equation above
V_x = Contract sample volume (1000 mL)
V_y = Contract injection volume (2 uL)
V_c = Contract concentrated extract volume (1000 uL if GPC is not performed, 500 uL if GPC was performed).

CRQL CALCULATIONS

Soil/Sediment:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(W_x)\ (V_t)\ (V_y)\ (Df)}{(V_c)\ (V_i)\ (W_s)\ (D)}$$

Where,

V_t, Df, W_s, V_i and D are defined in the sample concentration equation above
W_x = Contract sample weight (30 g for low level and 1 g for medium level soil/sediment samples).
V_y = Contract injection volume (2 uL)
V_c = Contract concentrated extract volume (1000 uL if GPC is not performed, 500 uL if GPC was performed).

SECTION XIV: TENTATIVELY IDENTIFIED COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIV-B for tentatively identified compound (TIC) data validation criteria and the following method TIC QC criteria:

The validator is required to report up to 30 TICs in the Data Validation Memorandum.

TENTATIVELY IDENTIFIED COMPOUND CONCENTRATION - the estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method using the following equations:

Sample concentration for water:

$$ug/L = \frac{(A_x)\ (IS)\ (V_t)\ (Df)\ (GPC)}{(A_{is})\ (RRF)\ (V_i)\ (V_o)}$$

Sample concentration for low and medium level soil/sediment:

$$ug/Kg = \frac{(A_x)\ (IS)\ (V_t)\ (Df)\ (GPC)}{(A_{is})\ (RRF)\ (V_i)\ (W_s)\ (D)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the non-target compound to be measured
A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor is assumed to be 1
Df = Dilution Factor - The dilution factor for analysis of water and soil/sediment samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.
W_s = Weight of sample extracted in grams (g)
D = $\frac{100 - \%\ Moisture}{100}$
V_t = Volume of the concentrated extract in microliters (uL)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (uL)
GPC = GPC factor (If no GPC is performed, GPC = 1; if GPC is performed, then GPC = 2.0)

SECTION XV: SEMIVOLATILE CLEANUP CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XV-B for semivolatile data validation criteria and the following method semivolatile cleanup QC criteria:

GPC

Initial GPC calibration consists of analyzing the GPC calibration solution to establish the correct "Collect" and "Dump" time periods and a GPC blank to ensure that the system is free of contaminants.

1. The GPC Calibration Solution contains the following analytes in methylene chloride at the specified concentrations:

   corn oil - 25.0 mg/mL
   bis-(2-ethylhexyl)phthalate - 0.5 mg/mL
   methoxychlor - 0.2 mg/mL
   perylene - 0.02 mg/mL
   sulfur (optional) - 0.08 mg/mL

2. The GPC blank consists of methylene chloride.

The GPC must be recalibrated every 7 days with the GPC Calibration Solution followed by a methylene chloride GPC blank.

Table App.B.XVI-1 - INITIAL AND CONTINUING GPC CALIBRATION CRITERIA

Peak Resolution	Corn Oil and phthalate peaks must exhibit > 85% resolution. Phthalate and methoxychlor peaks must exhibit > 85% resolution. Methoxychlor and perylene peaks must exhibit > 85% resolution. Perylene and sulfur peaks (if sulfur was added) must not be saturated and must exhibit > 90% baseline resolution.
Peak Shape	Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
Retention Time	The retention times must not vary more than ± 5.0% between calibrations.
Blanks	A GPC blank must be analyzed after each initial GPC calibration. Target analytes cannot be present at greater than the CRQL for any target analyte except phthalate esters, which must be < 5x CRQL.

SECTION XVI: SYSTEM PERFORMANCE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVI-B for system performance data validation criteria.

SECTION XVII: OVERALL ASSESSMENT CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVII-B for overall assessment data validation criteria.

Appendix C
CLP/SOW OLC02.1/Low Concentration Volatile Organic Analysis
Method QC criteria, Equations, and Definitions

APPENDIX C

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Organic Analysis, Low Concentration Water, OLC02.1, Exhibit D Volatiles**.

Note: MS/MSD are not applicable. MS/MSDs are not required for work under this SOW.

Capillary GC columns are mandatory. Packed columns cannot be used.

SECTION I: PRESERVATION & TECHNICAL HOLDING TIME CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-I-B for preservation and technical holding time data validation criteria.

SECTION II: GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING) CRITERIA

Refer to the following method GC/MS instrument performance (tuning) QC criteria for data validation:

The analysis of the instrument performance (tuning) check solution (50 ng BFB on column) must be performed at the beginning of each **12-hour** period during which samples or standards are analyzed. The tuning check, bromofluorobenzene (BFB), for volatile analysis must meet the ion abundance criteria given below:

m/z	ION ABUNDANCE CRITERIA
50	8.0 - 40.0% of m/z 95
75	30.0 - 66.0% of m/z 95
95	Base Peak, 100% Relative Abundance
96	5.0 - 9.0% of m/z 95 (see note)
173	Less than 2.0% of m/z 174
174	50.0 - 120.0% of m/z 95
175	4.0 - 9.0% of mass 174
176	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Part of the BFB peak must not be background subtracted.

SECTION III: INITIAL CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

The initial calibration standards must be analyzed upon contract award, whenever corrective action is taken which may change or affect the initial calibration criteria or if the continuing calibration acceptance criteria have not been met. Initial calibrations must be analyzed after the analysis of a compliant instrument performance check.

The initial calibration standards must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compound.

All initial calibration standards must be analyzed at the following concentration levels; 1.0, 2.0, 5.0, 10, and 25 ug/L except for the ketones which are analyzed at 5.0, 10, 25, 50, and 125 ug/L.

RELATIVE RESPONSE FACTOR (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. The RRF is calculated using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of primary quantitation ion response (EICP) for the internal standard
C_{is} = Concentration of the internal standard
C_x = Concentration of the compound to be measured

AVERAGE (MEAN) RELATIVE RESPONSE FACTOR (RRF) - The average or mean RRF is determined by the analysis of five different standard concentrations and is used in calculating a compound concentration in samples. The RRF is calculated using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{RRF_i}{n}$$

Where,

RRF_i = The individual RRFs for various concentration levels
n = The number of RRFs

PERCENT RELATIVE STANDARD DEVIATION (%RSD) - The % RSD for each compound is a measure of the linearity of the calibration curve. The % RSD is calculated using the following equation:

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

Where,

$$Standard\ Deviation = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}}$$

$\bar{x}$ = Mean
n = total number of values
x_i= each individual value used to calculate the mean

SECTION IV: CONTINUING CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IV-B for continuing calibration data validation criteria and the following method continuing calibration QC criteria:

The continuing calibration standard must be analyzed once every 12 hours, following the analysis of a compliant instrument performance check and initial calibration, and prior to the analysis of field samples, QC samples and blanks.

The continuing calibration standard must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compound.

Continuing calibrations must be analyzed at a final concentration of 5 ug/L for non-ketone compounds and at a final concentration 25 ug/L for the ketones.

Note: The low concentration method % Difference for continuing calibration differs from the Region I Functional Guidelines continuing calibration % Difference criteria (± 25.0 %). The low concentration method requires that the continuing calibration % Difference for most target compounds and surrogates be less than or equal to ± 30.0%. The following compounds do not have a minimum % D requirement but must meet a minimum RRF criterion of 0.010: carbon disulfide, chloroethane, chloromethane, cis-1,2-dichloroethene, trans-1,2-dichloroethene, 1,2-dichloropropane, and methylene chloride. Furthermore, OLC02.1 does not specify RRF or % D criteria for the following compounds: acetone, 2-butanone, 1,2-dibromo-3-chloropropane, 2-hexanone, 4-methyl-2-pentanone, and 1,2,4-trichlorobenzene. (If data quality objectives allow for greater variability of data, then expanded %D and minimum response factor criteria should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low RFs in sample calculations.

PERCENT DIFFERENCE (%D) - The % D is used to compare the initial calibration mean RRF with the continuing calibration RRF5. The % Difference indicates both the direction and the magnitude of the comparison, i.e., the % Difference may be either negative, positive or zero.

$$\% \ Difference = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \ x \ 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria
RRF_c = Relative response factor from continuing calibration standard

SECTION V: BLANK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-V-B for blank data validation criteria and the following method QC criteria.

Method Required Blanks

1. Method Blank - A 25.0 mL aliquot of reagent water that is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. The method blank must be analyzed at least once during every 12 hour time period on each GC/MS system used for volatile analysis.

2. Storage Blank - Consists of two 40 mL VOA vials filled with reagent water prepared by the laboratory when the first samples in the SDG are received. The vials are stored, under the same conditions, with the field samples. After all the samples in the SDG are analyzed, a 25.0 mL aliquot of the storage blank is analyzed to determine whether contamination was introduced during storage of the samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. A minimum of one storage blank must be analyzed per SDG after all samples for that SDG have been analyzed.

3. Instrument Blank - A 25.0 ml aliquot of reagent water that is carried through the entire analytical procedure and is analyzed following highly contaminated samples containing target compounds that exceed the initial calibration range. The instrument blanks are used to determine if contamination is introduced from a previous sample and the level associated with the analytical instrument. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. Instrument blanks are analyzed after a sample/dilution which contains a target compound at a concentration greater than 25 ug/L (ketones 125 ug/L) or a non-target compound at a concentration greater than 100 ug/L or saturated ions from a compound (excluding peaks in the solvent front).

SECTION VI: SURROGATE COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VI-B for surrogate compound data validation criteria and the following method surrogate compound QC criteria:

The proper surrogate compounds must be quantified using the correctly assigned internal standards and the correct primary quantitation ions.

The surrogate compound, 4-Bromofluorobenzene, is added to all samples, standards, QC samples, and blanks for a final concentration of 5 ug/L.

Table App.D.VI-1 - CHARACTERISTIC IONS FOR SURROGATE COMPOUNDS

Surrogate	Characteristic Ions		
	Primary Quantitation Ion	Secondary Ion(s)	Internal Standard
4-Bromofluorobenzene	174	95, 176	1,4-Difluorobenzene

The surrogate % recovery is calculated using the following equation:

$$Surrogate\ Percent\ Recovery = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of surrogate determined by analysis

Q_a = Quantity of surrogate added to sample/blank

Table App.D.VI-2 - SURROGATE RECOVERY LIMITS

Surrogate	Method QC Criteria
	Percent Recovery (Water)
4-Bromofluorobenzene	80-120

Sample reanalysis is required for samples that do not meet the surrogate recovery acceptance criteria.

SECTION VII: INTERNAL STANDARDS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VII-B for internal standard data validation criteria and the following method internal standard QC criteria:

The correct internal standard must be used for sample compound quantitation and the correct internal standard primary quantitation ion must be used for quantitation.

The internal standards Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d_5 are added to all samples, standards, QC samples, and blanks for a final concentration of 5.0 ug/L.

Table App.D.VII-1 - LOW CONCENTRATION VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

IS 1,4-Difluorobenzene	IS Chlorobenzene-d_5	IS 1,4-Dichlorobenzene-d_4
Acetone	Benzene	Bromoform
Bromochloromethane	Bromodichloromethane	1,2-Dibromo-3-chloropropane
Bromomethane	Carbon tetrachloride	1,2-Dichlorobenzene
2-Butanone	Chlorobenzene	1,3-Dichlorobenzene
Carbon disulfide	Dibromochloromethane	1,4-Dichlorobenzene
Chloroethane	1,2-Dibromoethane	1,2,4-Trichlorobenzene
Chloroform	1,2-Dichloropropane	
Chloromethane	cis-1,3-Dichloropropene	
1,1-Dichloroethane	trans-1,3-Dichloropropene	
1,2-Dichloroethane	Ethylbenzene	
1,1-Dichloroethene	2-Hexanone	
cis-1,2-Dichloroethene	4-Methyl-2-pentanone	
trans-1,2-Dichloroethene	Styrene	
Methylene chloride	1,1,2,2-Tetrachloroethane	
Vinyl chloride	Tetrachloroethene	
	Toluene	
	1,1,1-Trichloroethane	
4-Bromofluorobenzene (surr)	1,1,2-Trichloroethane	
	Trichloroethene	
	Xylenes (total)	

Table App.D.VII-2 - CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR LOW CONCENTRATION VOLATILE COMPOUNDS

Internal Standard	**Characteristic Ions**	
	Primary Quantitation Ion	**Secondary Ion(s)**
1,4-Difluorobenzene	114	63, 88
1,4-Dichlorobenzene-d4	152	115, 50
Chlorobenzene-d5	117	82, 119

Internal standard area counts for each of the internal standards must be within the inclusive range of ± 40% of the response of internal standards in the associated daily continuing calibration standard.

The retention time of the internal standard must not vary by more than ± 20 seconds from the retention time of the associated daily continuing calibration standard.

Sample reanalysis is required for samples that do not meet the internal standard acceptance criteria.

SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA

The Low Concentration method does not require MS/MSD analysis therefore, no method-specific criteria are available for MS/MSD. Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VIII-B for MS/MSD validation criteria if MS/MSD analyses are performed.

SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IX-B for field duplicate data validation criteria.

SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-X-B for sensitivity check data validation criteria.

SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XI-B for accuracy check data validation criteria and the following method accuracy check QC criteria:
The LCS is a method-required internal laboratory quality control sample that must be prepared, analyzed and reported once per SDG. It must be prepared and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples.

Compound	Final Concentration ug/L	Method Required QC % Recovery Limits
Vinyl chloride	5.0	60 - 140
1,2- Dichloroethane	5.0	60 - 140
Carbon tetrachloride	5.0	60 - 140
1,2-Dichloropropane	5.0	60 - 140
Trichloroethene	5.0	60 - 140
1,1,2-Trichloroethane	5.0	60 - 140
Benzene	5.0	60 - 140
cis-1,3-Dichloropropene	5.0	60 - 140
Bromoform	5.0	60 - 140
Tetrachloroethene	5.0	60 - 140
1,2-Dibromoethane	5.0	60 - 140
1,4-Dichlorobenzene	5.0	60 - 140

SECTION XII: TARGET COMPOUND IDENTIFICATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XII-B for target compound identification data validation criteria.

SECTION XIII: COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIII-B for compound quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Volatile target compounds must be quantitated using the internal standard method with the internal standards assigned in Appendix A, Section VII. The daily RRF5 must be used for sample quantitation. The sample target compounds must be quantified using the following primary quantitation ions and must be reported to the CRQLs listed below:

Table App.D.XIII-1 - TARGET COMPOUND LIST (TCL), PRIMARY QUANTITATION AND SECONDARY IONS, AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR OLCO2.0 SOW VOLATILE ORGANIC COMPOUNDS

		Quantitation Limits		Characteristic Ions	
Volatiles	CAS Number	Low Water ug/L	On Column (ng)	Primary	Secondary
Chloromethane	74)87)3	1	(25)	50	52
Bromochloromethane	74-97-5	1	(25)	128	49,130
1,2-Dibromoethane	106-93-4	1	(25)	107	109,188
Bromomethane	74)83)9	1	(25)	94	96
Vinyl Chloride	75)01)4	1	(25)	62	64
Chloroethane	75)00)3	1	(25)	64	66
Methylene Chloride	75)09)2	2	(50)	84	86,49
Acetone	67)64)1	5	(125)	43	58
Carbon Disulfide	75)15)0	1	(25)	76	78
1,1)Dichloroethene	75)35)4	1	(25)	96	61,63
1,1)Dichloroethane	75)34)3	1	(25)	63	65,83
cis-1,2)Dichloroethene	156)59)2	1	(25)	96	61,98
trans-1,2-Dichloroethene	156-60-5	1	(25)	96	61,98
Chloroform	67)66)3	1	(25)	83	85
1,2)Dichloroethane	107)06)2	1	(25)	62	98
2)Butanone	78)93)3	5	(125)	43	72*
1,1,1)Trichloroethane	71)55)6	1	(25)	97	99,61
Carbon Tetrachloride	56)23)5	1	(25)	117	119
Bromodichloromethane	75)27)4	1	(25)	83	85,127
1,2)Dichloropropane	78)87)5	1	(25)	63	112
cis)1,3)Dichloropropene	10061)01)5	1	(25)	75	77
Trichloroethene	79)01)6	1	(25)	95	130,132
Dibromochloromethane	124)48)1	1	(25)	129	127
1,1,2)Trichloroethane	79)00)5	1	(25)	97	83,85,99,132,134
Benzene	71)43)2	1	(25)	78	--
trans)1,3)Dichloropropene	10061)02)6	1	(25)	75	77
Bromoform	75)25)2	1	(25)	173	175,254
4)Methyl)2)pentanone	108)10)1	5	(125)	43	58,100
2)Hexanone	591)78)6	5	(125)	43	58,57,100
Tetrachloroethene	127)18)4	1	(25)	166	168,129
Toluene	108)88)3	1	(25)	91	92
1,1,2,2)Tetrachloroethane	79)34)5	1	(25)	83	131,85
1,2,4-Trichlorobenzene	120-82-1	1	(25)	180	182,145
1,2-Dichlorobenzene	95-50-1	1	(25)	146	111,148
1,3-Dichlorobenzene	541-73-1	1	(25)	146	111,148
1,4-Dichlorobenzene	106-46-7	1	(25)	146	111,148
Chlorobenzene	108)90)7	1	(25)	112	77,114
Ethylbenzene	100)41)4	1	(25)	91	106
Styrene	100)42)5	1	(25)	104	78
Xylenes (Total)	1330)20)7	1	(25)	106	91
1,2-Dibromo-3-chloropropane	96-12-8	1	(25)	75	155,157

* - m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

SAMPLE CONCENTRATION - The amount of analyte present in a sample is calculated using the RRF5 of the continuing calibration standard in the following equation:

Sample concentration for water:

$$ug/L = \frac{(A_x)(IS)(Df)}{(A_{is})(RRF)(V_o)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = The Relative Response Factor from the most recent continuing calibration standard
V_o = Total volume of water purged in milliliters (mL)
Df = Dilution Factor - The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. If no dilution is performed , Df=1.

CRQL CALCULATIONS

Water:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(V_x)}{(V_o)}\ x\ (Df)$$

Where,

V_o and Df are defined in the sample concentration equation above
V_x = Contract sample volume (5 to 25 mL)

SECTION XIV: TENTATIVELY IDENTIFIED COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIV-B for tentatively identified compound (TIC) data validation criteria and the following method TIC QC criteria:

The validator is required to report up to 30 TICs in the Data Validation Memorandum.

TENTATIVELY IDENTIFIED COMPOUND CONCENTRATION - the estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method using the following equations:

Sample concentration for water:

$$ug/L = \frac{(A_x)(IS)(Df)}{(A_{is})(RRF)(V_o)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the non-target compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor assumed to be 1
V_o = Total volume of water purged in milliliters (mL)
Df = Dilution Factor - The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. If no dilution is performed , Df=1.

SECTION XV: SEMIVOLATILE CLEANUP CRITERIA

Not applicable to low concentration volatile analysis.

SECTION XVI: SYSTEM PERFORMANCE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVI-B for system performance data validation criteria.

SECTION XVII: OVERALL ASSESSMENT CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVII-B for overall assessment data validation criteria.

APPENDIX D

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Organic Analysis, Low Concentration Water, OLC02.1, Exhibit D Semivolatiles**.

Note: MS/MSD are not applicable. MS/MSDs are not required for work under this SOW.

Capillary GC columns are mandatory. Packed columns cannot be used.

SECTION I: PRESERVATION & TECHNICAL HOLDING TIME CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-I-B for preservation and technical holding time data validation criteria.

SECTION II: GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING) CRITERIA

Refer to the following method GC/MS instrument performance (tuning) QC criteria for data validation:

The analysis of the instrument performance (tuning) check solution (50 ng DFTPP on column) must be performed at the beginning of each **12-hour** period during which samples or standards are analyzed. The tuning check, decafluorotriphenylphosphine (DFTPP), for semivolatile analysis must meet the ion abundance criteria given below:

m/z	ION ABUNDANCE CRITERIA
51	30.0 - 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 - 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base Peak, 100% Relative Abundance (see note)
199	5.0 - 9.0% of m/z 198
275	10.0 - 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 - 110.0% of m/z 198
443	15.0 - 24.0% of m/z 442

Note: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110% that of m/z 198.

The mass spectrum of DFTPP must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Part of the DFTPP peak must not be background subtracted.

SECTION III: INITIAL CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

The initial calibration standards must be analyzed upon contract award, whenever corrective action is taken which may change or affect the initial calibration criteria or if the continuing calibration acceptance criteria have not been met. Initial calibrations must be analyzed after the analysis of a compliant instrument performance check.

The initial calibration standards must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

1 uL volume of the initial calibration standard must be injected and all initial calibration standards must be analyzed at the following concentration levels; 5.0, 10, 20, 50, and 80 ng/uL except for eight target compounds and one surrogate compound. Compounds 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4-6-dinitro-2-methylphenol, pentachlorophenol, and 2,4,6-tribromophenol(surr.) must be analyzed at 20, 50, 80, 10, and 120 ng/uL.

RELATIVE RESPONSE FACTOR (RRF) - A measure of the relative mass spectral response of an analyte compared to its internal standard. The RRF is calculated using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x	=	Area of primary quantitation ion response (EICP) for the compound to be measured
A_{is}	=	Area of primary quantitation ion response (EICP) for the internal standard
C_{is}	=	Concentration of the internal standard
C_x	=	Concentration of the compound to be measured

AVERAGE (MEAN) RELATIVE RESPONSE FACTOR ($\overline{RRF}$) - The average or mean RRF is determined by the analysis of five different standard concentrations and is used in calculating a compound concentration in samples. The RRF is calculated using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{RRF_i}{n}$$

Where,

RRF_i	=	The individual RRFs for various concentration levels
n	=	The number of RRFs

PERCENT RELATIVE STANDARD DEVIATION (%RSD) - The % RSD for each compound is a measure of the linearity of the calibration curve. The % RSD is calculated using the following equation:

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

Where,

$$Standard\ Deviation = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}}$$

$\bar{x}$ = Mean
n = total number of values
x_i = each individual value used to calculate the mean

SECTION IV: CONTINUING CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IV-B for continuing calibration data validation criteria and the following method continuing calibration QC criteria:

The continuing calibration standard must be analyzed once every 12 hours, following the analysis of a compliant instrument performance check and initial calibration, and prior to the analysis of field samples, QC samples and blanks.

The continuing calibration standard must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

Continuing calibration standards must be analyzed at a final concentration of 20 ug/L for the majority of the compounds and 80 ug/L for the eight compounds specified in the initial calibration section.

Note: The Low Concentration method % Difference QC criteria for continuing calibration differs somewhat from the Region I Functional Guidelines continuing calibration % Difference criteria. The Low Concentration method requires that the continuing calibration % Difference be less than or equal to ± 30.0% for two compounds, 2-nitrophenol and 2,4,-dimethylphenol; whereas the Functional Guidelines requires qualification of all data associated with a continuing calibration with % Difference greater than ± 25.0%. Refer to CLP SOW OLC02.1 for those compounds that do not have % D requirements. If data quality objectives allow for greater variability of data, then expanded % D validation criteria should be documented in the site-specific QAPjP or amendment to the QAPjP.

PERCENT DIFFERENCE (%D) - The % D is used to compare the initial calibration mean RRF with the continuing calibration RRF. The % Difference indicates both the direction and the magnitude of the comparison, i.e., the % Difference may be either negative, positive or zero.

$$\% \, Difference = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where,

$\overline{RRFi}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria
RRFc = Relative response factor from continuing calibration standard

SECTION V: BLANK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-V-B for blank data validation criteria and the following method QC criteria:

Method Required Blank

Method Blank - A volume of reagent water approximate in volume to the samples which is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of the samples. All blanks are spiked with internal standard and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. A method blank must be extracted at least once for every SDG, for each 20 samples in an SDG, and whenever samples are extracted. Each method blank must be analyzed on each GC/MS used to analyzed the samples prepared with the method blank.

SECTION VI: SURROGATE COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VI-B for surrogate compound data validation criteria and the following method surrogate compound QC criteria:

Surrogate compounds must be quantified using the correctly assigned internal standards and the correct primary quantitation ions.

Surrogate compounds Nitrobenzene-d_5, 2-Fluorobiphenyl, Terphenyl-d_{14}, Phenol-d_5, and 2-Fluorophenol are added to all samples, standards, QC samples, and blanks at a concentration of 40 ug/mL and surrogate compound 2,4,6-Tribromophenol is added to all samples, standards, QC samples, and blanks at a concentration of 120 ug/mL.

Table App.E.VI-1 - CHARACTERISTIC IONS FOR SURROGATE COMPOUNDS

Surrogate	Characteristic Ions		Internal Standard
	Primary Quantitation Ion	Secondary Ion(s)	
Nitrobenzene-d_5	82	128, 54	Naphthalene-d_8
2-Fluorobiphenyl	172	171	Acenaphthene-d_{10}
Terphenyl-d_{14}	244	122, 212	Chrysene-d_{12}
Phenol-d_5	99	42, 71	1,4-Dichlorobenzene-d_4
2-Fluorophenol	112	64	1,4-Dichlorobenzene-d_4
2,4,6-Tribromophenol	330	332, 141	Phenanthrene-d_{10}

The surrogate % recovery is calculated using the following equation:

$$\textit{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of surrogate determined by analysis

Q_a = Quantity of surrogate added to sample/blank

Table App.E.VI-2 - SURROGATE RECOVERY LIMITS

Surrogate	Method QC Criteria
	Percent Recovery (Water)
Nitrobenzene-d_5	40-110
2-Fluorobiphenyl	30-110
Terphenyl-d_{14}	20-140
Phenol-d_5	15-115
2-Fluorophenol	15-110
2,4,6-Tribromophenol	15-130

If the surrogate acceptance criteria are not met, the laboratory should check calculations, surrogate standard solutions, and instrument performance. If the failed criteria are the result of instrument malfunction, only sample reanalysis is required to meet surrogate acceptance criteria. Sample re-extraction/reanalysis is required for samples that do not meet the surrogate recovery acceptance criteria, as a result of the incorrect surrogate standard solutions or any other unknown problem.

SECTION VII: INTERNAL STANDARDS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VII-B for internal standard data validation criteria and the following method internal standard QC criteria:

The correct internal standard must be used for sample compound quantification and the correct internal standard primary quantitation ion must be used for quantitation.

The internal standard compounds listed below are injected into all samples, standards, QC samples, and blanks at a concentration of 20 ng/uL.

App.E.VII-1 - LOW CONCENTRATION SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

IS 1,4–Dichlorobenzene–d4	IS Naphthalene–d8	IS Acenaphthene–d10	IS Phenanthrene–d10	IS Chrysene–d12	IS Perylene–d12
Phenol	Nitrobenzene	Hexachlorocyclo–pentadiene	4,6–Dinitro–2–methylphenol	Pyrene	Di–n–octyl-phthalate
bis(2–Chloroethyl) ether	Isophorone	2,4,6–Trichloro–phenol	N–nitroso-di–phenylamine	Butylbenzyl-phthalate	Benzo(b)fluor–anthene
2–Chlorophenol	2–Nitrophenol	2,4,5–Trichloro–phenol	4–Bromophenyl phenolether	3,3'–Dichloro–benzidine	Benzo(k)fluor–anthene
2-Methylphenol	2,4–Dimethyl–phenol	2–Chloronaphthalene	Hexachloro–benzene	Benzo(a)–anthracene	Benzo(a)pyrene
2,2'-oxybis-(1-Chloropropane)	bis(2–Chloro–ethoxy)methane	2–Nitroaniline	Pentachloro–phenol	bis(2–ethyl-hexyl)phthalate	Indeno(1,2,3–cd)-pyrene
4–Methylphenol	2,4–Dichloro–phenol	Dimethylphthalate	Phenanthrene	Chrysene	Dibenz(a,h)-anthracene
N-Nitroso-Di-n-propylamine	Naphthalene	Acenaphthylene	Anthracene	Terphenyl–d14 (surr)	Benzo(g,h,i)-perylene
Hexachloroethane	4-Chloroaniline	3–Nitroaniline	Di-n-butyl-phthalate		
2-Fluorophenol (surr)	Hexachloro–butadiene	Acenaphthene	Fluoranthene		
Phenol-d5 (surr)	4–Chloro–3–methylphenol	2,4–Dinitrophenol	2,4,6-Tribromo-phenol (surr)		
	2-Methylnaph-thalene	4–Nitrophenol			
	Nitrobenzene-d5 (surr)	Dibenzofuran			
		2,4–Dinitrotoluene			
		2,6–Dinitrotoluene			
		Diethylphthalate			
		4–Chlorophenyl-phenylether			
		Fluorene			
		4–Nitroaniline			
		2–Fluorobiphenyl (surr)			

(surr) = surrogate compound

Table App.E.VII-2 - CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR LOW CONCENTRATION SEMIVOLATILE COMPOUNDS

Internal Standard	Characteristic Ions	
	Primary Quantitation Ion	Secondary Ion(s)
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

Internal standard area counts for each of the internal standards must be within the inclusive range of - 50% and + 100% of the response of internal standards in the associated daily continuing calibration standard.

The retention time of the internal standard must not vary by more than ± 20 seconds from the retention time of the associated daily continuing calibration standard.

If the internal standard acceptance criteria are not met, the laboratory should check calculations, internal standard solutions, and instrument performance. If the failed criteria are the result of instrument malfunction, only sample reanalysis is required to meet surrogate acceptance criteria. Sample reanalysis is required for samples that do not meet the internal standard acceptance criteria, as a result of the incorrect internal standard solutions or any other unknown problem.

SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA

The Low Concentration method does not require MS/MSD analysis therefore, no method-specific criteria are available for MS/MSD. Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VIII-B for MS/MSD validation criteria, if MS/MSD analyses are performed.

SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IX-B for field duplicate data validation criteria.

SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-X-B for sensitivity check data validation criteria.

SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XI-B for accuracy check data validation criteria.

The LCS is a method required internal laboratory quality control sample that must be prepared, analyzed and reported once per SDG. It must be prepared and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples.

Compound	Final Concentration ug/L	Method Required QC % Recovery Limits
Phenol	40.0	40 - 120
2-Chlorophenol	40.0	50 - 110
4-Chloroaniline	40.0	10 - 120
2,4,6-Trichlorophenol	40.0	40 - 120
bis(2-Chloroethyl)ether	20.0	50 - 110
N-Nitroso-di-n-propylamine	20.0	30 - 110
Hexachloroethane	20.0	20 - 110
Isophorone	20.0	50 - 110
Naphthalene	20.0	30 - 110
2,4-Dinitrotoluene	20.0	30 - 120
Diethylphthalate	20.0	50 - 120
N-Nitrosodiphenylamine	20.0	30 - 110
Hexachlorobenzene	20.0	40 - 120
Benzo(a)pyrene	20.0	50 - 120

SECTION XII: TARGET COMPOUND IDENTIFICATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XII-B for target compound identification data validation criteria.

SECTION XIII: COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMIT CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIII-B for compound quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Semivolatile target compounds must be quantitated using the internal standard method with the internal standards assigned in Appendix E, Section VII. The daily RRF20 must be used for sample quantitation. The sample target compounds must be quantified using the following primary quantitation ions and must be reported to the CRQLs listed below:

App.E.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLCO2.0 SOW SEMIVOLATILE ORGANIC COMPOUNDS

		Quantitation Limits		
		Water	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	Primary	Secondary
Phenol	108–95–2	5	94	65, 66
bis(2–Chloroethyl) ether	111–44–4	5	93	63, 95
2–Chlorophenol	95–57–8	5	128	64, 130
2–Methylphenol	95–48–7	5	108	107
2,2'-oxybis (1-Chloropropane)#	108–60–1	5	45	77, 79
4–Methylphenol	106–44–5	5	108	107
N–Nitroso–di–n-propylamine	621–64–7	5	70	42, 101, 130
Hexachloroethane	67–72–1	5	117	201, 199
Nitrobenzene	98–95–3	5	77	123, 65
Isophorone	78–59–1	5	82	95, 138
2–Nitrophenol	88–75–5	5	139	65, 109
2,4–Dimethylphenol	105–67–9	5	107	121, 122
bis(2–Chloroethoxy) methane	111–91–1	5	93	95, 123
2,4–Dichlorophenol	120–83–2	5	162	164, 98
Naphthalene	91–20–3	5	128	129, 127
4–Chloroaniline	106–47–8	5	127	129
Hexachlorobutadiene	87–68–3	5	225	223, 227
4–Chloro–3–methylphenol	59–50–7	5	107	144, 142
2–Methylnaphthalene	91–57–6	5	142	141
Hexachlorocyclopentadiene	77–47–4	5	237	235, 272
2,4,6–Trichlorophenol	88–06–2	5	196	198, 200
2,4,5–Trichlorophenol	95–95–4	20	196	198, 200
2–Chloronaphthalene	91–58–7	5	162	164, 127
2–Nitroaniline	88–74–4	20	65	92, 138
Dimethylphthalate	131–11–3	5	163	194, 164
Acenaphthylene	208–96–8	5	152	151, 153
2,6–Dinitrotoluene	606–20–2	5	165	89, 121
3–Nitroaniline	99–09–2	20	138	108, 92
Acenaphthene	83–32–9	5	153	152, 154
2,4–Dinitrophenol	51–28–5	20	184	63, 154

Previously known by the name bis(2-Chloroisopropyl) ether

App.E.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLCO2. SOW SEMIVOLATILE ORGANIC COMPOUNDS (CONT.)

		Quantitation Limits		
		Water	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	Primary	Secondary
4–Nitrophenol	100–02–7	20	109	139, 65
Dibenzofuran	132–64–9	5	168	139
2,4–Dinitrotoluene	121–14–2	5	165	63, 182
Diethylphthalate	84–66–2	5	149	177, 150
4–Chlorophenyl–phenylether	7005–72–3	5	204	206, 141
Fluorene	86–73–7	5	166	165, 167
4–Nitroaniline	100–01–6	20	138	92, 108
4,6–Dinitro–2–methylphenol	534–52–1	20	198	182, 77
N–nitrosodiphenylamine	86–30–6	5	169	168, 167
4–Bromophenyl–phenylether	101–55–3	5	248	250, 141
Hexachlorobenzene	118–74–1	5	284	142, 249
Pentachlorophenol	87–86–5	20	266	264, 268
Phenanthrene	85–01–8	5	178	179, 176
Anthracene	120–12–7	5	178	179, 176
Di–n–butylphthalate	84–74–2	5	149	150, 104
Fluoranthene	206–44–0	5	202	101, 100
Pyrene	129–00–0	5	202	101, 100
Butylbenzylphthalate	85–68–7	5	149	91, 206
3,3'–Dichlorobenzidine	91–94–1	5	252	254, 126
Benzo(a)anthracene	56–55–3	5	228	229, 226
Chrysene	218–01–9	5	228	226, 229
bis(2–Ethylhexyl)phthalate	117–81–7	5	149	167, 279
Di–n–octylphthalate	117–84–0	5	149	---
Benzo(b)fluoranthene	205–99–2	5	252	253, 125
Benzo(k)fluoranthene	207–08–9	5	252	253, 125
Benzo(a)pyrene	50–32–8	5	252	253, 125
Indeno(1,2,3–cd)pyrene	193–39–5	5	276	138, 227
Dibenz(a,h)anthracene	53–70–3	5	278	139, 279
Benzo(g,h,i)perylene	191–24–2	5	276	138, 277

SAMPLE CONCENTRATION - The amount of analyte present in a sample is calculated using the RRF20 of the continuing calibration standard in the following equation:

Sample concentration for water:

$$ug/L = \frac{(A_x)\ (IS)\ (V_t)\ (Df)}{(A_{is})\ (RRF)\ (V_o)\ (V_i)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = The Relative Response Factor from the most recent continuing calibration standard
Df = Dilution Factor - The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.
V_t = Volume of the concentrated final extract in microliters (uL)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (uL)

CRQL CALCULATIONS

Water:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(V_x)\ (V_t)\ (V_y)\ (Df)}{(V_o)\ (V_c)\ (V_i)}$$

Where,

V_t, V_o, V_i and Df are defined in the sample concentration equation above
V_x = Contract sample volume (1000 mL)
V_y = Contract injection volume (1 uL)
V_c = Contract concentrated extract volume (1000 uL)

SECTION XIV: TENTATIVELY IDENTIFIED COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIV-B for tentatively identified compound (TIC) data validation criteria and the following method TIC QC criteria:

The validator is required to report up to 30 TICs in the Data Validation Memorandum.

TENTATIVELY IDENTIFIED COMPOUND CONCENTRATION - the estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method using the following equations:

Sample concentration for water:

$$ug/L = \frac{(A_x)\ (IS)\ (V_t)\ (Df)}{(A_{is})\ (RRF)\ (V_o)\ (V_i)}$$

Where,

A_x = Area of the primary quantitation ion response (EICP) for the non-target compound to be measured
A_{is} = Area of the primary quantitation ion response (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor of 1 (one) is assumed
Df = Dilution Factor - The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.1
V_t = Volume of the concentrated final extract in microliters (uL)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (uL)

SECTION XV: SEMIVOLATILE CLEANUP CRITERIA

Not applicable to low concentration semivolatile analysis.

SECTION XVI: SYSTEM PERFORMANCE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVI-B for system performance data validation criteria.

SECTION XVII: OVERALL ASSESSMENT

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVII-B for overall assessment data validation criteria.

Appendix E

VOA/SV Functional Guidelines Action Tables

Note: This appendix is a compilation of the data validation actions that appear in tabular format in Part II - VOLATILE/SEMIVOLATILE Data Validation Functional Guidelines. Other actions that are not presented in tabular format are not contained in this appendix and the validator must refer to Part II to obtain the complete set of actions.

APPENDIX E

Table VOA/SV-I-1:

QUALIFICATION OF VOLATILE ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION			TECHNICAL HOLDING TIMES			
Matrix	Refrig.& Light Protected	Acid Preserved	≤ 7 Days	7 < HT ≤ 14 Days	14 < HT ≤ 28 Days	> 28 Days
AQ	No	Yes or No	J - detects R-non-detects	J - detects R-non-detects	J - detects R-non-detects	J - detects R-non-detects
AQ	Yes	Yes	A	A	J - detects UJ-non-detects	J - detects R-non-detects
AQ	Yes	No	A	**Aromatics** J - detects R-non-detects **Non-aromatics** A - detects A-non-detects	**Aromatics** J - detects R-non-detects **Non-aromatics** J - detects UJ-non-detects	J - detects R-non-detects
S/S	No	N/A	J- detects R-non-detects	J - detects R-non-detects	J - detects R-non-detects	J - detects R-non-detects
S/S	Yes	N/A	A	A	J - detects UJ-non-detects	J - detects R-non-detects

Table VOA/SV-I-2:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION		TECHNICAL HOLDING TIMES		
Matrix	Refrig. & Light Protected	Extracted and/or Analyzed Within H.T.	Extracted and/or Analyzed Outside H.T.	If Extraction HT > 28 days and/or Analytical HT > 60 days
AQ and S/S	Yes	A	J - detects UJ - non-detects	J - detects R - non-detects
AQ and S/S	No	J - detects UJ - non-detects	J - detects UJ - non-detects	J - detects R - non-detects

Note: AQ = Aqueous, S/S = Soil/Sediment

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify or reject non-detects when Region I preservation and/or technical holding time criteria are not met.

For VOA aqueous samples containing excessive headspace (bubbles greater than 2 mm in diameter); J-detects, R-non-detects

Table VOA/SV-III-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON THE INITIAL CALIBRATION

Sample Results	**QC Criterion** $\overline{RRF} \geq 0.05$ %RSD ≤ 30.0%	**Situation 1** $\overline{RRF} < 0.05$ %RSD ≤ 30.0%	**Situation 2**** $\overline{RRF} \geq 0.05$ %RSD >30.0%	**Situation 3** $\overline{RRF} < 0.05$ %RSD > 30.0%
Detects	A	J	J	J
Non-detects	A	R	UJ	R

** See Table VOA/SV-III-2 for additional guidance.

Table VOA/SV-III-2:

EXPANDED INITIAL CALIBRATION VOA/SV ANALYTE QUALIFICATIONS

Sample Results	**Elimination of High or Low Calibration Points** %RSD > 30.0%	**Elimination of High Calibration Points** %RSD ≤ 30.0% RRF ≥ 0.05	**Elimination of Low Calibration Points** %RSD ≤ 30.0% RRF ≥ 0.05
Detects	J	A: On linear portion of curve J: On high end of curve outside linear portion	A: On linear portion of curve J: On low end of curve outside linear portion
Non-detects	UJ	A	UJ

Table VOA/SV-IV-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON THE CONTINUING CALIBRATION

Sample Results	**QC Criteria** RRF ≥ 0.05 %D ≤ ± 25.0%	**Situation 1** RRF < 0.05 %D ≤ ± 25.0%	**Situation 2** RRF ≥ 0.05 %D > ± 25.0%	**Situation 3** RRF < 0.05 %D > ± 25.0%
Detects	A	J	J	J

Non-Detects	A	R	UJ	R

Table VOA/SV-VI-1:

QUALIFICATION OF VOLATILE/SEMIVOLATILE ANALYTES BASED ON SURROGATE COMPOUND RECOVERIES

Surrogate Compound Recovery				
Sample Results	**one or more surrogates < 10%**	**one VOA, two B/N or two acid surrogates 10% ≤ %Rec < LL**	**all VOA, one B/N or one acid surrogate LL ≤ %Rec ≤ UL**	**one VOA, two B/N or two acid surrogates > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table VOA/SV-VII-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON INTERNAL STANDARD AREA COUNTS

Internal Standard Area Counts				
Sample Results	**Area Counts < 20% of associated calibration std. area**	**20% ≤ Area Counts < LL**	**LL ≤ Area Counts ≤ UL**	**Area Counts > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria based on associated calibration standard area
UL - Upper Limit of method QC acceptance criteria based on associated calibration standard area

Table VOA/SV-VIII-1:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MATRIX SPIKE RECOVERIES AND RPDs**

Sample Results	**Recovery < 10%**	**10% ≤ Recovery < Lower QC Limit**	**Lower QC Limit ≤ Recovery ≤ Upper QC Limit**	**Recovery > Upper QC Limit**	**RPD > QC Limit**
Detects	**J**	**J**	**A**	**J**	**J**
Non-detects	**R**	**UJ**	**A**	**A**	**UJ**

** Note that qualification and rejection generally are limited to the spiking compounds, however, the validator may use professional judgment to qualify or reject all positive detects or non-detects in the unspiked sample if the majority of spike compound recoveries and/or RPDs are outside the method QC acceptance criteria.

Table VOA/SV-VIII-2:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MS, MSD, AND UNSPIKED SAMPLE %RSD

Sample Results	%RSD ≤ 50%*	%RSD > 50%*	Two out of three sample results reported as non-detects
Detects	A	J	Professional Judgment
Non-detects	A	Professional Judgment	Professional Judgment

* If a non-detected result is reported for a compound in one of the samples in the MS, MSD or unspiked sample set, then the validator should use the sample quantitation limit value for that compound to calculate the %RSD.

Table VOA/SV-IX-1:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES - SITUATION 1: POSITIVE DETECTS IN BOTH FIELD DUPLICATES

Relative Percent Difference	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30 Non-Aqueous > 50%
Sample Results	Both duplicate sample concs. ≥ 2 X QL	QL ≤ both duplicate samples concs. < 2 X QL	One sample conc. ≥ 2 X QL QL ≤ Other sample conc. < 2 X QL
Detects	J	Professional Judgment	Professional Judgment
Non-detects	NA	NA	NA

* QL = Sample Quantitation Limit

Note: Qualification refers to field duplicate sample results only. Professional judgment may be utilized to apply field duplicate actions to all samples of the same matrix.

Table VOA/SV-IX-2:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES - SITUATION 2: POSITIVE DETECT IN ONLY ONE FIELD DUPLICATE**

Aqueous and Non-Aqueous		
Sample Results	One Sample Conc. = ND (or value reported as less than the QL) QL ≤ Other Sample Conc. < 2 X QL	One sample conc. = ND (or value reported as less than the QL) Other sample conc. ≥ 2 X QL
Detects	Professional Judgment	J
Non-detects	Professional Judgment	UJ

* QL = Sample Quantitation Limit

** RPD should not be evaluated for these duplicate pairs

Note: Qualification refers to field duplicate sample results only. Professional judgment may be utilized to apply field duplicate actions to all samples of the same matrix.

Table VOA/SV-X-1:

QUALIFICATION OF ORGANIC ANALYTES BASED ON MDL STUDY RESULTS

Sample Results	Mean % Recovery			
	%Rec < 10%	10% ≤ %Rec < 80%	80% ≤ %Rec ≤ 120%	%Rec > 120%
Detects	J	Professional Judgment*	A	Professional Judgment*
Non-Detects	R	Professional Judgment*	A	A

Sample Results	% RSD	
	> 25%	≤ 25%
Detects	Professional Judgment**	A
Non-detects	Professional Judgment**	A

* Taking into consideration LFB results.
** Taking into consideration initial calibration %RSDs.

Table VOA/SV-X-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE: ≤ ONE-HALF OF LFB COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	%Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target compounds at or below the quantitation limit.

Table VOA/SV-X-3:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE: > ONE-HALF OF LFB COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	%Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
All Detects	J	J	A	J

All Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target compounds at or below the quantitation limit.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table VOA/SV-XI-1:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON LCS RECOVERIES WHERE: ≤ ONE-HALF OF LCS COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec < LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table V/SV-XI-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LCS RECOVERIES WHERE: > ONE-HALF OF LCS COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS*

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec < LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table VOA/SV-XI-3:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON PES RESULTS WHERE: ≤ ONE-HALF OF PES COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	! Single Blind ! Double Blind PES < Lower Limit "Action Low"	! Single Blind ! Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	! Single Blind ! Double Blind PES > Upper Limit "Action High"
Detects	J	A	J
Non-Detects	R	A	A

Table VOA/SV-XI-4:

QUALIFICATION OF ORGANIC ANALYTES BASED ON PES RESULTS WHERE:
> ONE-HALF OF PES COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS *

Sample Results	! Single Blind ! Double Blind PES < Lower Limit "Action Low"	! Single Blind ! Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	! Single Blind ! Double Blind PES > Upper Limit "Action High"
All Detects	J	A	J
All Non-Detects	R	A	A

* Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table VOA/SV-XIII-1:

QUALIFICATION OF VOLATILE/SEMIVOLATILE ORGANIC ANALYTES BASED ON SAMPLE PERCENT SOLIDS

Sample Result	% Solids > 30%	10% ≤ % Solids ≤ 30%	% Solids < 10%
Detects	A	J	R
Non-detects	A	R	R

Table SV-XV-1:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON GPC CALIBRATION QUALITY CONTROL

	Criteria	Action
Peak Resolution	As per method QC acceptance criteria.	Professional Judgment
Peak Shape	Peak shapes must be symmetrical.	Professional Judgment

Retention Time Shift	Retention time shifts between GPC calibration checks must not exceed ± 5%.	Professional Judgment
GPC Instrument Blank	Target analytes must be < QL and surrogate compound recoveries and IS area counts and/or RTs (if added) must meet method QC acceptance criteria. (Note: CLP SOW OLM03.2 does not require the addition of surrogate compounds to the GPC instrument blank)	Refer to Section V for Blank Actions

Table VOA/SV-XI-2:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON GPC CLEANUP QUALITY CONTROL
WHERE: ≤ ONE-HALF OF GPC CALIBRATION CHECK COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec < LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table V/SV-XI-3:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON GPC CLEANUP QUALITY CONTROL
WHERE: > ONE-HALF OF GPC CALIBRATION CHECK COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec < LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

Note: Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Table SV-XV-4:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON SILICA GEL CLEANUP QUALITY CONTROL WHERE: ≤ ONE HALF OF SILICA GEL CHECK SOLUTION COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE CRITERIA

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec ≤ LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A
Silica Gel Column Blank	Target analytes must be < QL and surrogate compound recoveries and IS area counts and/or RTs (if added) must meet method QC acceptance criteria.			Refer to Section V for Blank Actions

Note: Professional judgment should be used in applying the guidance above to qualify or reject sample data.

LL - Lower Limit of method QC acceptance criteria.
UL - Upper Limit of method QC acceptance criteria.

Table V/SV-XI-5:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON SILICA GEL CLEANUP QUALITY CONTROL WHERE: > ONE-HALF OF SILICA GEL CHECK SOLUTION COMPOUNDS OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	**% Recovery**			
	%Rec < 10%	**10% ≤ %Rec < LL**	**LL ≤ %Rec ≤ UL**	**%Rec > UL**
All Detects	J	J	A	J

All Non-detects	R	UJ	A	A

Note: Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

PESTICIDE/PCB DATA VALIDATION FUNCTIONAL GUIDELINES - PART III

The requirements to be checked in validation are listed below. "CCS" indicates that the contractual requirements for these items will also be checked by Contract Compliance Screening (CCS). CCS requirements are not always the same as data validation criteria.

I. Preservation and Technical Holding Time ..... (CCS) Pest/PCB-I-1
II. GC/ECD Instrument Performance Check ..... (CCS) Pest/PCB-II-1
III. Initial Calibration ..... (CCS) Pest/PCB-III-1
IV. Calibration Verification ..... (CCS) Pest/PCB-IV-1
V. Blanks (Method Blanks Only) ..... (CCS) Pest/PCB-V-1
VI. Surrogate Analytes ..... (CCS) Pest/PCB-VI-1
VII. Pesticide/PCB Cleanup Pest/PCB-VII-1
VIII. Matrix Spike/Matrix Spike Duplicate ..... (CCS) Pest/PCB-VIII-1
IX. Field Duplicates Pest/PCB-IX-1
X. Sensitivity Check Pest/PCB-X-1
XI. PE Samples/Accuracy Check Pest/PCB-XI-1
XII. Target Analyte Identification Pest/PCB-XII-1
XIII. Analyte Quantitation and Reported Quantitation Limits Pest/PCB-XIII-1
XIV. System Performance Pest/PCB-XIV-1
XV. Overall Evaluation of Data Pest/PCB-XV-1

Appendices

Appendix F CLP SOW OLM04.3/Pesticide/PCB Analysis
Appendix G CLP SOW OLC03.2/Pesticide/PCB Analysis
Appendix H Pest/PCB Functional Guidelines Action Tables

I. PRESERVATION AND TECHNICAL HOLDING TIMES

A. OBJECTIVE

The objective is to ascertain the validity of the analytical results based on the preservation techniques which were used and the holding time of the sample from time of collection to time of sample preparation and sample analysis, as appropriate.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the Pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. REGION I PRESERVATION CRITERIA

SAMPLE TYPE	. PRESERVATION CODE
Pesticide/PCB Aqueous [a]	1,2,3
Pesticide/PCB Soil/Sediment [b]	1,3
Pesticide/PCB Sludge [b]	1,3
Oily Waste [b]	1,3
Biological Tissue [d]	3,4
Pest/PCB Air (PUF, Filters) [c]	3,5
Pest/PCB Wipes [c]	1,3
Pest/PCB Fly Ash [b]	1,3

Preservation Code:

1. Cool @ 4°C (± 2°)
2. Adjust to a pH range of 5.0 to 9.0 with NaOH or H_2SO_4 if the sample will not be extracted within 72 hours
3. Protect from light
4. Freeze at <-20°C
5. Room Temperature (Avoid excessive heat)

2. REGION I TECHNICAL HOLDING TIME CRITERIA

SAMPLE TYPE	CRITERIA
Pesticide/PCB Aqueous[a]	Extraction of properly preserved aqueous samples by liquid-liquid procedures must be completed within 7 days of sample collection. Extraction of properly preserved aqueous samples by separatory funnel or solid phase extraction (SPE) must be completed within 7 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Pesticide/PCB Soil/Sediment [b]	Extraction of properly preserved soil/sediment samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Pesticide/PCB Sludge [b]	Extraction of properly preserved soil/sediment samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Pesticide/PCB Oily Waste [b]	Extraction of properly preserved oily waste samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Pesticide/PCB Biological Tissue [d]	If the samples are not frozen, extraction of tissue samples must be completed within 14 days of sample collection. Frozen samples (≤-20°C) may be held for up to one year if stored at ≤-20°C. The tissue must remain frozen prior to and during homogenization. Extracts must be analyzed within 40 days following sample extraction.
Pesticide/PCB Air [c]	Analyses of properly preserved air samples must be completed within 14 days of sample collection. Pre-cleaned and certified air collection devices, i.e., PUFs, Florisil cartridges, and filters, must be utilized for sample collection within the method-specified time frame.

SAMPLE TYPE	CRITERIA
Pesticide/PCB Wipes [c]	Extraction of properly preserved wipe samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Pesticide/PCB Fly Ash [b]	Extraction of properly preserved fly ash samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from preservation and/or holding time anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Preservation Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form) laboratory tracking/storage forms, and the data package narrative to verify that samples were properly preserved by the sampler and the laboratory maintained preservation according to the preservation criteria on page Pest/PCB-I-1. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information.	1. Preservation If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume that the samples were not preserved and should document on the holding time worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify or reject the sample data. If the samples were not preserved properly in the field and/or if the laboratory failed to properly maintain sample preservation, then the validator should take the following actions:

C. EVALUATION	D. ACTION
1 a. Verify that samples were refrigerated or frozen (as required) and protected from light according to Region I preservation criteria.	1. a. If samples for aqueous and soil/sediment matrices were not refrigerated and/or protected from light according to Region I preservation criteria on page Pest/PCB-I-1, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples, regardless of whether or not technical holding time criteria were met. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when temperature and light protection preservation criteria were not met. Professional judgment should be used when the laboratory has reported transportation cooler temperatures that slightly exceed the upper limits of the preservation criteria (>+6°C). In this case, the laboratory procedure for monitoring cooler temperature may be in question. In this event, the validator should document all justifications for qualifying data or not qualifying data in the Data Validation Memorandum.
2. Technical Holding Times a. Verify that pesticide/PCB samples were extracted within technical holding time criteria. Establish extraction holding times by comparing sampling dates reported on the EPA Traffic Reports (TRs) and/or Chain of Custody (COC) Forms with dates of extraction reported on tabulated result forms. i. Verify that liquid-liquid extractions for pesticide/PCB aqueous samples were begun within 7 days of sample collection. ii. Verify that aqueous pesticide/PCB extractions by separatory funnel were completed within 7 days of sample collection. iii. Verify that aqueous pesticide/PCB extractions by solid phase extraction (SPE) or other extraction technique were completed within 7 days of sample collection.	2. Technical Holding Times a. If aqueous and soil/sediment pesticide/PCB samples were properly preserved, but the technical extraction holding time criteria were exceeded, then the validator should estimate (J) positive detects and estimate (UJ) non-detects. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when technical holding times are exceeded. For all matrices except frozen biological tissue, if pesticide/PCB extraction holding time criteria were grossly exceeded (> 28 days), then the validator should estimate (J) positive detects and use professional judgment to qualify (UJ) or reject (R) non-detects. The validator should take into account the environmental stability of Aroclors when validating the sample data.

C. EVALUATION	D. ACTION
iv. Verify that pesticide/PCB soil/sediment sample extractions by sonication or soxhlet procedures were completed within 14 days of sample collection. v. Verify that samples of other matrices, i.e., wipes, biological tissue were extracted within the Region I holding time criteria.	
2. b. Verify that pesticide/PCB samples and/or extracts (as required) were analyzed within technical holding time criteria for analysis. Establish analytical holding times by comparing collection and/or extraction dates (as required) and analysis dates reported on tabulated result forms.	2. b. If aqueous and soil/sediment pesticide/PCB samples were properly preserved, but the technical analytical holding time criteria were exceeded, then the validator should estimate (J) positive detects and estimate (UJ) non-detects. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when technical holding times are exceeded. For all matrices, if pesticide/PCB analytical holding time criteria were grossly exceeded (> 60 days), then the validator should estimate (J) positive detects and use professional judgment to qualify (UJ) or reject (R) non-detects. The validator should take into account the environmental stability of Aroclors and pesticides when validating the sample data.
* c. Check the raw data including extraction and instrument run logs to verify reported sample extraction and analysis dates.	c. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation: C.2.c**

Table Pest/PCB-I-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION		TECHNICAL HOLDING TIMES		
Matrix	Refrig.& Light Protected	Extracted and/or Analyzed Within H.T.	Extracted and/or Analyzed Outside H.T.	If Extraction HT > 28 days and/or Analytical HT > 60 days
AQ and S/S	Yes	A - acceptable results	J - detects UJ - non-detects	J - detects Professional Judgment to either UJ or R - non-detects*
AQ and S/S	No	J - detects UJ - non-detects	J - detects UJ - non-detects	J - detects Professional Judgment to either UJ or R - non-detects*

Note: AQ = Aqueous, S/S = Soil/Sediment

* The validator may use professional judgment to qualify or reject non-detected pesticides and multicomponent analytes based on their environmental stability.

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify (UJ) or reject (R) the non-detects when Region I preservation and/or technical holding time criteria are not met. The results are acceptable (A) when the criteria are met.

E. EXAMPLES

Example #1: (Proper preservation; Extraction holding times grossly exceeded)

PCB soil sample SAA44 was collected on 12/1/95 and received at the laboratory on 12/2/95. Upon examination of the Traffic Report, laboratory receipt information, and sample tracking records, the validator determines that the sample was properly preserved at 4°C and was light protected. The validator examines the sample extraction log sheet and discovers that the sample was extracted by the soxhlet procedure on 12/30/95, 29 days after sample collection. The validator estimates (J) the positive Aroclor 1260 detect in sample SAA44 and uses professional judgment to estimate (UJ) the Aroclor non-detects on the Data Summary Tables due to the grossly exceeded extraction holding times. The validator discusses these qualifications in the Data Validation Memorandum.

Example #2: (Proper preservation; Analytical holding times exceeded)

Pesticide soil sample SAA34 was analyzed by routine analysis following CLP SOW OLM04.3. This sample was received by the laboratory on 4/10/95. Review of the Traffic Report, the extraction log sheet and the run log sheet revealed that the sample was collected on 3/29/95, was extracted on 4/11/95, and was analyzed on 6/9/95. The laboratory documentation indicates that the sample was properly preserved at 4°C and protected from light. Only 4,4'DDT and dieldrin were detected. Although the analytical holding time exceeded the 40 day criteria, it was not "grossly exceeded" and was analyzed within 60 days of extraction. Therefore, the validator estimates (J) the positive detects for 4,4'-DDT and dieldrin and uses professional judgment to estimate (UJ) the non-detects. The validator discusses these qualifications in the Data Validation Memorandum.

Example #3: (Improper preservation; Holding times met)

Pesticide/PCB water samples SAA99 - SAA106 were analyzed by routine analysis following CLP SOW OLM04.3. The validator examines the Traffic Report and the sample log-in sheets contained in the data package. The sampler properly preserved and shipped the samples at 4°C, however, the laboratory notes in the data package narrative that the samples were removed from the cooler and left in a hood for 36 hours after sample receipt due to a misunderstanding between shifts.

The validator reviews the chain-of-custody form, the extraction log and the run log data. The sampling date for samples SAA99 - SAA106 was 12/1/95, the extraction date was 12/5/95 and the analysis date was 12/15/95. Since all holding time criteria were met, but preservation criteria were not, the validator estimates (J) the positive pesticide and PCB detects and estimates (UJ) the pesticide and PCB non-detects on the Data Summary Table and discusses these qualifications in the Data Validation Memorandum.

REFERENCES

a - 40 CFR, Part 136, Appendix A, 600 Series

b - SW 846, 8000 Series

c - Region I policy

d - Evaluation of Dredged Material for Discharge in Waters of the U.S. - Testing Manual, EPA 823-B-97-001, February 1997, and QA/QC Guidance for Sampling and Analysis of Sediments, Waters, and Tissue for Dredged Material Evaluations, Chemical Evaluation, EPA 823-B-95-001, April 1995.

II. GC/ECD INSTRUMENT PERFORMANCE CHECK

A. OBJECTIVE

Performance checks on the gas chromatograph/electron capture detector (GC/ECD) system are performed to ensure adequate chromatographic resolution, column performance and to check the accuracy of the initial calibration. The Resolution Check Mixture (RCM) is analyzed at the beginning of the initial calibration sequence to ensure that the GC column can adequately resolve target analytes. The Performance Evaluation Mixture (PEM) is analyzed at the beginning (following the RCM) and at the end of the initial calibration sequence and is also analyzed during the continuing calibration verification. The PEM is analyzed to assess chromatographic resolution, pesticide degradation, and to check the accuracy of the initial calibration for the analytes in the PEM.

B. CRITERIA

GC/ECD Instrument Performance criteria are not sample specific. Since conformance is determined using standard materials, these criteria should be met under all circumstances. The following validation criteria are based on the CLP-Pesticide/PCB method QC acceptance criteria as listed in Appendix F. These criteria should be used as the default criteria when none exist for the Pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method. Any deviations, modifications or non-CLP method-specific QC acceptance criteria must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. a. Adequate chromatographic resolution of GC peaks must be determined by analyzing a Resolution Check Mixture (RCM) and the Performance Evaluation Mixture (PEM) at the frequency, concentration, and composition stated in the method.

   b. The chromatographic resolution between any two adjacent peaks in the RCM must be greater than or equal to 60.0 percent. The chromatographic resolution between any two adjacent peaks in the PEM must be greater than or equal to 90.0 percent on each column.

2. Retention times of each of the single component pesticides and surrogates in the Resolution Check Mixture and Performance Evaluation Mixture standards must be within the calculated retention time windows.

3. The percent difference (%D) between the calculated amount and the nominal amount (amount added) for each of the single component pesticides and surrogates in the Performance Evaluation Mixture (PEM) must not exceed ± 25.0 percent for both GC columns.

4. The degree of pesticide degradation must be determined for each column used to analyze field samples. The percent breakdown is the amount of decomposition that 4,4'-DDT and endrin undergo when the PEM is analyzed on the GC column.

   a. For 4,4'-DDT, the percent breakdown is determined by the presence of 4,4'-DDD and/or 4,4'-DDE in the GC chromatogram.

      i. The percent breakdown for 4,4'-DDT in each PEM must be less than or equal to 20.0 percent for both GC columns.

b. For endrin, the percent breakdown is determined by the presence of endrin aldehyde and/or endrin ketone in the GC chromatogram.

ii. The percent breakdown for endrin in each PEM must be less than or equal to 20.0 percent for both GC columns.

c. Total breakdown for 4,4'-DDT and endrin is determined by adding these results.

iii. The combined percent breakdown for 4,4'-DDT and endrin in each PEM must be less than or equal to 30.0 percent for both GC columns.

5. The criteria mentioned above apply to the results from both chromatographic columns. In the event that one GC column meets criteria and the other does not, the validator may use professional judgement to evaluate the effect of the noncompliance on the associated samples. The validator may choose to accept a sample result reported from the compliant GC column. The validator must consider if the noncompliant GC column criteria were grossly exceeded and whether or not false negative or false positive results may be present.

The equations used to verify these calculations are provided in Appendix F.

Note: CLP SOW-OLM04.3 does not require the analysis of a multicomponent analyte performance check standard. If a multicomponent analyte performance check standard is required by a non-CLP method, then the above retention time criteria, resolution criteria, and % Difference criteria used to evaluate single component pesticides should be used to evaluate instrument performance for multicomponent analytes.

C. EVALUATION/ D. ACTION

The following evaluation procedures and actions are specific to CLP-SOW OLM04.3 data and can be modified for use in evaluating and qualifying non-CLP data.

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from GC/ECD instrument performance anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. **RESOLUTION CHECK** If resolution acceptance criteria are not achieved, quantitative and qualitative results may not be accurate due to coelution problems.	1. **RESOLUTION CHECK - Worksheet Pest/PCB-II-A**
Resolution Check Mixture:	Resolution Check Mixture:
a. Verify from Form VI PEST-4 that the Resolution Check Mixture (RCM) contained the required analytes and that the resolution between two adjacent peaks is greater than or equal to 60.0% on both GC columns. The required analytes are listed in Appendix F-1, Section II.	a. If the Resolution Check Mixture does not contain the correct analytes and/or they were not adequately resolved, then the validator should use professional judgment to accept, qualify or reject sample data taking into consideration the resolution results of the IND A/B standards and the PEMs analyzed in association with the samples.
b. Verify from Form VIII PEST that the RCM was analyzed at the beginning of the initial calibration sequence on each GC column and instrument used for analysis.	b. If the RCM was not analyzed in the correct sequence and at the proper frequency, then the validator should use professional judgment to determine the effect on the sample data.

C. EVALUATION	D. ACTION
1. **RESOLUTION CHECK - continued**	1. **RESOLUTION CHECK - continued**
* c. Examine the RCM chromatograms and raw data to verify that the resolution between two adjacent peaks for the required analytes is greater than or equal to 60.0% on both GC columns. Using equations found in Appendix F, recalculate the resolution between two RCM analytes to verify correct resolution calculations.	c. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used must be documented in the Data Validation Memorandum.
Performance Evaluation Mixture:	Performance Evaluation Mixture:
d. Verify from the Form VII PEST-1 that the Performance Evaluation Mixture (PEM) contained the proper analytes at the required concentrations. The analytes are listed in Appendix F-2, Section II.	d. If the PEM does not contain the correct analytes at the required concentrations and/or was not analyzed in the required sequence at the proper frequency, then the validator should use professional judgment to determine the effect on the sample data.
e. Verify from Form VIII PEST that the PEM was analyzed after the RCM in the initial calibration sequence and at the proper frequency throughout the analytical sequence.	e. If the PEM was not analyzed in the required sequence and at the proper frequency, then the validator should use professional judgment to determine the effect on the sample data.

C. EVALUATION	D. ACTION
1. f. Verify from Form VI PEST-5 that the resolution of all single component pesticide and surrogate peaks is greater than or equal to 90.0% on both GC columns.	1. f. i. If PEM analytes were not adequately resolved on both GC columns, then the validator should estimate all affected analytes positive (J) and non-detected (UJ) results. ii. If PEM analytes were not adequately resolved on one of the GC columns, then qualification may be necessary. If a tentatively identified positive result from the compliant column must be confirmed on the second but non-compliant column, then the positive result should be estimated (J). If resolution issues are noted in the PEM, qualification of positive results may not be necessary if only one analyte of the unresolved pair is present and if the positive result is reported from the compliant GC column. Professional judgment must be applied to evaluate whether or not data are acceptable, estimated (J) or rejected (R).
* g. Review the PEM raw data from the initial calibrations to verify that the resolution between adjacent peaks is greater than or equal to 90.0% on both GC columns. Using equations in Appendix C, recalculate the resolution between two PEM analytes to verify correct resolution calculations.	g. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* h. Review the Pesticide Standards Preparation Log entries to verify that the RCM solutions and PEM solutions contained the method required analytes at the required concentrations. The required concentrations are listed in Appendix F.	h. If the RCMs and PEMs did not contain the proper analytes at the required concentrations, then the validator should use professional judgment to determine the effect on the sample data.

C. EVALUATION	D. ACTION
2. **RETENTION TIME CHECK**	2. **RETENTION TIME CHECK - Worksheet Pest/PCB-II-B**
a. Check Form VII PEST-1 to verify that the absolute retention times for the PEM analytes in both PEM analyses in the initial calibration are within the calculated retention time windows based on the mean RT from the three-point initial calibration on each column. The retention time windows are from Table App. F.III-3 and page F-5 in the Appendix.	a. Retention time windows are used in qualitative identification. If the retention times of the pesticides in the PEM do not fall within the established retention time windows, then the associated sample results should be carefully evaluated for the possibility of false positives and false negatives. All samples injected after the last in-control standard are potentially affected and a Tier III level of validation should be performed to assess the impact on sample results.
* b. Review the raw data for samples analyzed after the last compliant PEM to assess the possibility of false positives and false negatives. Evaluate whether or not the sample chromatograms have any peaks which are close to any target pesticides retention time windows. These peaks could indicate qualitative inaccuracies.	b. If no peaks are present either within or close to expected retention time windows of a target pesticide, then non-detected values can be considered valid. If sample chromatograms contain peaks either close to or within the expected retention time window of target pesticides, then two options are available to the validator to determine the impact on the data: **Option 1** - In some cases, additional effort by the validator may be necessary to determine if sample peaks represent the pesticides of interest. For example: i. The validator should examine the data package for the presence of three or more standards containing the pesticides of interest that were run within a 72-hour period during which that sample was analyzed. ii. If three or more such standards are present, revised retention time windows may be created by utilizing the mean retention time as an absolute retention time, and using the windows listed in Table App.F.III-3, or using 3x the standard deviation calculated from the retention times in the standards, as appropriate for the analytical run conditions.

C. EVALUATION	D. ACTION
2. b. Continued from above.	**Option 1** - Continued from above. iii. If all standards, surrogates, and matrix spikes fall within the revised window, then the validity of the positive or non-detected sample results can be re-evaluated using the revised retention time window. iv. The Data Validation Memorandum must describe the data validation procedures performed by the validator and the impact on data usability. In addition, the supporting documentation should contain all calculations and comparisons generated by the validator. **Option 2** - If the validator cannot resolve the retention time problems at issue with the available data, then all positive detects and the sample quantitation limits for non-detects should be rejected (R).
* c. Check the PEM raw data from the initial calibrations to verify that the correct absolute retention times for the PEM analytes in each PEM analysis have been transcribed correctly and are within the calculated retention time windows based on the mean RT from the three-point initial calibration using the values shown in Table App. F.III-3.	c. If transcription and/or calculation errors are extensive, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used must be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. **ACCURACY (%D) CHECK** a. Verify from Form VII PEST-1 that the %D between the calculated amount and the nominal amount for each of the single component pesticides (and, if applicable, multicomponent standards) in both PEMs in the initial calibration sequence does not exceed ± 25.0 % on each GC column.	3. **ACCURACY (%D) CHECK - Worksheet Pest/PCB-II-C** a. i. If %D criteria are not met on both columns, then the validator should: ● Estimate (J) associated positive detects for the affected analyte for all samples associated with the unacceptable PEM. ● Estimate (UJ) sample quantitation limits for non-detects in samples associated with the non-compliant PEM. ii. If %D criteria are not met on only one of the columns, then the validator may choose to accept the non-detects and positive detects if they are reported from the GC column with the compliant %D. The validator should use professional judgement to estimate (J) and/or reject (R) sample data if %D criteria were grossly exceeded on one or both columns and considering the possibility of false negatives or false positives.
* b. Check and recalculate 10% of the PEM percent difference data. Verify that the recalculated values agree within 10% of the laboratory reported values.	b. If more than 10% of the calculations are in error, then the validator should perform a more comprehensive review to determine the extent of the problem. If the problem is extensive, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
4. **PESTICIDE DEGRADATION CHECK**	4. **PESTICIDE DEGRADATION CHECK - Worksheet Pest/PCB-II-D**
a. Verify from Form VII PEST-1 that the individual breakdown for 4,4'-DDT in both PEM analyses in the initial calibration sequence is less than or equal to 20.0%.	a. If 4,4'-DDT breakdown is greater than 20.0%, then the validator should: **4,4'-DDT Detected** i. Estimate (J) DDT, DDD, DDE positive detects and accept DDD and DDE non-detects for samples associated with the unacceptable PEM. **4,4'-DDT Not Detected** ii. Reject (R) DDT non-detects as unusable, accept DDD, DDE non-detects, and estimate (J) DDD, DDE positive detects and note the potential high bias for these two analytes. If the results from a second, compliant GC column are available, evaluate these results to determine to what extent DDD and DDE positive detects are due to DDT breakdown and use professional judgement to accept, estimate (J), or reject (R) results.
b. Verify from Form VII PEST-1 that the individual breakdown for endrin in both PEM analyses in the initial calibration sequence is less than or equal to 20.0%.	b. If endrin breakdown is greater than 20.0%, then the validator should: **Endrin Detected** i. Estimate (J) endrin, endrin ketone, endrin aldehyde positive detects, and accept endrin ketone, endrin aldehyde non-detects for samples associated with the unacceptable PEM. **Endrin Not Detected** ii. Reject (R) endrin non-detects, accept endrin ketone, endrin aldehyde non-detects, and estimate (J) endrin ketone endrin aldehyde positive detects and note the potential high bias for these two analytes. If the results from a second, compliant GC column are available, evaluate these results to determine to what extent endrin ketone and endrin aldehyde positive detects are the due to endrin breakdown and use professional judgement to accept, estimate (J), or reject (R) results.

C. EVALUATION	D. ACTION
4. c. Verify from Form VII PEST-1 that the combined breakdown for 4,4'-DDT and endrin in both PEM analyses is less than or equal to 30.0%.	4. c. If the combined 4,4'-DDT and endrin breakdown is greater than 30.0%, then the validator should: **4,4'-DDT Detected** i. Estimate (J) DDT, DDD, DDE positive detects and accept DDD and DDE non-detects for samples associated with the unacceptable PEM. **4,4'-DDT Not Detected** ii. Reject (R) DDT non-detects as unusable, accept DDD, DDE non-detects, and estimate (J) DDD, DDE positive detects and note the potential high bias for these two analytes. **Endrin Detected** iii. Estimate (J) endrin, endrin ketone, endrin aldehyde positive detects, and accept endrin ketone, endrin aldehyde non-detects for samples associated with the unacceptable PEM. **Endrin Not Detected** iv. Reject (R) endrin non-detects, accept endrin ketone, endrin aldehyde non-detects, and estimate (J) endrin ketone endrin aldehyde positive detects and note the potential high bias for these two analytes. If the results from a second, compliant GC column are available, evaluate these results to determine to what extent DDD, DDE, endrin ketone, and endrin aldehyde positive detects are the due to DDT and endrin breakdown and use professional judgement to accept, estimate (J), or reject (R) results.

C. EVALUATION	D. ACTION
*4. d. Check and recalculate 10% of the DDT and endrin breakdown data. Verify that the recalculated values agree within 10% of the laboratory values.	4. d. If more than 10% of the calculations are in error, then the validator should perform a more comprehensive review to determine the extent of the problem. If the problem is extensive, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.1.c, C.1.g, C.1.h, C.2.b, C.2.c, C.3.b, C.4.d

Table Pest/PCB-II-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON THE RESOLUTION CHECK MIXTURE (RCM) - Resolution Check

Sample Results	Resolution ≥ 60.0%	Resolution < 60.0%
Detects	A	Professional Judgment
Non-Detects	A	Professional Judgment

Table Pest/PCB-II-2:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON THE PERFORMANCE EVALUATION MIXTURE (PEM) - Resolution Check

Sample Results	Resolution ≥ 90.0%	Resolution < 90.0%
Detects	A	J
Non-detects	A	Professional Judgment

Table Pest/PCB-II-3:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON THE PERFORMANCE EVALUATION MIXTURE (PEM) - CALIBRATION CHECK - Accuracy Check

Sample Results	%D ≤ ±25.0%	%D > ±25.0%	One column meets criteria but the other exceeds
Detects	A	J	Professional judgement
Non-Detects	A	UJ	Professional judgement

Table Pest/PCB-II-4:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON 4,4'-DDT/ENDRIN BREAKDOWN - PESTICIDE DEGRADATION CHECK

Sample Results	4,4'-DDT Breakdown ≤ 20.0%	4,4'-DDT Breakdown > 20.0% and 4,4'-DDT detected	4,4'-DDT Breakdown > 20.0% and 4,4'-DDT not detected	Endrin Breakdown ≤ 20.0%	Endrin Breakdown > 20.0% and Endrin detected	Endrin Breakdown > 20.0% and Endrin not detected	Combined Breakdown ≤ 30.0%	Combined Breakdown > 30.0% and 4,4'-DDT and/or Endrin detected	Combined Breakdown > 30.0% and 4,4'-DDT and/or Endrin not detected
4,4'-DDT	A	J	R (NDs)	N/A	N/A	N/A	A	J	R (NDs)
DDD	A	J (detects) A (NDs)	J (detects) A (NDs)	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)
DDE	A	J (detects) A (NDs)	J (detects) A (NDs)	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)
Endrin	N/A	N/A	N/A	A	J	R (NDs)	A	J	R (NDs)
Endrin Aldehyde	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)	A	J (detects) A (NDs)	J (detects) A (NDs)
Endrin Ketone	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)	A	J (detects) A (NDs)	J (detects) A (NDs)

N/A = Not Applicable
J = Estimate result
R (NDs) = Reject non-detects
A (NDs) = Accept non-detects

Note: The validator must always discuss negative and positive bias in sample data in the Data Validation Memorandum.

E. EXAMPLES

Example #1: Resolution (Non-compliant RCM; Compliant PEM)

The resolution between 4,4'-DDE and dieldrin is 59.0% in the RCM. The validator reviews the PEM results, which were analyzed following the RCM to determine if the resolution problem is specific to the RCM analysis or to the entire analytical sequence run. The resolution between the PEM analytes was found to be acceptable and the validator uses professional judgment to accept the sample data associated with the RCM.

Example #2: (Retention Time Evaluation)

Historically heptachlor has been found at Site X and is a contaminant of concern. The RPM requested a Tier II validation and was concerned that heptachlor was not identified in any of the field samples. A Tier III validation was subsequently requested to verify that heptachlor was not present in the samples. The retention time window for heptachlor is 13.30 - 13.40, and a peak that elutes during that retention time window may be considered to be a positive detect for heptachlor if confirmed by second column analysis. A positive identification for heptachlor is obtained on the first column but not on the second. The validator reviews the second column chromatograms and computer print-outs to check if a peak eluted just prior to or after the retention time window on the secondary column (15.30 - 15.40). The validator notes that there is a peak at retention time 15.28 in many of the field samples which he suspects may be heptachlor. To investigate this, the validator reviews the standards and PEMs which were analyzed during the same time period as the samples in question. The mean and standard deviation of the retention time window are re-evaluated. All the standards and matrix spikes fall within the revised windows, and the validator redetermines sample results using these revised windows and reports the presence of heptachlor in the field samples. The validator documents all extra efforts and calculations in the Data Validation Memorandum.

Example #3: (Non-compliant %D)

The validator reviews the initial calibration data and notes that on one column the PEMs analyzed before and after the initial three point calibration standard analyses had %Ds for endrin of 36.0% and 41.0%, respectively. The validator reviews the subsequent continuing calibration Individual Standards A and B data analyzed 12 hours later and determines that endrin had a %D of 25.0% and was compliant. The validator estimates (J) the positive endrin detects and estimates (UJ) the endrin non-detects on the Data Summary Table for the samples analyzed following the non-compliant initial calibration PEMs and before the compliant Individual Standards A and B.

E. EXAMPLES

Example #4: (Pesticide Degradation)

The validator reviews the DDT/Endrin breakdown data and notes that the DDT breakdown in the PEM, which was analyzed after the 3 point calibration Individual Standards A and B curve, is 45.0%. The validator then reviews the subsequent PEM analyzed 12 hours later and notes that the DDT breakdown is less than 20.0% and is acceptable. For samples that have positive DDT detects; the validator estimates (J) the DDT detects, and estimates (J) positive DDD and DDE detects and accepts DDD and DDE non-detects for all samples analyzed following the non-compliant PEM and before the compliant PEM. For samples that have DDT non-detects; the validator rejects (R) the DDT non-detects and estimates (J) DDD and/or DDE detects and accepts DDD and DDE non-detects. It is noted in the Data Validation Memorandum that when the breakdown for 4,4'-DDT is high, the values for 4,4'-DDT are potentially biased low and the values for 4,4'-DDD and 4,4'-DDE are potentially biased high.

III. INITIAL CALIBRATION

A. OBJECTIVE

Compliance requirements for initial calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Initial calibration data demonstrate that the instrument is capable of satisfactory performance at the beginning of the analytical sequence by producing a linear calibration curve.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the Pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. Single Component Pesticides

   a. Calibration standard mixtures containing all of the single component pesticides and surrogates must be analyzed at low, mid, and high concentration levels during the initial calibration sequence, on each GC column and instrument used for analysis prior to the analysis of any field samples or blanks. If the continuing calibration method QC acceptance criteria are not achieved, an Initial Calibration must be performed.

      The low concentration standard must be at or below the quantitation level for each analyte; the midpoint concentration must be 4 times the low concentration standard; and the high concentration standard must be at least 16 times the low concentration standard, but a higher concentration may be chosen. See Appendix F, Table App. F. III-1.

   b. The chromatographic resolution between any two adjacent peaks in the midpoint concentration of the calibration standard mixtures in the initial calibration must be greater than or equal to 90.0 percent on each column. See Appendix F-1, Section II.

   c. The absolute retention times for each of the single component pesticides and surrogates are determined from an initial three point calibration. A list of the windows and an example for calculating retention time windows are provided in Appendix F, Table App. F.III-3.

   d. Calibration factors for single component pesticides are calculated for each of the three standard concentrations. The midpoint calibration standard is used for sample quantitation. (The instrument linearity is checked by calculating the %RSD; see Appendix F-8, Section III. The calculation uses the mean calibration factor.) Calibration factors can be calculated using either peak area or peak height. However, the calculation procedure, peak height or area, must be consistent for field, QC, and blank sample calculations.

   e. The Percent Relative Standard Deviation (%RSD) of the calibration factors for each of the single component pesticides in the initial calibration on both columns for the calibration standards mixtures must be less than or equal to 20.0 percent, except for alpha-BHC and delta-BHC which must be less than or equal to 25 percent. The %RSD of the calibration factors for the surrogates must be less than or equal to 30.0 percent.

f. The chromatograms that result from the analyses of the Resolution Check Mixture, the PEM, and Individual Standard Mixtures A and B during the initial calibration sequence must display the single component analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

g. The chromatograms for at least one of the three analyses each of Individual Standard Mixtures A and B from the initial calibration sequence must display the single component analytes at greater than 50 percent and less than 100 percent of full scale.

h. For any standard containing alpha-BHC, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

2. Multicomponent Analytes

a. The multicomponent analytes (the 7 Aroclors and Toxaphene) must each be analyzed separately (except Aroclors 1016 and 1260 which may be analyzed together in the same mixture) at a minimum of one concentration level during the initial calibration sequence on each GC column and instrument used for analysis prior to the analysis of any field samples or blanks. If the continuing calibration method QC acceptance criteria are not achieved an Initial Calibration must be performed (for concentrations, see Appendix F, Table App.F.III-2). The pesticide surrogates must be analyzed along with the multicomponent target analytes.

b. The absolute retention times for 3 to 5 peaks for each multicomponent analyte are determined from the initial calibration based on a minimum of at least one concentration point. A window of ±0.07 minutes is used to calculate the retention time windows for each of the 3 to 5 peaks from the multicomponent analyte standard.

c. i. Calibration factors for multicomponent analytes are calculated based on a one point standard concentration. Calibration factors can be calculated using either peak area or peak height. However, the calculation procedure, peak height or area, must be consistent for field, QC and blank sample calculations.

ii. Calibration factor data generated from the multicomponent analyte standard must be provided for each of the 3 to 5 peaks used to quantitate that multicomponent analyte in the field, QC and blank samples.

d. If a multi-point calibration is analyzed for a multicomponent analyte (not required in CLP OLM04.3), then the %RSD of each of the 3 to 5 identifying peaks must be less than or equal to 25.0 percent on both columns. The %RSD of the calibration factors for the surrogates must be less than or equal to 30.0 percent.

e. The chromatograms of the multicomponent analyte standards analyzed during the initial calibration sequence must display the peaks chosen for identification of each analyte at greater than 25 percent and less than 100 percent of full scale deflection.

3. The criteria mentioned above apply to the results from both chromatographic columns. In the event that one GC column meets criteria and the other does not, the validator may use professional judgement to evaluate the effect of the noncompliance on the associated samples. The validator may choose to accept a sample result reported from the compliant GC column. The validator must consider whether the noncompliant GC column criteria were grossly exceeded, and the possibility of false negatives or false positives.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from initial calibration anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Single Component Pesticides	1. Single Component Pesticides
a. i. Verify from Form VIII PEST that the Individual Standard Mixtures A and B were analyzed at the required frequency and in the proper sequence on each GC column and instrument used for analysis.	a. i. If Individual Standard Mixtures A and B were not analyzed at the required frequency and in the proper sequence on each GC column and instrument, then professional judgment must be used to evaluate the effect of the non-compliance on the sample data. This non-compliance should be noted in the Data Validation Memorandum (Worksheet Pest/PCB-III).
* ii. Review the raw data to verify that analysis times were accurately reported for the initial calibration standards on Form VIII PEST.	ii. If the laboratory has made transcription errors, then the validator should contact the laboratory to obtain corrected data and forms.
b. i. Verify from the Forms VI PEST-1 and PEST-2 that the low point standard concentrations correspond to the quantitation limit for each analyte; that the midpoint standard concentration is 4 times the low point; and that the high point is at least 16 times the low point.	b. i. If the Individual Standard Mixtures A and B were not analyzed at the required concentration levels, then the sample data may be adversely affected. The validator must use professional judgment to determine the severity of the effect on the linear range of the instrument and the resultant sample data and these data should be qualified accordingly (Worksheet Pest/PCB-III).

C. EVALUATION	D. ACTION
*1. b. ii. Review the raw data to verify that the low point standard concentrations, the midpoint standard concentrations, and the high point standard concentrations were accurately reported on Forms VI PEST-1 and PEST-2.	1. b. ii. If the laboratory has made transcription errors in the recording the concentrations of the standards, then the validator should have the laboratory resubmit all corrected raw data and forms.
iii. Verify from Forms VI PEST-1 and PEST-2 that all the required analytes are reported for the initial calibration from the Individual Standard Mixtures A and B.	iii. If errors are detected in the reporting of all the required analytes, then the validator should perform a more comprehensive review that includes the review of raw data as described in C.1.b.iv.
* iv. Review the raw data to verify that all the required analytes were analyzed in the standards and were accurately reported on Form VI PEST-1 and PEST-2.	iv. If the laboratory has made transcription errors, then the validator should have the laboratory resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be estimated (J) or rejected (R). If the required analytes were not analyzed in the initial calibration, then the sample data may be adversely affected. The accuracy and quantitation of the affected analyte(s) are questionable. The sample data associated with the initial calibration should be rejected (R), unless an acceptable alternate method of quantitation or detection limit determination was used. The validator must use professional judgement to determine whether the sample data should be accepted (A), estimated (J) or rejected (R). A discussion of the reasons for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
1. c. i. Verify from Forms VI PEST-6 and -7 that resolution is greater than or equal to 90.0 percent for any two adjacent peaks in the mid-point concentration of the Individual Standard Mixtures A and B in the initial calibration on both columns.	1. c. i. If resolution criteria are not met, then the quantitative results may not be accurate due to peak overlap and inadequate resolution. Positive detects for analytes that were not adequately resolved should be estimated (J). Qualitative identifications may be questionable if coelution exists. Non-detects that elute in the region of coelution may not be valid depending upon the extent of the coelution problem and professional judgment should be used to accept (A), estimate (J), or reject (R) non-detects as unusable. Refer to Section II (GC/ECD Instrument Performance Check Criteria) and Section XII (Target Analyte Identification Criteria) of the Pesticide/PCB Functional Guidelines for additional guidance (Worksheets Pest/PCB-II-A and Pest/PCB-XII). A discussion of the reasons for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* ii. Evaluate the raw data to verify that the reported resolution is correctly calculated and accurately transcribed for Individual Standard Mixtures A and B in the initial calibration on both columns. See Appendix F, Section II for the method of calculation.	ii. If the laboratory made calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the reasons for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
1. d. i. Review Form VI PEST-1 to verify that retention time windows were reported for all single component pesticides.	1. d. i. If the laboratory did not report retention time windows for all single component pesticides, then the validator should have the laboratory resubmit all corrected raw data and forms.
ii. Review 10% of the tabulated mean RTs and retention time windows reported on Form VI PEST-1 to verify that they were calculated correctly. See Appendix F, Table App.F.III-3 for retention time windows.	ii. If errors are detected in the calculations of the mean RTs or retention time windows, then the validator should perform a more comprehensive review that includes the review of raw data as described in d.iii.
* iii. Review the Individual Standard Mixtures A and B raw retention time data for calculation and transcription errors.	iii. If the laboratory made calculation and/or transcription errors, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
e. i. Review Form VI PEST-2 to verify that low, mid and high calibration factors were reported for each single component pesticide and surrogate on each column.	e. i. If the laboratory did not report low, mid and high calibration factors for each single component pesticide and surrogate, then the validator should contact the laboratory to obtain omitted data and corrected forms.
ii. Review 10% of the tabulated %RSD and Mean CF results reported on Form VI PEST-2 to verify that they were calculated correctly.	ii. If an error rate of greater than 10% is detected in the calculations of the Mean CFs and %RSDs, then the validator should perform a more comprehensive review that includes the review of raw data as described in C.1.g.

C. EVALUATION	D. ACTION
1. f. Review Form VI PEST-2 to verify that the %RSD for the calibration factors in each of the single component pesticides in the initial calibration analyses on both columns are in compliance with the linearity criteria as described in B.1.e.	1. f. If the %RSD linearity criteria (Pest/PCB-III-12, Table Pest/PCB-III-1) are not met on one or both columns for the single component pesticides being quantified, then the validator should: i. Estimate (J) positive detects for that affected analyte for all samples associated with the initial calibration. ii. Estimate (UJ) non-detects for that affected analyte for all samples associated with the initial calibration. iii. Use professional judgment if the %RSD is exceeded on one column and positive detects are quantified using CFs generated from a dissimilar column with a compliant initial calibration. The validator may choose to accept a sample result if the analyte was reported from the compliant GC column. However, qualification may be necessary if a tentative identification is made on the compliant GC column that requires confirmation on the noncompliant GC column. The validator should use professional judgement to estimate (J) and/or reject (R) sample data if %RSD criteria were grossly exceeded on one or both columns and the possibility of false negatives or false positives.
g. Review Form VI PEST-2 to verify that the %RSD for the calibration factors for each of the surrogates in the initial calibration analyses on both columns are in compliance with the linearity criteria as described in B.1.e.	g. If a surrogate analyte fails to meet %RSD criteria, then the % surrogate recoveries in the samples, QC samples and blanks associated with the initial calibration may be biased high or low. In this case, the validator should use professional judgment to assess the impact of surrogate analyte calibration data on the sample results.

C. EVALUATION	D. ACTION
*1. h. Review the raw calibration factor data and recalculate the calibration factors and %RSD for one or more of the single component pesticides; verify that the recalculated values agree within 10% of the reported values.	1. h. If errors of greater than 10% are detected in the calibration factor and %RSD calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. This review should recalculate at least 20% of the calibration factors and %RSDs and should use the DQOs to decide which analytes to recalculate. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* i. Confirm from the Individual Standard Mixtures A and B chromatograms that at least one of the three analyses for each of Individual Standard Mixtures A and B from the initial calibration sequence displays the single component pesticides at greater than 50 percent and less than 100 percent of full scale.	i. If none of the chromatograms for the initial calibration Individual Standard Mixtures A and B have peaks that are between 50 and 100 percent of full scale, then the validator should use professional judgment which may include that the laboratory should replot and resubmit corrected data and forms. The validator may use professional judgement to accept the data if it is determined that adequate standard chromatographic data are available to confirm positive analyte identifications and quantitation limits for non-detects.

C. EVALUATION	D. ACTION
2. Multicomponent Analytes	2. Multicomponent Analytes
a. i. Verify from Form VIII PEST that each multicomponent analyte standard was analyzed at the required frequency and in the proper sequence on each GC column and instrument used for analysis.	a. i. If the multicomponent analyte standards were not analyzed at the required frequency and in the proper sequence on each GC column and instrument, then the sample data may be adversely affected. The validator must use professional judgment to determine the severity of the non-compliance on the sample data and these data should be qualified accordingly.
* ii. Review the raw data to verify that analytical run times were accurately reported.	ii. If the laboratory has made transcription errors, then the validator should have the laboratory resubmit all corrected raw data and forms.
b. i. Verify from the Form VI PEST-3 that the multicomponent analytes and surrogates were analyzed at the required concentration.	b. i. If multicomponent analyte standards were not analyzed at the required concentration, then the sample data may be adversely affected. The validator must use professional judgment to determine the severity of the effect on the sample data and these data should be qualified accordingly.
* ii. Review the raw data to verify that the multicomponent analyte and surrogate concentrations were accurately reported.	ii. If the laboratory has made transcription errors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms.

C. EVALUATION	D. ACTION
2. c. i. Review Form VI PEST-3 to verify that retention time windows were reported for at least 3 peaks for each multicomponent analyte.	2. c. i. If the laboratory did not report retention time windows for at least three peaks, then the validator should have the laboratory resubmit all corrected raw data and forms.
ii. Review 10% of the tabulated retention time windows reported on Form VI PEST-3 to verify that they were calculated correctly.	ii. If errors were detected in the retention time window calculations, then the validator should perform a more comprehensive review that includes the review of raw data as described in C.2.c.iii.
* iii. Review the multicomponent analyte raw retention time data for calculation and transcription errors.	iii. If the laboratory made calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
d. Verify from Form VI PEST-3 that at least three peaks were used for the initial calibration and that calibration factor data are available for each peak.	d. If at least three peaks were not used for quantitation, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms.

C. EVALUATION	D. ACTION
**2.e. If applicable, review tabulated results of %RSD for multicomponent analyte standards.	2. e. If the %RSD linearity criteria are not met for the multicomponent analytes being quantified, then the validator should: i. Estimate (J) positive detects for that affected analyte for all samples associated with the initial calibration. ii. Estimate (UJ) non-detects for that affected analyte for all samples associated with the initial calibration. iii. Use professional judgment if the %RSD is exceeded on one column and positive detects are quantified using CFs generated from a dissimilar column with a compliant initial calibration. The validator may choose to accept a sample result if the analyte was reported from the compliant GC column. However, qualification may be necessary if a tentative identification is made on the compliant GC column that requires confirmation on the noncompliant GC column. The validator should use professional judgement to estimate (J) and/or reject (R) sample data if %RSD criteria were grossly exceeded on one or both columns and the possibility of false negatives or false positives.
f. If applicable, review tabulated results of %RSD for surrogate analytes.	f. If any surrogate analyte fails to meet %RSD criteria of 30%, then the % surrogate recoveries in the samples, QC samples and blanks associated with the initial calibration may be biased high or low resulting in unacceptable surrogate recoveries. In this case the validator should use professional judgment to assess the impact of surrogate analyte calibration data on the sample results.

C.	EVALUATION	D.	ACTION
*2. g.	Review and recalculate the calibration factors (and %RSD if applicable) for one or more multicomponent analytes; verify that the recalculated values agree within 10% of the reported values.	2. g.	If errors greater than 10% are detected in the calibration factor and/or %RSD (if applicable to method) calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* h.	Confirm that the standard chromatogram peaks chosen for multicomponent analyte identification are greater than 25% and less than 100% of full scale deflection.	h.	If none of the chromatograms for the initial calibration multicomponent analytes have peaks that are between 25 and 100% of full scale, then the validator should use professional judgment to decide if the laboratory should be required to replot and resubmit corrected data and forms. The validator may determine that adequate standard chromatographic data are available to confirm positive analyte identification and quantitation limits for non-detected results.

C. EVALUATION	D. ACTION
3. Single and Multicomponent Analytes	3. Single and Multicomponent Analytes
* a. Review Standard Preparation Logs (if provided in the data package) to ensure that primary and secondary initial calibration standard concentrations are accurate and traceable to NIST standards.	a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to facilitate the validation of sample data. If necessary, the validator should contact the laboratory to obtain standards information, including traceability information. If standards preparation data were submitted and found not to be NIST traceable, second source standards, PES, and other QC data should be evaluated. The validator must use professional judgement to accept (A), estimate (J), or reject (R) the sample data. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* b. Review and recalculate the calculated initial calibration standard concentration for one single component and one multicomponent analyte (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.a.ii, C.1.b.ii, C.1.b.iv, C.1.c.ii, C.1.d.iii, C.1.h, C.1.i, C.2.a.ii, C.2.b.ii, C.2.c.iii, C.2.g, C.2.h, C.3.a, C.3.b

** **Not required in CLP-SOW OLM04.3**

Table Pest/PCB-III-1:

QUALIFICATION OF PESTICIDE/MULTICOMPONENT* ANALYTES BASED ON THE INITIAL CALIBRATION

Sample Results	%RSD ≤ 20.0% (alpha-BHC & delta-BHC %RSD ≤ 25.0%)	%RSD > 20.0% (alpha-BHC & delta-BHC %RSD > 25.0%)	If applicable, multicomponent analyte %RSD ≤ 25.0%	If applicable, multicomponent analyte %RSD > 25.0%	One column meets criteria but the other exceeds
Detects	A	J	A	J	Professional judgement
Non-detects	A	UJ	A	UJ	Professional judgement

* **OLM04.3 does not require analysis of more than one initial calibration standard concentration for multicomponent analytes.**

E. EXAMPLES

Example #1: (High %RSD)

The Percent Relative Standard Deviation (%RSD) of the calibration factors for 4,4'-DDT is 80.0% on the reporting column. Due to erratic instrument performance, the validator estimates (J) all positive 4,4'-DDT detects and estimates (UJ) the 4,4'-DDT non-detects in the field samples associated with the initial calibration on the Data Summary Tables and discusses this in the Data Validation Report.

Example #2: (Non-compliant chromatographic scaling factors)

The validator cannot differentiate between Aroclor 1242 and Aroclor 1016 in the standards because the peaks are well below 25% of full scale. After examining the sample chromatograms, the presence of either Aroclors 1016 or 1242 is detected. The validator contacts the laboratory and requests that they replot and resubmit the standards data at full scale. The validator is able to differentiate the two Aroclors in the resubmitted data and completes the validation. Telephone logs of all communications between the validator and the laboratory are incorporated into the Data Validation Report.

Example #3: (Non-compliant standard concentrations)

The laboratory ran Individual Standard Mixtures A and B at concentrations higher than required by the analytical specifications. The reported quantitation limits are higher than the CRQLs. The Data Quality Objectives (DQOs) were designed to assess human health risks due to contamination at this site. The CRQLs were designated by the project specifications and the reported quantitation limits do not provide the required information to meet the DQOs. The validator apprises the EPA of the situation and payment is denied for the non-compliant analyses. All telephone logs between the validator and the laboratory and the validator and the EPA are incorporated into the Data Validation Report and the recommendation for non-payment.

IV. CALIBRATION VERIFICATION

A. OBJECTIVE

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Successful completion of the calibration verification procedures ensure satisfactory instrument performance. Calibration verification is performed to confirm the accuracy of the calibration at designated intervals within the analytical sequence. Calibration verification procedures include the analysis of instrument blanks to verify the presence or absence of instrument contamination, and single component pesticide and multicomponent analyte calibration standards to verify the accuracy of the initial calibration. Also, a Performance Evaluation Mixture (PEM) is analyzed to verify that chromatographic resolution and pesticide degradation acceptance criteria are achieved.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F Section IV should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. Calibration verification is performed once every 12 hours of sample analysis, on each GC column and instrument used for analysis. An instrument blank and the PEM must bracket one end of a 12-hour period during which sample data are collected, and a second instrument blank and the midpoint concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period. Samples may be injected for 12 hours from the injection of the instrument blank. The multicomponent analyte standard must be analyzed within 72 hours of that multicomponent analyte being detected in a sample chromatogram.

2. The chromatographic resolution between any two adjacent peaks in the calibration verification midpoint single component pesticide calibration standard mixtures (Individual Standard Mixtures A & B) and the PEMs must be greater than or equal to 90.0 percent.

3. The absolute retention time for each single component pesticide, surrogate and multicomponent analyte in the calibration verification standards and the PEM must be within the retention time windows determined from the initial calibration.

4. The Percent Difference (%D) between the calculated amount and the nominal amount for each of the single component pesticides, multicomponent analytes and surrogates in the calibration verification standards and the PEM must not exceed ± 25.0 percent.

5. The degree of pesticide degradation must be determined for each column used to analyze field samples. See Section II, GC/ECD Instrument Performance Check for additional information.
   a. The percent breakdown for 4,4'-DDT in each PEM must be less than or equal to 20.0 percent for both GC columns.
   b. The percent breakdown for endrin in each PEM must be less than or equal to 20.0 percent for both GC columns.

c. The combined percent breakdown for 4,4'-DDT and endrin in each PEM must be less than or equal to 30.0 percent for both GC columns.

6. The chromatograms that result from the analyses of the calibration verification PEMs, single component calibration standard mixtures, and multicomponent analyte standards must display the analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

7. The instrument blanks must meet all acceptance criteria as stated in Section V. Blanks.

8. The criteria mentioned above apply to the results from both chromatographic columns. In the event that one GC column meets criteria and the other does not, the validator may use professional judgement to evaluate the effect of the noncompliance on the associated samples. The validator may choose to accept a sample result reported from the compliant GC column. The validator must consider whether the noncompliant GC column criteria were grossly exceeded, and the possibility of false negatives or false positives.

Note: CLP SOW-OLM04.3 does not require the analysis of a multicomponent analyte calibration verification. If a multicomponent analyte calibration verification standard is required by a non-CLP method, then the above frequency criteria, retention time criteria, and % Difference criteria used to evaluate single component pesticides should be used to evaluate calibration verification for multicomponent analytes.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential and actual impacts on the sample data resulting from calibration verification anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Review the Form VIII PEST to verify that the instrument blanks, PEMs, and Individual Standard Mixtures were analyzed at the required frequency on each GC column and instrument used for analysis and that no more than 12 hours elapsed between calibration verification brackets in an ongoing analytical sequence. Confirm that a multicomponent analyte standard was analyzed within 72 hours of that multicomponent analyte being detected in a sample chromatogram.	1. a. If the calibration verification sequence did not meet method requirements then professional judgment must be used to evaluate the effect of the non-compliance on the sample data. If the non-compliance affects the data, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.

C. EVALUATION	D. ACTION
*1. b. Review the raw data to verify that analytical run times are reported accurately by comparing the date and time of injection reported on the chromatograms with the date and time analyzed reported on Form VIII PEST.	1. b. If the laboratory has made transcription errors, then the validator should have the laboratory resubmit all corrected raw data and forms.
2. **RESOLUTION CHECK** a. Review Form VI PEST-5, Form VI PEST-6 and Form VI PEST-7 to verify that the resolution between any two adjacent peaks is greater than or equal to 90.0% in the PEMs and in the midpoint concentrations of Individual Standard Mixtures A and B. * b. Examine the chromatograms to verify that the reported peak resolution is correctly calculated and transcribed for the PEMs and the Individual Standard Mixtures A and B in the calibration verifications on both columns. See Appendix F, Section II for the method of calculations.	2. **RESOLUTION CHECK - Worksheet Pest/PCB-II-A** a. If the resolution QC criteria for the method are not met, then the quantitative results may not be accurate due to inadequate resolution. Estimate (J) positive detects for analytes that were not adequately resolved. Qualitative identifications may be questionable if coelution exists. Non-detects that elute in the region of coelution may not be valid depending upon the extent of the coelution problem. Before rejecting (R) any of the non-detects as unusable, proceed to the next section, C.2.b, and examine the chromatograms (Tier III level) to verify that the analytical results have been calculated and reported accurately. If the results have been calculated and reported accurately, then professional judgement should be used to reject (R) non-detects as unusable. b. If the laboratory made calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to determine the more accurate value. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. **RETENTION TIME CHECK**	3. **RETENTION TIME CHECK - Worksheet Pest/PCB-II-B**
a. Review Form VII PEST-1 and Form VII PEST-2 to verify that the absolute retention times of the analytes in the PEMs and the Individual Standard Mixtures A and B are within the appropriate retention time window limits.	a. Retention time windows are used in qualitative identifications. If the retention times of the pesticides in the calibration verification standards or PEMs do not fall within the established retention time windows, then the associated sample results should be carefully evaluated for the possibility of false positives and false negatives. All samples injected after the last in-control standard are potentially affected and a Tier III level of validation should be performed to assess the impact on sample results.
* b. Review the raw data for samples analyzed after the last compliant calibration verification (either Individual Standard Mixtures A and B or PEM). Review the sample chromatograms to verify the presence or absence of peaks close to the expected retention time windows for any target pesticide.	b. If no peaks are present within or close to expected retention time windows for any target pesticide, then non-detected values can be considered valid. If sample chromatograms contain peaks close to or within the expected retention time window of target pesticides, then the validator should follow the guidelines provided in Section II.D.2.b Options 1 and 2 to determine the action to be taken.
* c. Review the retention time data for each of the single component pesticides and surrogates in the midpoint concentration of Individual Standard Mixtures A and B and PEMs to verify that the absolute retention times have been correctly calculated and reported and are within the appropriate retention time windows. Review the retention time data on the chromatograms for each of the multicomponent analytes analyzed in the calibration verification.	c. If the laboratory made calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
4. **ACCURACY (%D) CHECK** a. Review Form VII PEST-1 and Form VII PEST-2 to verify that the %D between the calculated amount and the nominal amount for each of the single component pesticides and surrogates does not exceed ± 25.0% in either the Individual Standard Mixtures or the PEMs. If applicable, check any tabulated results for %Ds between the calculated amount and the nominal amount for each of the multicomponent analytes and surrogates to verify that the %Ds do not exceed ± 25.0%. See Appendix F, Section II for the method of calculating %D.	4. **ACCURACY (%D) CHECK - Worksheet Pest/PCB-IV** a. If the % D exceeds ± 25.0 % for the single component or multicomponent analytes being quantified, then the validator should: i. Estimate (J) positive detects for the affected analyte for all samples associated with the unacceptable PEM and/or Individual Standard Mixture. Note that the associated samples are the ones analyzed before and after the non-compliant PEM/Individual Standard and between compliant standards. ii. Estimate (UJ) non-detects for that affected analyte for all samples associated with the unacceptable PEM and/or Individual Standard Mixture. iii. If any surrogate analyte in the PEM and/or Individual Standard Mixture A or B fails to meet %D criteria, then the % surrogate recoveries in the samples, QC samples and blanks associated with the calibration verification may be biased high or low resulting in unacceptable surrogate recoveries. In this case, the validator should use professional judgment to assess the impact of surrogate analyte calibration data on the sample results. iv. Use professional judgment if the %D is exceeded on one column. The validator may choose to accept a sample result if the analyte was reported from the compliant GC column. However, qualification may be necessary if a tentative identification is made on the compliant GC column that requires confirmation on the noncompliant GC column. The validator should use professional judgement to estimate (J) and/or reject (R) sample data if %D criteria were grossly exceeded on one or both columns considering the possibility of false negatives or false positives.

C. EVALUATION	D. ACTION
4. a. Continued from above.	4. a. v. Before estimating (J, UJ) large amounts of data, professional judgement may be used to determine that a Tier III level of data validation is warranted. In this case, proceed to section C.4.b and verify that the %D results have been calculated and reported correctly.
* b. Review the raw data from the midpoint concentration of Individual Standard Mixtures A and B and the PEMs to verify that the % D has been correctly calculated and reported. If applicable, review the raw data for each of the multicomponent analytes analyzed in the calibration verification to verify that the %D has been correctly calculated and reported.	b. If the laboratory made calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
c. Review the Form VII PEST-1 and VII PEST-2 to verify that the calibration verification standards were analyzed at the correct concentrations for each analyte.	c. If the calibration verification standards were not analyzed at the correct concentrations then professional judgment must be used to evaluate the effect of the non-compliance on the sample data. If the non-compliance affects the data, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.
* d. Review the raw data to verify that the concentrations reported on Form VII-PEST-1 and VII-PEST-2 were reported accurately.	d. If the laboratory has made transcription errors, then the validator should have the laboratory resubmit all corrected raw data and forms.

C. EVALUATION	D. ACTION
5. **PESTICIDE DEGRADATION CHECK** a. Verify from Form VII PEST-1 that the individual breakdown for 4,4'-DDT is less than or equal to 20.0% in the calibration verification PEM analyses.	5. **PESTICIDE DEGRADATION CHECK - Worksheet Pest/PCB-II-D** a. If 4,4'-DDT breakdown is greater than 20.0%, then the validator should: **4,4'-DDT Detected** i. Estimate (J) DDT, DDD, DDE positive detects and accept DDD and DDE non-detects for samples associated with the unacceptable PEM. **4,4'-DDT Not Detected** ii. Reject (R) DDT non-detects as unusable, accept DDD, DDE non-detects, and estimate (J) DDD, DDE positive detects and note the potential high bias for these two analytes. If the results from a second, compliant GC column are available, evaluate these results to determine to what extent DDD and DDE positive detects are due to DDT breakdown and use professional judgement to accept, estimate (J), or reject (R) results.

C. EVALUATION	D. ACTION
5. b. Verify from Form VII PEST-1 that the individual breakdown for endrin is less than or equal to 20.0% in the calibration verification PEM analyses.	5. b. If endrin breakdown is greater than 20.0%, then the validator should: **Endrin Detected** i. Estimate (J) endrin, endrin ketone, endrin aldehyde positive detects, and accept endrin ketone, endrin aldehyde non-detects for samples associated with the unacceptable PEM. **Endrin Not Detected** ii. Reject (R) endrin non-detects, accept endrin ketone, endrin aldehyde non-detects, and estimate (J) endrin ketone endrin aldehyde positive detects and note the potential high bias for these two analytes. If the results from a second, compliant GC column are available, evaluate these results to determine to what extent endrin ketone and endrin aldehyde positive detects are the due to endrin breakdown and use professional judgement to accept, estimate (J), or reject (R) results.

C. EVALUATION	D. ACTION
5. c. Verify from Form VII PEST-1 that the combined breakdown for 4,4'-DDT and endrin is less than or equal to 30.0% in the calibration verification PEM analyses.	5. c. If the combined 4,4'-DDT and endrin breakdown is greater than 30.0%, then the validator should: **4,4'-DDT Detected** i. Estimate (J) DDT, DDD, DDE positive detects and accept DDD and DDE non-detects for samples associated with the unacceptable PEM. **4,4'-DDT Not Detected** ii. Reject (R) DDT non-detects as unusable, accept DDD, DDE non-detects, and estimate (J) DDD, DDE positive detects and note the potential high bias for these two analytes. **Endrin Detected** iii. Estimate (J) endrin, endrin ketone, endrin aldehyde positive detects, and accept endrin ketone, endrin aldehyde non-detects for samples associated with the unacceptable PEM. **Endrin Not Detected** iv. Reject (R) endrin non-detects, accept endrin ketone, endrin aldehyde non-detects, and estimate (J) endrin ketone endrin aldehyde positive detects and note the potential high bias for these two analytes. If the results from a second, compliant GC column are available, evaluate these results to determine to what extent DDD, DDE, endrin ketone, and endrin aldehyde positive detects are the due to DDT and endrin breakdown and use professional judgement to accept, estimate (J), or reject (R) results.

C. EVALUATION	D. ACTION
*5. d. Review and recalculate 10% of the DDT and endrin breakdown data in the PEMs. Verify that the recalculated values agree within 10% of the laboratory values.	5. d. If errors greater than 10% are detected, then the validator should perform a more comprehensive review to determine the extent of the problem. If the problem is extensive, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*6. Review the raw data to verify that the chromatographic peaks of the single component pesticides and multicomponent analytes calibration standard mixtures are greater than 10% of full scale but less than 100% of full scale.	6. If none of the chromatograms for the calibration verification standards yield peaks that are between 10 and 100% of full scale, then the validator should use professional judgment to decide whether or not the laboratory should be required to replot and resubmit corrected data and forms. Alternatively, the validator may determine that adequate standard chromatographic data are available to confirm positive analyte identifications and non-detects.

C. EVALUATION	D. ACTION
*7. a. Review Standard Preparation Logs (if provided in the data package) to ensure that primary and secondary calibration verification standard concentrations are accurate and traceable to NIST standards.	7. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine whether or not standards preparation data are necessary to facilitate the validation of sample data. If necessary, the validator should contact the laboratory to obtain standards information including traceability information.
* b. Review and recalculate the calibration verification standard concentration for one single component target analyte and one multicomponent target analyte (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.2.b, C.3.b, C.3.c, C.4.b, C.4.d, C.5.d, C.6, C.7.a, C.7.b

Table Pest/PCB-IV-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON CALIBRATION VERIFICATION

Sample Results	%D ≤ ±25.0%	%D > ±25.0%	One column meets criteria but the other exceeds
Detects	A	J	Professional judgement
Non-detects	A	UJ	Professional judgement

Table Pest/PCB-IV-2:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON 4,4'-DDT/ENDRIN BREAKDOWN - PESTICIDE DEGRADATION CHECK

Sample Results	4,4'-DDT Breakdown ≤ 20.0%	4,4'-DDT Breakdown > 20.0% and 4,4'-DDT detected	4,4'-DDT Breakdown > 20.0% and 4,4'-DDT not detected	Endrin Breakdown ≤ 20.0%	Endrin Breakdown > 20.0% and Endrin detected	Endrin Breakdown > 20.0% and Endrin not detected	Combined Breakdown ≤ 30.0%	Combined Breakdown > 30.0% and 4,4'-DDT and/or Endrin detected	Combined Breakdown > 30.0% and 4,4'-DDT and/or Endrin not detected
4,4'-DDT	A	J	R (NDs)	N/A	N/A	N/A	A	J	R (NDs)
DDD	A	J (detects) A (NDs)	J (detects) A (NDs)	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)
DDE	A	J (detects) A (NDs)	J (detects) A (NDs)	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)
Endrin	N/A	N/A	N/A	A	J	R (NDs)	A	J	R (NDs)
Endrin Aldehyde	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)	A	J (detects) A (NDs)	J (detects) A (NDs)
Endrin Ketone	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)	A	J (detects) A (NDs)	J (detects) A (NDs)

N/A = Not Applicable
J = Estimate result
R (NDs) = Reject non-detects
A (NDs) = Accept non-detects

Note: The validator must always discuss negative and positive bias in sample data in the Data Validation Memorandum.

E. EXAMPLES

Example #1: (Non-compliant analytical sequence)

The validator checks Form VIII PEST and determines that 14 hours elapsed between calibration verification standard brackets in an ongoing analytical sequence. The validator examines the sample data and determines that all calibration criteria were met (retention times were within the required retention time windows and %Ds were within criteria. Using professional judgment the validator reports the sample results unqualified on the Data Summary Table and notes the non-compliance in the Data Validation Memorandum.

Example #2: (Non-compliant chromatographic resolution)

The validator checks Form VI PEST-6 to verify that the peak resolution criteria were met in Individual Mixture A. The resolution between endosulfan I and heptachlor is found to be 88.0% on column DB1701. The validator also reviews the previous and subsequent standard runs containing these analytes to assess the extent of non-compliance for these analytes. Since the resolution is only slightly out of criteria in one Individual Mixture A and the resolution between these analytes on column DB608 was met, the validator reports the sample results unqualified on the Data Summary Table and notes the non-compliance in the Data Validation Memorandum.

Example #3: (Non-compliant retention time)

The validator checks Form VII PEST-2 to verify that the retention time criteria were met. The retention time window for methoxychlor is from 11.64 to 11.78. The calibration verification retention time for methoxychlor in Individual Mixture A is 11.86. The validator examines the sample chromatograms and determines that no peaks are present either within or near the retention time window. The validator uses professional judgment to accept the methoxychlor non-detects and reports the methoxychlor quantitation limits unqualified on the Data Summary Table and notes the non-compliance in the Data Validation Memorandum.

Example #4: (Non-compliant %D)

The validator reviews Form VII PEST-2 to check percent differences between the calculated amount and the nominal amount. Gamma-Chlordane has a %D of 65.0% in Individual Mixture B. The validator checks the raw data and verifies that the %D has been properly calculated. The validator reviews the previous and subsequent standard runs containing gamma-chlordane to assess the extent of the non-compliance for this analyte. Due to variable instrument performance, the validator estimates (J) the positive gamma-chlordane detects and estimates (UJ) the gamma-chlordane non-detects for all samples associated with the non-compliant calibration verifications on the Data Summary Table. The validator discusses the sample qualification in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (Pesticide Degradation)

The validator reviews the DDT/Endrin breakdown data and notes that the endrin breakdown in the PEM, which was analyzed as the calibration verification for this sequence, is 39.0%. The validator then reviews the subsequent PEMs that were analyzed 24 hours before and after and notes that the endrin breakdown is less than 20.0%. For samples that have positive endrin detects; the validator estimates (J) the endrin detects, and estimates (J) positive endrin ketone and endrin aldehyde detects and accepts endrin ketone and endrin aldehyde non-detects for all samples analyzed before and after the non-compliant PEM. For samples that have endrin non-detects; the validator rejects (R) the endrin non-detects and estimates (J) endrin ketone and/or endrin aldehyde detects and accepts endrin ketone and endrin aldehyde non-detects. It is noted in the Data Validation Memorandum that when the breakdown for endrin is high, the values for endrin are potentially biased low and the values for endrin ketone and endrin aldehyde are potentially biased high.

V. BLANKS

A. OBJECTIVE

The purpose of blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory and/or field activities and to subsequently assess their contribution to measurement error. The criteria for evaluation of laboratory blanks (method blanks, instrument blanks, and sulfur cleanup blanks) may be applied to any blank associated with the samples. If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent measurement error associated with the entire data set, or if the problem is an isolated occurrence limited to specific samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines For Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. The frequency and types of blanks collected and analyzed must support the site-specific Data Quality Objectives (DQOs) as documented in the EPA approved QAPP or SAP. Different types of blanks may be used to identify the source of potential contamination resulting in analytical and/or sampling measurement error. The following table lists types of blanks, the environment of these blanks, and the possible sources of contamination associated with those blanks:

BLANK	LABORATORY/FIELD	IDENTIFIES CONTAMINATION FROM
Method Blank	Laboratory	Laboratory and Reagents
Instrument Blank	Laboratory	Instrumentation
Bottle Blank	Field	Sample Container
Equipment Blank (Rinsate)	Field	Sampling Equipment

Note: Aqueous equipment (rinsate) blank results and bottle blank results will be used to determine blank action levels for aqueous samples based on a volume of 1 liter of blank sample. Ideally soil/sediment blanks should be used to determine soil/sediment blank actions for soil/sediment samples based on a known weight of blank sample. However, frequently aqueous equipment blanks and bottle blanks are collected to evaluate soil/sediment contamination associated with sampling. **Aqueous equipment (rinsate) blank results and bottle blank results will not be used to determine blank action levels for non-aqueous samples.** Analytes that are present in both the non-aqueous sample and the associated aqueous equipment blank or bottle blank will be flagged EB (Equipment Blank) or BB (Bottle Blank), respectively. The degree of "sampling error" that this flagged sample result represents will be left to the determination of the end user.

If a contaminant is found in a blank but not in the aqueous sample, no action is taken. If a

contaminant is found in both a blank and an aqueous sample, then the validator should note this problem in the Data Validation Memorandum and qualify the data according to the following guidance:

> If the blank action level for an analyte is determined using the value from a bottle blank or equipment blank, then the positive values in the bottle or equipment blank should be reported unqualified on the Data Summary Tables. However, if the blank action is determined using the value from the laboratory blank (e.g., method, cleanup, or instrument), then the positive values in the bottle or equipment blank should be qualified. (See example # 6)

If analytes are present in both the non-aqueous sample and the associated aqueous equipment blank or bottle blank, then the results for these analytes in the non-aqueous sample will be flagged EB (Equipment Blank) or BB (Bottle Blank), respectively. The degree of sampling error that this flagged sample result represents will be left to the determination of the end user. However, the data validator should note this problem in the Data Validation Memorandum.

Use of professional judgment is suggested when equipment and bottle blanks are associated with highly contaminated samples and are not likely to have contributed to sample contamination.

For aqueous and non-aqueous samples, contamination found in the equipment or bottle blank must be reported to the sampler and the EPA Regional Project Manager.

2. Method Blanks:

   a. An acceptable pesticide/PCB method blank must be extracted with each sample delivery group or each 20 samples of similar matrix in each sample delivery group or whenever a sample extract procedure is performed. The method blank must undergo all cleanup procedures including Gel Permeation Chromatography (GPC) and Florisil. The method blank must be analyzed on each gas chromatography (GC) system used to analyze samples.

   b. A sulfur cleanup blank must be analyzed whenever part of a set of samples extracted together requires sulfur cleanup. If the <u>entire</u> set of samples associated with a method blank requires sulfur cleanup, then the method blank must be carried through the sulfur cleanup procedures. In that case, the method blank can also be considered a sulfur cleanup blank and no separate sulfur cleanup blank is required.

3. GC Instrument Blanks:

   a. An acceptable GC instrument blank must be analyzed at least once every 12 hours and immediately prior to the analysis of either the continuing calibration standards (INDA and INDB) or the Performance Evaluation Mixture (PEM).

   b. A GC instrument blank should be analyzed after any sample with peaks that exceed the calibration range to demonstrate that the system is not contaminated.

4. GPC Instrument Blanks:

   a. A GPC blank must be analyzed after each GPC calibration. The GPC blank consists of 5 mLs of methylene chloride and is not spiked with surrogates.

5. All blanks (except GPC blanks) must be spiked with surrogate analytes according to the method.

   a. The retention times of the surrogates in each blank must be within the retention time windows calculated from the initial calibration.

   b. Blank surrogate analytes must meet method surrogate analyte QC acceptance criteria for recovery. The default criteria are listed in Appendix F.

6. No contaminants should be present in the blanks. The concentrations of any contaminants found in the blanks should be less than the QC acceptance criteria specified in the QAPP/SAP.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from blank anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. **Qualification should be based upon a comparison of the sample concentration(s) with the highest blank concentration associated with the sample delivery group.** However, in cases of specific instrument and/or method blank contamination, the validator should use professional judgment to qualify only those samples associated with that isolated blank contamination. Likewise, the validator may need to apply blank qualifications to a sample delivery group based on associated equipment, trip, or bottle blank data that exists in another sample group data package. **Sample results must not be corrected by subtracting any blank values.**

C. EVALUATION	D. ACTION
1. a. Verify that the correct number and type of blanks have been collected and analyzed in accordance with the EPA approved QAPP or SAP. b. Ascertain if aqueous equipment (rinsate) blanks or aqueous bottle blanks have been collected with non-aqueous samples to identify sources of field contamination.	1. a. If the correct number and type of blanks have not been collected and analyzed, then the validator should note this deviation from the EPA approved QAPP or SAP in the Data Validation Memorandum. The validator should use professional judgment to qualify sample data when blank data are absent. When required equipment (rinsate) or bottle blanks are not identified on the chain of custody, then the validator must contact the sampler or site project manager to obtain this information and note this contact on the Blank Analysis validation worksheet. b. If positive results are detected in the aqueous equipment (rinsate) blanks and/or bottle blanks <u>and</u> the associated <u>non-aqueous</u> samples, then the validator should flag (EB or BB) those detected analytes in the associated non-aqueous samples to indicate to the end user that an indeterminate amount of sampling error has potentially affected the sample results.
2. a. Verify that a method blank analysis has been reported per matrix, per concentration level, for each 12-hour time period on each GC system used to analyze each extraction batch of pesticide/PCB samples. The validator should review Form IV PEST (the pesticide Method Blank Summary) to identify the samples associated with each method blank. b. Verify that sulfur cleanup blanks were analyzed at the required frequency. Verify that a Form IV PEST was completed listing all the samples associated with the method blank, and that a <u>second</u> Form IV PEST was completed listing only those samples associated with the sulfur cleanup blank.	2. a. If method blanks were not analyzed at the required frequency and for each matrix and concentration level, extraction technique and batch and on each GC system used to analyze sample extracts, then the validator should use professional judgment to determine whether or not the associated sample data should be qualified. b. If a required sulfur blank was not analyzed, then the validator should use professional judgment to determine the effect on the data and qualify the sample results accordingly.

C. EVALUATION	D. ACTION
*2. c. Verify from the raw data that the extraction and/or analysis dates and times, sample IDs, file IDs, instrument IDs, etc. are accurately reported on the tabulated result forms.	2. c. If review of the raw data reveals discrepancies and/or transcription errors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
3. a. Verify from Form VIII PEST that an instrument blank was analyzed every 12 hours as part of the required analytical sequence. * b. Verify from the raw data that a GC instrument blank was analyzed after each sample with peaks that exceeded the calibration range.	3. a. If instrument blanks were not analyzed at the required frequency on each GC system used to analyze sample extracts, then the validator should use professional judgment to determine whether the associated sample data should be qualified. b. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), then sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument contamination has affected any positive analyte identification and/or quantitation, and to determine whether or not the affected sample data should be qualified or rejected. If contamination is suggested, then this should be noted in the Data Validation Memorandum.
*4. Verify from the raw GPC data that a GPC instrument blank was analyzed after the GPC calibration and prior to sample analysis.	4. If a GPC instrument blank was not analyzed at the method-required frequency, then the validator should evaluate the method blank data and use professional judgment to qualify sample data associated with that GPC cleanup procedure.

C. EVALUATION	D. ACTION
5. a. Verify from Form VIII PEST that the surrogate retention times for all blanks analyzed are within the established retention time windows.	5. a. If blank surrogate retention times have shifted, then the validator should use professional judgment in applying blank actions. The possibility of false positives or false negatives being incorrectly reported for the blank should be evaluated.
b. Verify from Form VI PEST-1 that the surrogate retention time windows have been correctly calculated.	b. If the retention time windows have not been calculated correctly, then the validator should have the laboratory recalculate and resubmit Form VI PEST-1.
* c. Review the raw data for each blank to confirm that retention time data have been correctly transcribed to the tabulated forms. Review the blank chromatograms and quantitation reports to ensure that contamination has been accurately reported. For additional guidance refer to Section XII, Target Analyte Identification, in Part III.	c. If the laboratory has reported a false positive or a false negative and/or has incorrectly transcribed data, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
d. Verify from Form II PEST-1 and PEST-2 that blank surrogate recoveries meet method QC criteria.	d. If blank surrogate recoveries do not meet method criteria, then the validator should refer to Section VI, C.2.d for guidance.
* e. Check 10% of the raw blank data to confirm that surrogate recovery data has been accurately calculated and transcribed to the tabulated result forms.	e. If the laboratory has incorrectly calculated and/or transcribed blank surrogate recovery data, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. Review the tabulated reported results of all the blanks associated with the SDG.	6. If a contaminant is found in a blank but not in the aqueous sample, no action is taken. If a contaminant is found in both a blank and an aqueous sample, then the validator should note this problem in the Data Validation Memorandum and qualify the data according to the following guidance: **Note: If the blank action level for an analyte is determined using the value from a bottle blank or equipment blank, then the positive values in the bottle or equipment blank should be reported unqualified on the Data Summary Tables. However, if the blank action is determined using the value from the laboratory blank (e.g., method, cleanup, or instrument), then the positive values in the bottle or equipment blank should be qualified. (See example # 6)** If analytes are present in both the non-aqueous sample and the associated aqueous equipment blank or bottle blank, then the results for these analytes in the non-aqueous sample will be flagged EB (Equipment Blank) or BB (Bottle Blank), respectively. The degree of sampling error that this flagged sample result represents will be left to the determination of the end user. However, the data validator should note this problem in the Data Validation Memorandum. For aqueous and non-aqueous samples, contamination found in the equipment or bottle blank must be reported to the sampler and the EPA Regional Project Manager. Use of professional judgment is suggested when equipment and bottle blanks are associated with highly contaminated samples and are not likely to have contributed to sample contamination. If an analyte must be estimated (J) due to other validation criteria and an EB or BB is also applied, then use JEB or JBB in the Data Summary Table.

C. EVALUATION	D. ACTION
6. a. Determine whether or not any target analytes are present at or above the quantitation limit/CRQL in any of the blanks.	6. a. **Target Analyte Contaminants at or Above the Quantitation Limit/CRQL:** i. If the sample result for an analyte is greater than 5 times the highest concentration in any blank, then the analyte's concentration should be reported as unqualified. (See example #3 - 5x rule). ii. If the sample result for an analyte is less than or equal to 5 times the highest concentration of the analyte in any blank but greater than the quantitation limit, then the quantitation limit for that analyte should be elevated to the concentration found in the sample and reported as not detected (U). The validator should use professional judgment to determine if further elevation of the quantitation limit is required. (See example #1 - 5x rule). Note: The validator should note that blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" criteria, such that a comparison of the total amount of contamination is actually made. (See example #5). Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample data is deemed necessary. If the validator determines that the contamination originates from a source other than the sample, the sample data should be qualified. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. In this case, the "5x" rule may not apply; the target analyte should be reported as not detected (U), and an explanation of the data qualification rationale should be provided in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. b. Determine if low level contamination below the quantitation limit exists in any of the blanks.	6. b. **Target Analytes Below the Quantitation Limit/CRQL:** i. If a positive sample result is reported at less than the quantitation limit and is also less than the blank action level, then the sample quantitation limit should be reported as non-detected (U) on the Data Summary Tables. (See example #2 - 5x rule). ii. If one or several target analytes are found at low levels, below the quantitation limit, in the laboratory blank(s), it may indicate a systemic problem in the laboratory and should be noted in the Data Validation Memorandum. iii. If contamination exists solely in the bottle or equipment (rinsate) blanks, then the validator should notify the sampler. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.
c. Determine if gross contamination greater than 10x CRQL for any analyte exists in any of the blanks.	c. **Gross Contamination** i. If gross contamination, greater than 10x CRQL for any analyte, exists in any blank, then the validator should reject (R) all positive hits for the affected analytes and accept the non-detects in samples associated with that blank due to interference. This serious problem should be discussed in the Data Validation Memorandum. ii. If gross contamination exists solely in the bottle or equipment (rinsate) blanks, then the validator should notify the sampler and the EPA Regional Project Manager. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.

C. EVALUATION	D. ACTION
*6. d. Determine if instrument contamination is isolated to specific sample sequences or isolated to one column.	6. d. If contamination is limited to a few samples due to instrument contamination or limited to one column, then the validator may use professional judgment to accept or qualify sample data in samples associated with the instrument blank contamination.
e. Review the raw data (chromatograms, mass spectra confirmatory data and quantitation reports) to confirm the presence of target analytes in the blanks and to evaluate the presence of additional contaminants.	e. If review of raw data suggests that additional contaminants are present or, conversely, the review indicates false positives have been reported, then the validator should contact the laboratory to obtain additional information and/or have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
7. Evaluate the overall contamination in each type of blank to ascertain the probable source(s) of contamination. For example, a contaminated equipment blank might indicate decontamination problems, if the method, instrument, and bottle blanks were all clean.	7. If a review of the various types of blanks identifies a potential source of blank contamination, then the validator should discuss this problem in the Data Validation Memorandum. The validator should identify whether the measurement error is a result of either sampling or analytical error or both (see Data Validation Manual p.1).

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.2.c, C.3.b, C.4, C.5.b, C.5.d, C.6.e

E. EXAMPLES

Example #1: (Bottle blank target analyte contaminant ≥ CRQL, < 5x blank action level)

4,4'-DDT is detected in a water sample at greater than the CRQL, but less than 5x the bottle blank concentration.

5x Rule

	ug/L
Bottle Blank Result	1.0
CRQL	0.5
4,4'-DDT Sample Result	4.0
Action Level	5.0
"Qualified" Sample Result	4.0U

In this case, all sample results where 4,4'-DDT is less than 5.0 ug/L (5 x 1.0) are reported as non-detected at an elevated quantitation limit on the Data Summary Table. The validator notes in the Data Validation Memorandum that the bottle blank was contaminated with 4,4'-DDT and documents the lot number of the sample bottle, and alerts the site project manager regarding a contaminated lot of bottles.

Example #2: (Instrument blank target analyte contaminant < CRQL, < 5x blank action level)

Endrin is detected in a water sample at less than the CRQL, and also less than 5x the instrument blank concentration. The instrument blank contained the highest concentration of endrin of all blanks analyzed. In addition, all field samples analyzed were associated with the same contaminated instrument blank.

5x Rule

	ug/L
Instrument Blank Result	1.0
CRQL	0.5
Endrin Sample Result	0.4
Action Level	5.0
"Qualified" Sample Result	0.5U

In this case, the endrin sample result is less than 5.0 ug/L (1 x 5) and is reported non-detected at the CRQL on the Data Summary Table. The validator also notes the sample qualifications in the Data Validation Memorandum.

E. EXAMPLES

Example #3: (Blank target analyte contaminant > 5x blank action level)

Gamma-BHC is detected in a water sample at greater than 5x the instrument blank concentration.

5x Rule

	ug/L
Blank Result	1.0
CRQL	0.5
gamma-BHC Sample Result	10.0
Action Level	5.0
"Qualified" Sample Result	10

In this case the gamma-BHC sample result exceeded the blank action level of 5 ug/L (5 x 1.0) and the gamma-BHC sample result is unqualified on the Data Summary Table.

Example #4: (Blank target analyte contamination in aqueous equipment blank collected with soil samples)

An aqueous equipment blank (rinsate) was included in a sample delivery group of soil samples. The validator examines the data and finds that the equipment contains 0.08 ug/L of dieldrin. The validator then reviews all other blank data and finds no further dieldrin contamination. One soil sample contains 7.0 ug/kg of dieldrin. The validator reports the soil sample result on the Data Summary Table as 7.0 (EB) to indicate to the end user that sampling error has potentially affected the sample results and notes this information in the Data Validation Memorandum.

Example #5: (Application of sample weights and volumes with the 5x Rule)

Soil sample TAA35 was analyzed as a routine pesticide/PCB soil sample under CLP SOW OLM04.3 and contained 70% solids. The method blank was found to be contaminated with aldrin (2.2 ug/kg) and dieldrin (1.5 ug/kg). These blank results were reported by the laboratory on a dry weight basis and were the maximum levels of contamination found for these analytes in this sample delivery group. The validator determines the blank action level by applying the 5x rule. For the method blank the action level for aldrin was calculated to be 11.0 ug/kg (2.2 x 5), and the action level for dieldrin was calculated to be 7.5 ug/kg (1.5 x 5). The validator reviewed the sample results and found dieldrin (6.0 ug/kg) and aldrin (0.5 ug/kg) in TAA35.

The validator calculates the Quantitation Limits for dieldrin and aldrin:

$$\text{dieldrin QL} = \frac{\text{CRQL}}{\text{\% solids}} = \frac{3.3\ \text{ug/kg}}{0.7} = 4.7\ \text{ug/kg}$$

$$\text{aldrin QL} = \frac{\text{CRQL}}{\text{\% solids}} = \frac{1.7\text{ug/kg}}{0.7} = 2.4\ \text{ug/kg}$$

E. EXAMPLES

Example #5: (Continued)

The validator applies the following action to the dieldrin and aldrin results of sample TAA35:

Dieldrin		Aldrin	
5x Rule		5x Rule	
	ug/kg		ug/kg
Blank Result	1.5	Blank Result	2.2
CRQL	4.7	CRQL	2.4
Sample Result	6.0	Sample Result	0.5
Action Level	7.5	Action Level	11.0
"Qualified" Sample Result	6.0 U	"Qualified" Sample Result	2.4 U

- The dieldrin quantitation limit is elevated to the sample concentration result on the Data Summary Table as 6.0U, since the result is between the quantitation limit and the blank action level.

- The aldrin sample result on the Data Summary Table is replaced with the sample quantitation limit and is reported on the Data Summary Table as 2.4U, since the positive sample detect of 0.5, is below the quantitation limit and the blank action level.

The validator notes all actions taken in the Data Validation Memorandum.

Example #6: (Application of laboratory blank action levels to equipment blanks)

The method blank for an aqueous batch of samples was contaminated with 0.5 ug/L of endrin. The equipment blank for this batch of samples was contaminated with 0.35 ug/L of endrin and 0.6 ug/L of dieldrin. Since endrin was detected in both the method blank and the equipment blank, the highest detected concentration is used to determine the blank action level. The method blank is therefore used to determine the blank action level of endrin.

Endrin		Dieldrin	
	ug/L		ug/L
Method Blank Result	0.5	Method Blank Result	ND
Equipment Blank Result	0.35	Equipment Blank Result	0.6
CRQL	0.5	CRQL	0.5
Blank Action Level	2.5(5x0.5)	Blank Action Level	3.0 (5x0.6)

The endrin positive detect is qualified in the equipment blank and reported as 0.35U ug/L on the Data Summary Table. The blank action level for dieldrin is determined using the value from the equipment blank and as a result the dieldrin positive detect in the equipment blank is reported unqualified as 0.6 ug/L on the Data Summary Table. If positive hits for dieldrin are found in the samples, then the blank action level is applied at 3.0 ug/L (5 x 0.6 ug/L).

VI. SURROGATE ANALYTES

A. OBJECTIVE

Sample matrix effects and laboratory performance on individual samples are assessed by spiking the samples with surrogate analytes prior to extraction and analysis and determining their recoveries. Evaluation of surrogate recoveries is not necessarily straightforward. Interfering matrix effects, including high concentrations of target and/or non-target analytes, are frequently outside the control of the laboratory and may present relatively unique problems. Therefore, the evaluation and review of the surrogate analyte results are frequently subjective, demanding extensive analytical experience and professional judgment. Accordingly, this section consists primarily of guidance with several optional approaches suggested.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. The correct method-required surrogate analytes must be added to all samples, QC samples and blanks at the proper concentrations.

2. Recoveries for mandatory and advisory surrogate analytes in samples, QC samples and blanks must be within the QC acceptance criteria specified in the method.

3. The retention times for surrogates in samples, QC samples and blanks must be within the calculated retention time windows.

4. If surrogate analyte recoveries are outside the method QC acceptance criteria, then the pesticide/PCB sample must be reanalyzed in accordance with method requirements. If the recoveries are still outside the criteria, then the samples must be reextracted and reanalyzed.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from surrogate analyte anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
*1. Verify that the correct analytes were used as surrogate analytes and were added at the required concentration and frequency to all samples, QC samples, and blanks.	1. a. If surrogate analytes were not added to all samples, QC samples, and blanks, were added at the wrong concentration (for example a sample was "double" spiked) or an incorrect analyte was used, then the validator should use professional judgment to qualify or reject sample data. b. If surrogate analytes were diluted out of a sample, then the validator should use professional judgment to qualify or reject sample data. Greater than five-fold dilutions result in surrogate recovery data that may be analytically unusable.
2. Review Form II PEST to verify that no mandatory or advisory surrogate analyte recovery is outside the method QC acceptance criteria for pesticide/PCB field, QC, and blank samples. a. Determine whether or not a surrogate analyte was reported with a recovery above the upper QC acceptance limit on any GC column.	2. a. If a surrogate analyte in the pesticide/PCB sample has a recovery greater than the upper QC acceptance limit on any GC column, then the validator should: i. Use professional judgment to qualify positive detects in the affected sample based on the magnitude of the recovery and whether or not the upper limit was exceeded on more than one column. ii. Accept non-detects in the affected sample.

C. EVALUATION	D. ACTION
2. b. Determine whether or not a surrogate analyte was reported with a recovery below the lower QC acceptance limit on any GC column. If low surrogate recoveries are observed, then the validator should investigate whether the low recoveries were a result of sample dilution.	2. b. If a surrogate analyte in the pesticide/PCB sample has a recovery greater than or equal to 10% but less than the lower QC acceptance limit on any GC column, then the validator should: i. Use professional judgment to qualify positive detects in the affected sample based on the magnitude of the recovery and whether or not the lower limit was exceeded on more than one column. ii. Estimate (UJ) the sample quantitation limit for non-detects in the affected sample.
c. Determine if surrogate analytes were reported with extremely low recoveries, less than 10% on any GC column.	c. If a surrogate analyte in the pesticide/PCB sample recovers at less than 10% on any column, then the validator should: i. Estimate (J) positive detects in the affected sample. ii. Reject (R) non-detects as unusable in the affected sample. iii. If extremely low surrogate recoveries (less than 10%) were reported for the majority of samples in the sample delivery group, then the validator should use professional judgment to reject the entire pesticide/PCB fraction as unusable.

C. EVALUATION	D. ACTION
2. d. Determine if blank surrogate analyte recovery results meet method QC acceptance criteria.	2. d. In the special case of a blank analysis with a surrogate analyte recovery outside the method QC acceptance criteria, the validator must give special consideration to the validity of the associated sample data. The basic concern is whether or not the blank results represent an isolated problem with the blank, or whether there is a fundamental problem with the analytical process. For example, if most of the samples including other types of blanks in the batch demonstrate acceptable surrogate analyte recoveries, then the validator may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows the use of some of the affected data, analytical problems should be noted in the Data Validation Memorandum. All samples that were extracted with or analyzed after an out of control blank should be noted in the Data Validation Memorandum. Also, note in the Data Validation Memorandum if there are potential contractual problems associated with the failure to reextract and/or reanalyze blanks with surrogate analyte recoveries that were outside the method QC criteria.
3. a. Verify from Form VIII PEST that the absolute retention times for surrogates in the samples, QC samples and blanks are within the established retention time windows.	3. a. Retention time windows are essential to the qualitative identification of target analytes. Non-target analytes may appear as interferences in the retention time windows. The validator should be on guard for this possibility and look for interference trends throughout the entire case. If the surrogate analytes are not within the established retention time windows, then the validator should carefully evaluate the associated sample, QC sample, and blank results and raw data. This will necessitate a Tier III review.
* b. If reported retention times of the surrogate analytes are not within the established retention time windows, check the raw data for accurate identification of GC peaks. Non-recovery of surrogates may be due to shifts in retention time or matrix interference.	b. If the retention time of a surrogate analyte in the samples, QC samples, or blanks is outside of the calculated retention time windows, then the validator must use professional judgment to qualify the sample data. Refer to Section II. GC/ECD Instrument Performance Check, D.2 for guidance.

C. EVALUATION	D. ACTION
*3. c. Ten percent of the surrogate analyte raw retention time data should be checked for calculation and/or transcription errors. If errors are detected in this ten percent, then an additional ten percent should be checked. If errors are found in the additional ten percent, then the retention times of all peaks in the data package should be checked to evaluate whether or not results were reported accurately.	3. c. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
4. For Pesticide/PCB samples, verify that if surrogate analytes are outside the method QC acceptance criteria, then the required reextraction/reanalysis was performed to confirm that the non-compliance was due to sample matrix effects rather than poor laboratory performance.	4. If a laboratory fails to reextract and reanalyze a sample which is out of specification, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.
*5. a. Check raw data (e.g., chromatograms and quantitation reports) to verify that surrogate recoveries were reported accurately on the Surrogate Recovery Forms (Form II PEST-1 and Form II PEST-2). * b. Ten percent of the surrogate analyte recovery data should be checked for calculation and/or transcription errors. If errors are detected in this ten percent, then an additional ten percent of the data should be checked. If errors are found in the additional ten percent, then all surrogate analyte recovery calculations and transcriptions in the data package should be checked.	5. a. If there are any transcription errors, then the validator should have the laboratory resubmit all corrected raw data and forms. b. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1, C.3.b, C.3.c, C.5.a, C.5.b

Table Pest/PCB-VI-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON SURROGATE ANALYTE RECOVERIES

Surrogate Analyte Recovery				
Sample Results	**One or more surrogates %Rec < 10%**	**One or more surrogates 10% ≤ %Rec < LL**	**All surrogates LL ≤ %Rec ≤ UL**	**One or more surrogates %Rec > UL**
Detects	J	Professional Judgment	A	Professional Judgment
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Note: The surrogate recoveries in the method blank and the instrument blank must be within criteria for the analytical sequence to be valid.

E. EXAMPLES

Example #1: (Both pesticide surrogate recoveries < 10% on both columns)

Soil sample SA521, analyzed by CLP SOW OLM04.3, had TCX and DCB recoveries below 10% on both columns. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. SA521	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	5	7	30 - 150
Column 2	8	6	30 - 150

The validator estimates (J) positive detects and rejects (R) non-detects in sample SA521 on the Data Summary Table. The validator notes that low recoveries may be due to losses that occurred during the clean-up/extraction processes or chromatography problems and notes this in the Data Validation Memorandum.

E. EXAMPLES

Example #2: (One pesticide surrogate analyte recovery high on one column)

Soil sample MY207, analyzed by CLP SOW OLM04.3, had DCB recovered within advisory QC acceptance criteria on Column #1 and outside the upper limit of acceptance criteria on Column #2. TCX met advisory QC acceptance criteria on both columns. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. MY207	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	80	110	30 - 150
Column 2	125	155*	30 - 150

Non-detects are accepted. The validator uses professional judgment to determine that the high DCB surrogate recovery on Column #2 does not warrant qualification of the positive detects given that the criteria was only slightly exceeded on one column. The validator notes this in the Data Validation Memorandum.

Example #3: (Both pesticide surrogate analyte recoveries low on one column)

Soil sample NA351, analyzed by CLP SOW OLM04.3, had TCX and DCB recovered below the lower limit of QC acceptance criteria on Column #1 only (but greater than 10%). The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. NA351	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	15	12	30 - 150
Column 2	65	60	30 - 150

The validator uses professional judgment to determine that the TCX and DCB surrogate recoveries on Column #1 warrants qualification of the data. The validator notes that Column #1 data are suspect due to low recoveries and uses Column #2 to quantitate sample results. The validator estimates (J) positive detects and estimates (UJ) non-detects on the Data Summary Table. The validator documents this in the Data Validation Memorandum.

E. EXAMPLES

Example #4: (One pesticide surrogate analyte recovery high on both columns)

Soil sample ZY409, analyzed by CLP SOW OLM04.3, had DCB recovered above the upper limit of QC acceptance criteria on both Column #1 and #2. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. ZY409	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	100	200	30 - 150
Column 2	95	180	30 - 150

Aroclor 1254 and 1260 were detected in sample ZY409. The validator reviews the sample and standard chromatograms. The validator uses professional judgment to surmise that DCB recoveries were enhanced by coelution with unidentified contamination from the sample and disregards the high DCB surrogate recoveries. The validator also notes that the multicomponent peaks chosen for quantitation did not interfere with the DCB peak, and therefore, determines that the Aroclor quantitation is accurate. The validator accepts positive Aroclor detects in sample ZY409 based upon the compliant TCX surrogate recoveries. The validator notes this in the Data Validation Memorandum.

Example #5: (Both pesticide surrogate analytes low on both columns)

Aqueous sample QA129, analyzed by CLP SOW OLM04.3, had TCX and DCB recovered below the lower limit of QC acceptance criteria on both Column #1 and #2, but above 10%. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. QA129	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	27	23	30 - 150
Column 2	25	28	30 - 150

The validator uses professional judgment to determine that the low TCX and DCB surrogate recoveries on both Column #1 and #2 warrants qualification of positive detects. The validator estimates (J) the positive detects and estimates (UJ) the non-detects in sample QA129. The validator notes this in the Data Validation Memorandum.

VII. PESTICIDE/PCB CLEANUP

A. OBJECTIVE

Pesticide/PCB cleanup procedures are utilized to remove matrix interferences from sample extracts prior to analysis. If not removed from the sample extracts, matrix interferences can inhibit accurate analyte identification and quantitation resulting in highly suspect data. Pesticide/PCB cleanup procedures are evaluated by spiking the cleanup columns or cartridges with target analytes and assessing the recovery of these analytes through the cleanup procedure.

Several types of pesticide cleanup procedures exist, including but not limited to:

1. **Gel Permeation Chromatography (GPC)** - removes high molecular weight contaminants

   GPC is a size exclusion procedure that utilizes organic solvents and hydrophobic gels to separate of macromolecules. The packing gel is porous and is characterized by the exclusion range (range of uniformity) of that pore size. The exclusion range must be greater than those of the molecules to be separated.

   General applications of GPC as a cleanup procedure include the removal of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids and dispersed high molecular-weight analytes from the sample extract.

   Under CLP SOW OLM04.3, the GPC column is packed with bead-like packing and connected to a UV detector. After the GPC is calibrated and a blank analyzed, sample extracts are loaded into sample loops and an automated sequence is started. The target analytes are eluted with methylene chloride and collected during the pre-determined retention times. The high molecular weight interferences, those outside the exclusion range, elute earlier than the pesticide/PCB analytes during the “dump” phase, while the smaller interferents such as sulfur elute with a later volume of solvent during the “wash” phase.

2. **Florisil Cartridge Cleanup** - reduces matrix interferences

   Florisil is a magnesium silicate with basic properties that is used in column chromatography to reduce matrix interferences caused by polar analytes in pesticide/PCB sample extracts.

   Florisil is used in the cleanup of pesticide residues and other chlorinated hydrocarbons, the separation of nitrogen analytes from hydrocarbons, and the separation of aromatic analytes from aliphatic-aromatic mixtures. Florisil is also used in separating steroids, esters, ketones, glycerides, alkaloids, and some carbohydrates from pesticide analytes.

   A Florisil cleanup of pesticide/PCB extracts in hexane may be performed by transferring the extract to the top of a Florisil column and then eluting the column with a hexane/acetone mixture. The interferences are retained on the Florisil and the pesticide/PCB fraction is collected, concentrated and analyzed. Refer to CLP SOW OLM04.3 for method specific requirements.

   In some methods the Florisil cleanup is performed using multiple elutions of the cleanup column with hexane/ether mixtures of increasing polarity. The various eluant fractions, each containing different pesticide/PCB analytes, are then concentrated and analyzed either separately or as a combined extract. Refer to the EPA SW-846 method 3620B, December 1996 (or most recent revision), or EPA water method 608 for method-specific requirements.

3. **Sulfur Cleanup** - removes sulfur

Sulfur cleanup eliminates elemental sulfur. Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analysis of the later eluting pesticides. Three techniques available to remove sulfur are: the Mercury Technique, the Copper Technique and the Tetrabutylammonium (TBA) -Sulfite Reagent Technique. Refer to the CLP SOW OLMO4.2 for mercury and copper clean-up method-specific requirements. Refer to the EPA SW-846 method 3660B, December 1996 (or most recent revision) for copper and TBA-sulfite clean-up method-specific requirements.

4. **Sulfuric Acid/Permanganate Cleanup - suitable only for PCB analysis** - removes most organic chemicals

Sulfuric Acid/Permanganate cleanup destroys most organic chemicals including the pesticides Aldrin, Dieldrin, Endrin, Endosulfan (I and II), and Endosulfan sulfate. This method is suitable for the rigorous cleanup of sample extracts prior to analysis for PCBs. This method is used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs. Refer to the EPA SW-846 method 3665A, December 1996 (or most recent revision) for method-specific requirements.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. **Gel Permeation Chromatography**

a. Pesticide/PCB sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

b. The GPC system must be calibrated initially in accordance with the method prior to the analysis of field samples, QC samples, or blanks to ensure acceptable solid phase activation, peak shape, and resolution of target analytes and interferents.

i. GPC system must be calibrated and verified on a continuing basis at the frequency specified in the method.

ii. The method-required GPC calibration and calibration verification solutions must contain target analytes and interferents at the method-required concentrations.

iii. The calibration verification solution must be analyzed according to the analytical method. Target analyte recoveries must meet method QC acceptance criteria.

iv. Aroclor patterns between standards that have undergone GPC and those that have not must be similar.

v. Peak shapes must be symmetrical and resolution must meet method QC criteria.

vi. Retention time shifts between GPC calibrations must not exceed ±5% for bis(2-ethylhexyl)phthalate and perylene.

c. i. A GPC instrument blank must be analyzed after each GPC calibration and prior to sample analysis.

ii. Target analytes must not be present at greater than or equal to the quantitation limit for any target analyte in the GPC instrument blank.

2. **Florisil Cartridge Cleanup**

a. Pesticide/PCB sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

b. Each lot number of cleanup cartridges must be checked in accordance with the method prior to use to ensure acceptable solid phase activation and acceptable recovery of target analytes.

c. i. The cartridge performance check must be conducted at the frequency specified in the method.

ii. The cartridge performance check must be analyzed on a GC/EC meeting the initial calibration and calibration verification technical acceptance criteria.

iii. Percent recoveries for Florisil Cartridge Performance Check solutions, which contain analytes of interest and surrogate analytes must meet method QC acceptance criteria.

d. i. All QC samples associated with the sample extracts that are cleaned up using this method must also be processed through this cleanup method. QC samples must meet method QC acceptance criteria after Florisil cartridge cleanup.

3. **Sulfur Cleanup**

a. Pesticide/PCB sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

b. Sulfur removal is used for sample extracts containing sulfur that may interfere with the analysis of target analytes.

c. i. A sulfur blank is prepared separately when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then the method blank must also be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

ii. The sulfur cleanup blank is a modified form of the method blank, and, other than the frequency stated above, must meet all method QC criteria specified for the method blank.

4. **Sulfuric Acid/Permanganate Cleanup - suitable only for PCB analysis**

a. Sample extracts, QC sample extracts, and method blank extracts for PCB analysis must undergo all cleanup procedures required by the method.

b. Sulfuric Acid/Permanganate cleanup is used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs.

c. i. Blanks and replicate analysis samples must be subjected to the same cleanup procedures as the samples associated with them.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from pesticide cleanup anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Gel Permeation Chromatography (GPC)	**1. Gel Permeation Chromatography (GPC)**
a. Verify from Form I PEST and Form IX PEST-2 that GPC cleanup was performed according to the analytical method on all method-required sample extracts, QC sample extracts, and method blank extracts.	a. If GPC was not performed according to the analytical method on all method-required extracts, then the raw data should be reviewed for the presence of high molecular weight contaminants and professional judgment should be used to qualify or reject sample data. The validator should request sample cleanup and reanalysis if GPC was required by the method.

C. EVALUATION	D. ACTION
*1. b. GPC Calibration	1. b. GPC Calibration
* i. Verify that the GPC system was calibrated initially in accordance with the method requirements and that peak shape and resolution criteria were met.	i. If the GPC system was not calibrated initially in accordance with the method (prior to the analysis of field samples, QC samples or blanks) or fails to meet peak shape and/or resolution criteria or the initial calibration data are not available for review, then the validator should evaluate the last calibration verification analyzed just prior to sample analysis.
* ii. Review the raw GPC calibration data to verify that peaks are symmetrical and resolution meets method QC acceptance criteria for target analytes and interferents in the GPC calibration solution.	ii. If the GPC calibration method QC acceptance criteria do not meet peak shape and analyte resolution, then the raw sample data should be examined for the presence of high molecular-weight interferences or the loss of late eluting target analytes and professional judgment should be used to qualify or reject sample data. The validator should discuss the impact of unacceptable peak shape and resolution on the sample data in terms of high or low bias and/or the possibility of false negatives and note this in the Data Validation Memorandum.
* iii. Check the raw GPC calibration data to verify that retention times for bis(2-ethylhexyl)phthalate and perylene in the GPC calibration solution did not vary more than ± 5% between calibrations	iii. Retention time shifts indicate instrument performance problems that require laboratory corrective actions. If retention time shifts are excessive, the GPC cleanup procedure may be the cause of analyte losses and false negatives, and the validator should evaluate the sample data carefully and document all deficiencies in the Data Validation Memorandum.
* iv. Check the collect and dump cycle times in the GPC calibration chromatogram and compare it with the samples collect and dump cycle times. Verify that retention times have not shifted between the calibration and the sample runs.	iv. All samples collect and dump cycle times should be consistent with the calibration. If retention times have shifted, the dump and collection times determined by the calibration standard no longer will be appropriate. Professional judgement should be used to evaluate the data and qualify the data appropriately.

C. EVALUATION	D. ACTION
*1. c. GPC Blank	1. c. GPC Blank
* i. Verify that a GPC instrument blank was analyzed after each GPC calibration and prior to sample analysis.	i. If a GPC instrument blank was not analyzed at the correct frequency and in the proper sequence, then the validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
* ii. Verify that there are no target analytes present at greater than or equal to the quantitation limit in the GPC instrument blank.	ii. If any target analytes are detected in the GPC instrument blank at greater than or equal to the quantitation limit, then the quality of the GPC operation is suspect. The validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
d. GPC Calibration Verification	d. GPC Calibration Verification
* i. Confirm from the raw data that the GPC calibration verification was performed at the method-required frequency.	i. If GPC calibration verifications have not been performed at the method-required frequency, then the quality of the GPC operation may be suspect and the validator should use professional judgment to qualify or reject sample data.
* ii. Verify that a GPC calibration verification solution was analyzed in accordance with the method and that the correct target analytes, interferents, and concentrations were used.	ii. If a GPC calibration verification solution was not analyzed in accordance with the method or the correct analytes and/or concentrations were not used, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.

C. EVALUATION	D. ACTION
1. d. iii. Verify from Form IX PEST-2 that GPC calibration verification solution analyses meet method QC acceptance criteria for target analyte recoveries.	1. d. iii. If GPC calibration verification method QC acceptance criteria are not met, then the GPC calibration verification solution results should be used to qualify sample data for specific analytes included in the check solution. Professional judgment should be used to qualify or reject sample data for non-check solution analytes, taking into consideration the analyte's chemical class. The validator should discuss the impact of unacceptable recoveries on the sample data in terms of high or low bias and note this in the Data Validation Memorandum. • If a GPC calibration verification analyte recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that GPC calibration verification to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that GPC calibration verification.

C. EVALUATION	D. ACTION
1. d. iii. Continued from above.	1. d. iii. Continued from above. • If more than half of the GPC calibration verification analyte recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that GPC calibration verification to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that GPC calibration verification. Professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the GPC calibration verification analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the GPC column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum. • If a GPC calibration verification analyte recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that GPC calibration verification to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that GPC calibration verification to indicate potential low bias.

C. EVALUATION	D. ACTION
1. d. iii. Continued from above.	1. d. iii. Continued from above. • If more than half of the GPC calibration verification analyte recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that GPC calibration verification to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that GPC calibration verification to indicate potential low bias. Professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the GPC calibration verification analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the GPC column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum. • If a GPC calibration verification analyte recovery is less than 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that GPC calibration verification to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that GPC calibration verification to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
1. d. iii. Continued from above.	1. d. iii. Continued from above. • If more than half of the GPC calibration verification analyte recoveries are less than 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that GPC calibration verification to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that GPC calibration verification to indicate that the data are unusable due to the possibility of false negatives. Professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the GPC calibration verification analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the GPC column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum. • If more than half of the GPC calibration verification analyte recoveries are outside the method QC acceptance limits in one GPC calibration verification, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes, or the entire fraction for samples associated with that GPC calibration verification.

C. EVALUATION	D. ACTION
1. d. iv. Verify that Aroclor patterns in the GPC calibration verification analysis are similar to the corresponding Aroclor standard patterns of the Initial Calibration sequence.	1. d. iv. If Aroclor patterns of GPC calibration verification are not similar to the corresponding Aroclor patterns of the Initial Calibration sequence, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.
* e. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred. If result forms are not available, then the validator must review the cleanup logs to confirm that method required cleanups were performed.	e. If the laboratory made any calculation and/or transcription errors, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
f. Review surrogate, MS/MSD, and PES data to evaluate the operational effectiveness of the GPC cleanup.	f. If any analyte or analyte class has zero recovery indicating the possibility of false negatives and/or recovers low indicating a potential low bias, then the validator should discuss the possible false negatives and/or potential low bias in the Data Validation Memorandum and qualify and/or reject sample results according to the guidance provided in Sections VI, VIII and XI of Part III-Pest/PCB.

C. EVALUATION	D. ACTION
2. Florisil Cartridge Cleanup	**2. Florisil Cartridge Cleanup**
a. Verify from Form IX PEST-1 that a Florisil cartridge cleanup was performed according to the analytical method on all method-required sample extracts, QC sample extracts, and method blank extracts.	a. If Florisil cartridge cleanup was not performed according to the analytical method on all method-required extracts, then the data should be reviewed for the presence of interferents and professional judgment should be used to qualify or reject sample data. The validator should request sample cleanup and reanalysis if Florisil cartridge cleanup was required by the method.
b. i. Verify from Form IX PEST-1 that each Florisil cartridge lot used to cleanup samples was checked at least once prior to use, and at the proper frequency, in accordance with method requirements. Cartridges should be checked at least once for every 300 cartridges of a particular lot (EPA SW-846 method 3620B) or every 6 months of use for a particular lot (CLP SOW OLM04.3).	b. i. If each Florisil cartridge lot was not checked or was not checked at the proper frequency, then the solid phase may not be properly activated potentially resulting in unacceptable target analyte recoveries, the presence of interferents and possibly the loss of target analytes (false negatives). The validator should review the Florisil Cartridge Check recovery data associated with each batch of Florisil cartridge cleanups to ascertain if any target analytes should be qualified or rejected using the guidance provided in b.iii and c.
ii. Verify from Form IX PEST-1 that a Florisil Cartridge Performance Check solution was prepared and analyzed in accordance with the method and that the correct target and surrogate analytes, interferents, and concentrations were used.	ii. If a Florisil Cartridge Performance Check solution was not prepared and analyzed in accordance with the method or the correct analytes and/or concentrations were not used, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.

C. EVALUATION	D. ACTION
2. b. iii. Check the reported data from the Florisil Cartridge Performance Check solution analyses on Form IX PEST-1 to verify that the target analyte recoveries meet method QC acceptance criteria.	2. b. iii. If Florisil Cartridge Check method QC acceptance criteria are not met, then the Florisil Cartridge Performance Check solution results should be used to qualify sample data for specific analytes included in the check solution. Professional judgment should be used to qualify or reject sample data for non-check solution analytes. The validator should discuss the impact of unacceptable recoveries on the sample data in terms of high or low bias and note this in the Data Validation Memorandum. • If a Florisil Cartridge Performance Check solution analyte recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that Florisil Cartridge Performance Check solution. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that Florisil Cartridge Performance Check solution.

C. EVALUATION	D. ACTION
2. b. iii. Continued from above.	2. b. iii. Continued from above. • If a Florisil Cartridge Performance Check solution analyte recovery is less than the lower limit but greater than 10% of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential low bias.

C. EVALUATION	D. ACTION
2. b. iii. Continued from above.	2. b. iii. Continued from above. • If a Florisil Cartridge Performance Check solution analyte recovery is less than the 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that Florisil Cartridge Performance Check solution to indicate that the data are unusable due to the possibility of false negatives. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are less 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. b. iii. Continued from above.	2. b. iii. Continued from above. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are outside the method QC acceptance limits in one Florisil Cartridge Check, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes, or the entire fraction for samples associated with that Florisil Cartridge Check. • If the 2,4,5-trichlorophenol recovery was ≥ 5% in the analysis of the Florisil Cartridge Performance Check solution, then the Florisil is not adequately removing non-target analytes and the sample data must be evaluated for potential interferences. Professional judgment should be used to qualify or reject sample data. This may necessitate a Tier III validation. In all cases, professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the Florisil Cartridge Performance Check solution analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the Florisil column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum.
* iv. Verify that surrogate analyte recoveries in the Florisil Cartridge Performance Check solution meet method QC acceptance criteria.	iv. If surrogate analyte recoveries and/or retention times in the Florisil Performance Check solution do not meet method QC acceptance criteria, then the validator should use professional judgement to assess the sample data and qualify the sample data in accordance with Section VI, Surrogate Analytes.

C. EVALUATION	D. ACTION
2. c. Verify from Form IX PEST-1 that all QC samples and method blanks associated with the sample extracts that were Florisil cleaned were also Florisil cleaned. All QC samples and method blanks must meet method-specified criteria after Florisil cleanup.	2. c. If Florisil cartridge cleanup was not performed for associated QC samples and/or method blanks, then the data should be reviewed for potential impacts and professional judgment should be used to qualify or reject sample data. If the QC samples and method blanks do not meet QC criteria after Florisil cleanup, then the validator should refer to the appropriate section of Part III-Pest/PCB, and use professional judgment to qualify sample data.
* d. Compare the raw data, if available, to the reported results and verify that no calculation and/or transcription errors have occurred. If result forms are not available, then the validator must review the cleanup logs to confirm that method required cleanups were performed.	d. If the laboratory made any calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
e. Review MS/MSD, surrogate, and PES data to evaluate the operational effectiveness of the Florisil Cartridge cleanup.	e. If any analyte or analyte class has zero recovery indicating the possibility of false negatives and/or recovers low indicating a potential low bias, then the validator should discuss the possible false negatives and/or potential low bias in the Data Validation Memorandum and qualify and/or reject sample results according to the guidance provided in Sections VI, VIII and XI of Part III-Pest/PCB.

C. EVALUATION	D. ACTION
3. Sulfur Cleanup	**3. Sulfur Cleanup**
a. i. Review the Form I Pest to ascertain if sulfur cleanup was performed on any sample extracts, and associated QC samples and method blanks.	a. i. If a Tier II validation is being performed, then the validator should note that sulfur cleanup was performed and that reducing conditions may exist at the sample site location.
* ii. Check the field sample GC chromatograms to determine whether or not there is a flat baseline. A rising baseline may indicate the presence of sulfur. Confirm that all pesticide/PCB peaks are adequately resolved and are symmetrical.	ii. If a method-required sulfur cleanup was not performed on sample extracts that contain sulfur or adequate sulfur removal was not achieved, then the validator should carefully assess the impact on the sample data. If only minor sulfur interference is observed, then the validator should use professional judgment to estimate (J) positive detects for analyte(s) that coelute with sulfur and reject (R) non-detects. If the sulfur peak obscures a limited, discrete portion of the chromatogram, then the validator should use professional judgment to reject (R) the positive detects and non-detects for analytes coeluting with sulfur in that portion of the chromatogram and accept the unaffected sample results. If the sulfur contamination is gross and the majority of the chromatogram is obscured, then the validator should use professional judgment to reject (R) the entire pesticide/PCB analysis for that sample. The validator should request sample reanalysis that includes sulfur removal.
* iii. Confirm from the raw data, laboratory bench sheets, or SDG Narrative, that a method-required cleanup technique was used to remove any sulfur present in the samples	iii. If a method-required sulfur cleanup technique was not used for sulfur removal, then the validator should request sample cleanup and reanalysis and document all technical decisions in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. b. i. Verify from Form IV PEST that a sulfur cleanup blank was prepared and analyzed along with samples, or that the associated method blank was also sulfur cleaned.	3. b. i. If a sulfur cleanup blank was not prepared and/or analyzed with the samples, or the associated method blank was not also sulfur cleaned, then the validator should use professional judgment to qualify sample data.
ii. Verify that the sulfur cleanup blank met all method QC acceptance criteria specified for the method blank (refer to Section V, Blanks).	ii. If the sulfur cleanup blank does not meet QC criteria after sulfur cleanup, then the validator should refer to Section V, Blanks, and use professional judgment to qualify sample data.
* iii. Verify from the raw data that there are no target analytes greater than the quantitation limit present in the sulfur cleanup blank.	iii. If any target analytes are detected in the sulfur cleanup blank greater than or equal to the quantitation limit, then the sulfur cleanup may be a source of contamination. The validator must use professional judgment in conjunction with guidance provided in Section V, Blanks to qualify sample data.
* iv. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred.	iv. If discrepancies between the raw and reported data are found, the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
4. Sulfuric Acid/Permanganate Cleanup - suitable for PCB analysis only	**4. Sulfuric Acid/Permanganate Cleanup**
* a. i. Review the raw data, laboratory bench sheets, or SDG Narrative, to ascertain if sulfuric acid/permanganate cleanup was performed on all method-required sample extracts, QC sample extracts, and method blank extracts	a. i. If a method-required sulfuric acid/permanganate cleanup technique was not used, then the validator should request sample cleanup and reanalysis and document all technical decisions in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*4. a. ii. Check the field sample GC chromatograms to determine whether or not there are interferences causing elevated baselines or overly complex chromatograms. Confirm that all PCB peaks are adequately resolved and are symmetrical.	4. a. ii. If a method-required sulfuric acid/permanganate cleanup was not performed on sample extracts that contain interferences, or adequate interference removal was not achieved, then the validator should carefully assess the impact on the sample data. The validator should use professional judgment to accept, qualify, or reject the data.
* b. i. Verify from the raw data that the associated QC samples and method blanks was also sulfuric acid/ permanganate cleaned.	b. i. If the associated QC samples and/or method blank was not also sulfuric acid/permanganate cleaned, then the validator should assess the potential impacts on the sample data and use professional judgment to qualify the data.
ii. Verify that the associated QC samples and method blanks met all method-specified QC acceptance criteria after sulfuric acid/permanganate cleanup (refer to the appropriate sections of Part III-Pest/PCB).	ii. If the QC samples and method blanks does not meet QC criteria after sulfuric acid/permanganate cleanup, then the validator should refer to the appropriate sections of Part III-Pest/PCB, and use professional judgment to qualify sample data.
* iii. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred.	iii. If discrepancies between the raw and reported data are found, the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.1.c.i, C.1.c.ii, C.1.c.iv, C.1.c.v, C.1.c.vi, C.1.d.i, C.1.d.ii, C.1.e, C.2.b.iv, C.2.d, C.3.a.ii, C.3.a.iii, C.3.b.iii, C.3.b.iv, C.4.a.i, C.4.a.ii, C.4.b.i, C.4.b.iii.

Table Pest/PCB-VII-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON GPC CALIBRATION QUALITY CONTROL

	Criteria	Action
Peak Resolution	As per method QC acceptance criteria.	Professional Judgment
Peak Shape	Peak shapes must be symmetrical.	Professional Judgment
Aroclor Pattern	After GPC is performed, Aroclor 1016 and 1260 standard patterns must be similar to Aroclor patterns in the Initial Calibration sequence.	Professional Judgment
Retention Time	Retention time shifts between GPC calibrations for bis(2-ethylhexyl)phthalate and perylene must not exceed ± 5%.	Professional Judgment
GPC Instrument Blank	Target analytes must be less than the quantitation limit.	Refer to Section V for Blank Actions

Table Pest/PCB-VII-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON GPC CALIBRATION VERIFICATION QUALITY CONTROL

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < Lower Limit	Lower Limit ≤ %Rec ≤ Upper Limit	%Rec > Upper Limit
Detects	J	J	A	J
Non-detects	R	UJ	A	A

Note: Professional judgment should be used in applying the guidance above to qualify or reject sample data.

Table Pest/PCB-VII-3:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON FLORISIL CARTRIDGE CLEANUP QUALITY CONTROL

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < Lower Limit	Lower Limit ≤ %Rec ≤ Upper Limit	%Rec > Upper Limit
Detects	J	J	A	J
Non-detects	R	UJ	A	A
2,4,5-TCP Recovery Criterion	If 2,4,5-Trichlorophenol recovers at ≥ 5%, then the Florisil is not working properly and the data must be evaluated for potential interferences.			

Note: Professional judgment should be used to qualify the data when a combination of low recoveries and high recoveries are obtained.

Table Pest/PCB-VII-4:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON SULFUR CLEANUP QUALITY CONTROL

Sample Results	Degree of Sulfur Interference		
	Minor	Limited to discrete part of the sample chromatogram	Major
Detects	Estimate (J) positive detects for the affected analytes.	Accept positive detects that are not impacted by sulfur interference. Reject (R) positive detects for those analytes coeluting with the sulfur peak.	Reject (R) all detects for the affected sample and request sample reanalysis that includes sulfur cleanup.
Non-detects	Use professional judgement to evaluate the non-detects.	Accept non-detects that are not impacted by sulfur interference. Reject (R) non-detects for those analytes coeluting with the sulfur peak.	Reject (R) all non-detects for the affected sample and request sample reanalysis that includes sulfur cleanup.

Note: Professional judgment should be used in applying the above guidance to qualify or reject sample data.

E. EXAMPLES

Example #1: (Florisil % Rec > 120% for one analyte)

The validator examines Form IX PEST-1 to verify that the percent recoveries from the Florisil Cartridge Check Solution analysis meet QC acceptance criteria (80-120%). The validator notes that dieldrin was recovered at 150%. The validator uses professional judgment to estimate (J) the positive dieldrin detects associated with this Florisil batch and accept (A) the quantitation limits for dieldrin non-detects on the Data Summary Table. The validator notes in the Data Validation Memorandum that a high bias exists for dieldrin and that positive detects for dieldrin may actually be lower than the reported results.

Example #2: (Florisil % Rec < 80% for six analytes)

The validator examines Form IX PEST-1 to verify that the percent recoveries from the Florisil Cartridge Check Solution analysis meet QC acceptance criteria (80-120%). The validator notes that alpha-BHC, heptachlor, endosulfan I, endrin, 4,4'-DDT, and methoxychlor showed the following recoveries: 75%, 65%, 32%, 70%, 41%, and 9%, respectively. The validator concludes that the Florisil batch used for sample cleanup has resulted in a low bias for pesticide and PCB results. Therefore, the validator uses professional judgment to qualify all sample data associated with this Florisil batch. The validator estimates (J) the positive pesticide/PCB detects and estimates (UJ) all the quantitation limits for non-detects with the exception of the quantitation limits for methoxychlor which are rejected (R). The validator reports the qualified data on the Data Summary Table and discusses the low bias in the Data Validation Memorandum.

Example #3: (GPC % Rec < 80% for one analyte)

The validator examines Form IX PEST-2 to verify that the percent recoveries from the GPC Calibration Verification meet QC acceptance criteria (80-110%). The validator notes that endrin was recovered at 60%. The validator also reviews the GPC calibration data for peak shape, resolution, and retention time shift to verify that the proper collection and dump cycles were utilized to ensure that all interferences were removed without loss of target analytes. The validator concludes that the GPC was calibrated correctly. The validator uses professional judgment to estimate (J) the positive endrin detects and estimate (UJ) the quantitation limits for endrin non-detects for all samples associated with the non-compliant GPC Calibration Verification. The validator reports the qualified data on the Data Summary Table and discusses the reasons for sample qualification and the low bias in the Data Validation Memorandum.

VIII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. OBJECTIVE

Data for matrix spike/matrix spike duplicates (MS/MSDs) are generated at the time of sample preparation and analysis to determine laboratory precision and method bias for specific sample matrices. MS/MSD data can be used to determine long-term interlaboratory precision and bias of an analytical method for various matrices and are used in setting quality control acceptance criteria for spiking analytes. MS/MSD data should be used in conjunction with other QC data, such as field duplicate data and surrogate analyte recoveries, to determine if a sample or an entire sample group should be qualified.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP. If no deviations or modifications to the method QC acceptance criteria have been defined, then the QC acceptance criteria in the method would be applied in the data validation.

1. In accordance with the SAP, QAPP, and/or method, a field sample of each matrix is spiked in duplicate with known concentrations of specific target analytes to generate an MS/MSD pair. Concurrently, the laboratory analyzes an unspiked aliquot and the MS/MSD pair of the field sample.

2. a. Field samples (not trip, equipment, or bottle blanks and not PE samples) must be spiked to assess matrix effects.

   b. Field samples chosen for MS/MSD analysis should not contain high levels of MS/MSD spiking analytes prior to spiking. Preferably, field samples chosen for MS/MSD analysis should contain low levels of the spiking analytes.

3. Recovery of the spiked analytes must be within the QC acceptance criteria specified in the QAPP/SAP or method.

4. Relative percent differences (RPDs) between MS and MSD recoveries must be within the QC acceptance criteria specified in the QAPP/SAP or method.

5. The percent relative standard deviation (%RSD) between positively detected non-spiked analytes in the unspiked sample, MS, and MSD must be less than or equal to 50%.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from matrix spike/matrix spike duplicate anomalies should be noted in the Data Validation Memorandum. Contractual non-compliance issues concerning the MS/MSD requirements must be discussed in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct analytes were added at the required concentrations; that MS/MSD samples were analyzed at the proper frequency; and that MS/MSD results are provided for each sample matrix.	1. If the laboratory did not use the required analytes at the concentration and frequency specified in the method for each sample matrix, then the validator must use professional judgment and the results from the other QC parameters, such as surrogate analyte recoveries and field duplicate precision, to determine the proper qualifications for the sample results.
2. a. Verify that a field sample was chosen for the MS/MSD.	2. a. If an equipment or bottle blank, or PE sample was spiked with the MS solution for the MS/MSD, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing laboratory precision, method bias, sample matrix effects and ultimately data usability.
b. Determine if an inappropriate sample containing high levels of the spiking analytes was chosen for the MS/MSD pair.	b. If the MS/MSD analytes were present in the field sample at high concentrations (e.g., 4x spike concentration) before spiking, then the validator must use professional judgment in assessing matrix spike recoveries and RPDs.
c. Ascertain if the MS/MSD analyses required dilutions.	c. If no MS/MSD data can be reported because of sample dilution, then the validator should note this problem in the Data Validation Memorandum and discuss its impact on assessing data usability in the case where laboratory precision and method bias information are absent.

C. EVALUATION	D. ACTION
3. Verify that all spike recoveries are within the QC acceptance criteria specified in the QAPP/SAP or method.	3. a. If any spiked analyte recovery result is greater than the upper limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Accept the non-detect for that affected analyte in the unspiked sample. b. If any recovery result is greater than or equal to 10%, but less than the lower limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Estimate (UJ) the non-detect for that affected analyte in the unspiked sample. c. If any recovery result is less than 10%, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Reject (R) the non-detect for that affected analyte in the unspiked sample. d. If the majority of spike analyte recoveries are outside the method QC acceptance criteria, then the validator may use professional judgment to estimate (J) or reject (R) <u>all</u> positive detects and estimate (UJ) or reject (R) <u>all</u> non-detects in the unspiked sample. Consideration should also be given to qualifying all the results of a particular matrix. See section C.8 for additional guidance.

C. EVALUATION	D. ACTION
4. Verify that the RPDs between the MS and MSD meet the QC acceptance criteria specified in the QAPP/SAP or method.	4. If an RPD result is outside the method QC acceptance criteria, then the validator should: a. Assess whether or not the appropriate RPD acceptance criteria were applied for the situation at hand. b. Estimate (J) the positive detect for that affected analyte in the unspiked sample. c. Estimate (UJ) the non-detect for that affected analyte in the unspiked sample. d. If the majority of the matrix spike RPDs are outside method QC acceptance criteria, then the validator should use professional judgment to estimate (J) <u>all</u> positive detects and estimate (UJ) or reject (R) <u>all</u> non-detects in the unspiked sample. Consideration should also be given to the possibility of qualifying all the results of a particular matrix. Refer to section C. 8 and 9 for additional guidance.
5. a. Calculate the % RSD for the non-spiked target positive detects in the unspiked sample, the MS, and the MSD.	5. a. If a non-detected result or a detect less than the quantitation limit is reported for an analyte in one of the samples in the MS, MSD, or unspiked sample set, then the validator should use the sample quantitation limit value for that analyte to calculate the %RSD. If a non-detected result or a detect less than the quantitation limit is reported for an analyte in two of the samples in the MS, MSD, or unspiked sample set, then the validator should not calculate the %RSD but should use professional judgment to qualify sample data.

C. EVALUATION	D. ACTION
5. b. The unspiked sample, MS, and MSD may be considered a triplicate in determining the overall precision of the analytical method. Therefore, evaluate the %RSD data for positive detects in the triplicate set.	5. b. If any %RSD is greater than the method-specific criteria, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Use professional judgment to estimate (UJ) or accept the non-detect for that affected analyte in the unspiked sample. If overall laboratory precision for the unspiked field sample, MS, and MSD is poor, then the validator may use professional judgment to qualify <u>all</u> positive detects and non-detects in the unspiked sample. The Data Validation Memorandum should include a discussion of the potential impact of laboratory precision on representativeness and usability of the data in meeting the project DQOs.
*6. Check and recalculate the analytical concentrations and percent recovery for at least one spiked analyte per MS/MSD fraction. Verify that the recalculated value agrees within ± 10% of the reported value.	6. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*7. Check and recalculate the RPD for at least one spiked analyte per MS/MSD fraction. Verify that the recalculated value agrees within ± 10% of the reported value.	7. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
8. Evaluate the appropriateness of qualifying the entire data set based on MS/MSD laboratory precision and method/matrix bias results.	8. Generally, no action is taken based on the MS/MSD data alone to qualify all samples of a particular matrix. The qualification is limited to the unspiked sample associated with the MS/MSD. However, professional judgment may be used to qualify sample results across a matrix (i.e., all associated groundwater samples or a homogeneous soil matrix).
9. Evaluate MS/MSD precision data to confirm the laboratory's ability to generate precise data in conjunction with surrogate analyte recoveries and field duplicate precision data to assess overall precision.	9. If precision data for the laboratory MS/MSD pair, surrogate analyte recoveries, and the field duplicate pair indicate a heterogenous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify all affected analytes and/or all field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting the project DQOs should be discussed. Refer to Section IX for additional guidance.

* **Note:** **The following subsections are applicable only to a Tier III data validation:**

C.6, C.7

Table Pest/PCB-VIII-1:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MATRIX SPIKE RECOVERIES AND RPDS **

Sample Results	Recovery < 10%	10% ≤ Recovery < Lower QC Limit	Lower QC Limit ≤ Recovery ≤ Upper QC Limit	Recovery > Upper QC Limit	RPD > QC Limit
Detects	J	J	A	J	J
Non-detects	R	UJ	A	A	UJ

** Note that qualification and rejection generally are limited to the spiking analytes, however, the validator may use professional judgment to qualify or reject all positive detects or non-detects in the unspiked sample, or even all results of a particular matrix, if the majority of spike analyte recoveries and/or RPDs are outside the method QC acceptance criteria.

Table Pest/PCB-VIII-2:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MS, MSD, AND UNSPIKED SAMPLE %RSD

Sample Results	%RSD ≤ 50%*	%RSD > 50%*	Two out of three sample results reported as non-detects
Detects	A	J	Professional Judgment
Non-detects	A	Professional Judgment	Professional Judgment

* If a non-detect is reported for an analyte in only one of the samples in the MS, MSD, or unspiked sample set, then the validator should use the sample quantitation limit value for that analyte to calculate the %RSD.

E. EXAMPLES

Example #1: (High MS/MSD RPD for one analyte)

Soil QC samples SAA99MS and SAA99MSD, analyzed under CLP SOW OLM04.3, have unacceptable RPD results for aldrin. Aldrin was detected in the unspiked sample, SAA99.

Sample No.	Analyte	MS/MSD % Recovery	MS/MSD % Criteria	MS/MSD RPD	MS/MSD RPD Criteria
SAA99MS SAA99MSD	Aldrin	60/116	34 - 132	64*	43

* outside QC limit

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator then concludes that the lack of laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) the positive detect for aldrin in the unspiked sample, SAA99, on the Data Summary Table. The validator discusses the lack of laboratory precision for one analyte, aldrin, in the Data Validation Memorandum and notes that laboratory precision for other pesticide matrix spike analytes was acceptable.

Example #2: (Low MS/MSD recoveries for one analyte)

Soil QC samples SAA22MS and SAA22MSD, analyzed under CLP SOW OLM04.3, have one unacceptable recovery result but an acceptable RPD result for heptachlor. Heptachlor was not detected in the unspiked sample, SAA22.

Sample No.	Analyte	MS/MSD Recovery	MS/MSD Recovery Criteria	MS/MSD RPD	MS/MSD RPD Criteria
SAA22MS SAA22MSD	Heptachlor	30*/40	35 - 130	29	31

*outside QC limit

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator concludes that the sample matrix causes a reproducible negative bias for heptachlor in soil samples SAA22MS and SAA22MSD. The validator estimates (UJ) the non-detect for heptachlor in the unspiked sample, SAA22, on the Data Summary Table. The validator discusses the low matrix spike recovery in the Data Validation Memorandum and notes that recoveries for the other pesticide matrix spike analytes were acceptable.

Example #3: (High %RSD; High RPD, poor laboratory precision)

Soil samples SAA55, SAA55MS, and SAA55MSD, analyzed under CLP SOW OLM04.3, had high RSDs for 4,4'-DDD and 4,4'-DDE. The validator assesses the matrix spike results and notes that the pesticides had acceptable recoveries, however, three of the pesticides, gamma-BHC (67%), heptachlor (55%), and aldrin (72%) had high RPDs.

Sample No.	Analyte	MS Conc. Dry Weight (ug/kg)	MSD Conc. Dry Weight (ug/kg)	Unspiked Sample Conc. Dry Weight (ug/kg)	% RSD	% RSD Criteria
SAA55	4,4'-DDD	22	7	40	72*	50
SAA55	4,4'-DDE	5	13	3.3U	73*	50

* outside QC limit

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator uses professional judgement to estimate (J, UJ) just the three MS analytes (gamma-BHC, heptachlor, and aldrin) with high % RPD in only the unspiked sample SAA55.

The unspiked sample chromatogram is also examined and no interfering peaks are noted. The validator considers that although the DDT MS recovery was acceptable, the poor laboratory precision was a result of inlet degradation effects that interfere with the efficient reproducible analysis of 4,4'-DDD and 4,4'-DDE. The validator uses professional judgment to estimate (J) the positive 4,4'-DDD detect and (UJ) the 4,4'-DDE non-detect on the Data Summary Table. The validator notes the sample qualifications in the Data Validation Memorandum.

E. EXAMPLES

Example #4: (Low MS/MSD recoveries for multiple analytes)

Soil QC samples SAA09MS and SAA09MSD, analyzed under CLP SOW OLM04.3, have low spike analyte recoveries for four of the six analytes in the matrix spike and matrix spike duplicate (less than the specified QC acceptance criteria but greater than 10%). The validator notes that the recoveries for both pesticide surrogates are acceptable but slightly low in the MS and MSD unspiked samples.

Sample No.	Analyte	MS % Recovery	MSD % Recovery	RPD	QC Acceptance Criteria	
					% Recovery	RPD
SAA09 MS/MSD	Heptachlor	21*	28*	29	35-130	31
	Aldrin	29*	31*	6.7	34-132	43
	Dieldrin	27*	21*	25	31-134	38
	Endrin	30*	34*	13	42-139	45
	DCB (surrogate)	40	45	NA	30-150	NA
	TCX (surrogate)	38	35	NA	30-150	NA

* outside QC limit

Upon review of the MS/MSD results and surrogate recoveries, the validator notes that the sample matrix causes a reproducible negative bias for pesticide analytes in the MS/MSD samples. The validator reviews the unspiked sample surrogate analyte recoveries and notes that they are also low but within QC acceptance criteria (at the low end of the QC acceptance range). The validator then reviews the surrogate analyte recoveries for all samples with this matrix associated with the sample delivery group to ascertain if surrogate recoveries are also low in the remaining samples.

Several samples, including the field duplicates, show low surrogate recoveries that were greater than 10%. The validator estimates (J) all positive detects in the unspiked sample and estimates (UJ) all non-detects in the unspiked sample. The validator uses professional judgment to estimate (J) the positive detects and estimate (UJ) the non-detects in all other samples associated with this sample delivery group in which surrogates recovered low. The validator reports qualified data in the Data Summary Table and discusses the low bias in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (High MS/MSD RPDs for multiple analytes)

Aqueous QC samples SAA01MS and SAA01MSD, analyzed under CLP SOW OLM04.3, have high RPD values for five out of the six analytes in the matrix spike/matrix spike duplicate pair. The matrix spike and matrix spike duplicate analyte recoveries were all within QC acceptance criteria. The surrogate recoveries were acceptable for both the MS and the MSD. The validator notes the lack of precision in the unspiked analytes.

Sample No.	Analyte	MS % Recovery	MSD % Recovery	RPD	QC Acceptance Criteria	
					% Recovery	RPD
SAA01	gamma-BHC (Lindane)	58	86	39*	56-123	15
	Heptachlor	45	85	62*	40-131	20
	Aldrin	70	118	51*	40-120	22
	Endrin	75	120	46*	56-121	21
	4,4'-DDT	50	100	66*	38-127	27

*outside QC limits

Sample No.	Analyte	MS Conc. (ug/L)	MSD Conc. (ug/L)	Unspiked Sample Conc. (ug/L)	% RSD	% RSD Criteria
SAA01	4,4'-DDE	10	65	90	74*	50
	delta-BHC	45	25	10U	93*	50

*outside QC limits

Upon review of the MS/MSD results, surrogate recoveries, and the % RSDs, the validator notes the laboratory imprecision and suspects that problems occurred during extraction and/or analysis of the MS/MSD sample and/or unspiked sample. The validator then reviews the field duplicate data and surrogate recoveries for the remaining samples and QC samples in the sample delivery group to assess precision and bias data.

Surrogate recoveries in all other samples were acceptable. The field duplicate RPD data were also acceptable. Therefore, the validator determines that poor precision was limited to the MS/MSD pair. The validator used professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects in the unspiked sample SAA01 on the Data Summary Table. The validator notes this problem in the Data Validation Memorandum.

IX. FIELD DUPLICATES

A. OBJECTIVE

Field duplicates measure the cumulative effects of both field and laboratory precision and hence provide an indication of overall precision. Therefore, field duplicates may have greater variability than laboratory duplicates which measure only laboratory precision. It is also expected that non-aqueous matrices will have a greater variance than aqueous matrices due to the heterogeneity of most non-aqueous samples (such as soil/sediment samples).

B. CRITERIA

1. The frequency of field duplicate analysis must support the site-specific Data Quality Objectives (DQOs) and be documented in the EPA approved QAPP or SAP.

2. a. The Relative Percent Difference (RPD) for all analytes detected at concentrations greater than the sample quantitation limit in aqueous matrices must be less than or equal to 30 percent.

   b. The RPD for all analytes detected at concentrations greater than the sample quantitation limit in non-aqueous matrices must be less than or equal to 50 percent.

3. In situations where the RPD criteria are not specified in the QAPP, it is recommended to use the criteria found in 2.a. and 2.b. above.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from field duplicate anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Identify which samples are field duplicates from the Chain-of-Custody form and/or the Traffic Report.	1. a. If field duplicates are not listed on the Chain-of-Custody form or the Traffic Report, then the validator should contact the sampler to determine if field duplicates were collected. If the forms were completed incorrectly or if field duplicates were not collected, then the validator should document this on the Data Validation Worksheet and in the Data Validation Memorandum.
b. Verify that the appropriate number of field duplicates per matrix sampled were collected and analyzed to support the project DQOs.	b. If field duplicates were not collected at the required frequency to support the project DQOs, then the validator should note the absence of field precision data in the Data Validation Memorandum and discuss how the lack of field precision data might potentially increase uncertainty surrounding site decisions.

C. EVALUATION	D. ACTION
2. Calculate the RPD for all analytes detected at concentrations greater than or equal to the sample quantitation limit in the field duplicate sets. Record the RPDs on the appropriate worksheet.	2. a. If any analyte is detected at a concentration greater than or equal to twice the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should estimate (J) the positive detects for that analyte in both samples. If any analyte is detected at a concentration greater than or equal to the sample quantitation limit but less than twice the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should use professional judgment to accept, qualify, or reject the positive detects for that analyte in the field duplicate samples taking into consideration the increased variability of data near the sample quantitation limit and the site-specific DQOs. b. If any analyte is detected at a concentration greater than or equal to twice the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should estimate (J) the positive detects for that analyte in both samples. If any analyte is detected at a concentration greater than or equal to the sample quantitation limit but less than twice the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should use professional judgment to accept, qualify, or reject the positive detects for that analyte in the field duplicate samples taking into consideration the increased variability of data near the sample quantitation limit and the site-specific DQOs.

C. EVALUATION	D. ACTION
2. Continued from above.	2. c. If any analyte in a field duplicate pair has one positive detect that is greater than or equal to twice the sample quantitation limit and a duplicate positive detect that is less than twice the sample quantitation limit, and the RPD exceeds field duplicate precision criteria for that matrix, then the validator should use professional judgment to qualify the positive detects for that analyte in the field duplicate samples. d. If any analyte in a field duplicate pair has one non-detect and a duplicate positive detect that is greater than or equal to twice the sample quantitation limit, then the validator should estimate (J) the positive detect and (UJ) the non-detect for that analyte in the field duplicate samples. (RPDs should not be evaluated for those duplicate pairs.) e. If any analyte in a field duplicate pair has one non-detect or a reported value below the sample quantitation limit and a duplicate positive detect that is at or above the sample quantitation limit but less than twice the sample quantitation limit, then the validator should use professional judgment to qualify the positive detects and non-detects for that analyte in the field duplicate samples taking into consideration the increased variability of data at the sample quantitation limit and the project DQOs. (RPDs should not be evaluated for those duplicate pairs.) f. If any analyte in a field duplicate pair has one non-detect or a reported value below the sample quantitation limit and a duplicate positive detect that is less than the quantitation limit, then the validator should use professional judgment to qualify the positive detects and non-detects for that analyte in the field duplicate samples taking into consideration the increased variability of data at the sample quantitation limit and the project DQOs. (RPDs should not be evaluated for those duplicate pairs.)

C. EVALUATION	D. ACTION
*3. Check and recalculate the analytical concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or soil sample) for each fraction, in every field duplicate sample, in accordance with Section Pest/PCB-XIII, C.	3. If calculation and/or transcription errors are detected, then the validator should follow the procedures outlined in Section Pest/PCB XIII, D.
4. Evaluate the appropriateness of qualifying the entire data set based on field duplicate results.	4. If field duplicate data indicate poor field precision and general sample heterogeneity and/or possible sampling error, then professional judgment may be used to qualify data for all samples of the same matrix.
5. Evaluate field duplicate precision data to assess overall precision and to verify the field sampler's ability to collect representative duplicate samples. MS/MSD precision data should be evaluated to verify the laboratory's ability to generate precise data. Surrogate recovery data can also be evaluated to identify laboratory precision issues and overall matrix precision issues.	5. If precision data for the field duplicate pair, surrogate analyte recoveries, and the laboratory MS/MSD pair indicate a heterogeneous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify all affected analytes and/or all affected field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting project DQOs should be discussed. Refer to Section VIII for additional guidance.

*** Note: The following subsections are applicable only to a Tier III validation:**

C.3

Table Pest/PCB-IX-1:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES
SITUATION 1: POSITIVE DETECTS IN BOTH FIELD DUPLICATES

Relative Percent Difference	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30% Non-Aqueous > 50%
Sample Results	**Both duplicate sample concs. ≥ 2 X QL**	**QL ≤ both duplicate samples concs. < 2 X QL**	**One sample conc. ≥ 2 X QL QL ≤ One sample conc. < 2 X QL**
Detects	J	Professional Judgment	Professional Judgment

* QL = Sample Quantitation Limit
N/A = Not Applicable

Note: Qualification refers to field duplicate sample results only. Professional judgment may be used to apply field duplicate actions to all samples of the same matrix.

Table Pest/PCB-IX-2:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES
SITUATION 2: POSITIVE DETECT IN ONLY ONE FIELD DUPLICATE**

Aqueous and Non-Aqueous		
Sample Results	**One Sample Conc. = ND (or values reported as less than the QL) QL ≤ One Sample Conc. < 2 X QL**	**One sample conc. = ND (or values reported as less than the QL) One sample conc. ≥ 2 X QL**
Detects	Professional Judgment	J
Non-detects	Professional Judgment	UJ

* QL = Sample Quantitation Limit

** RPD should not be evaluated for these duplicate pairs

Note: Qualification refers to field duplicate sample results only. Professional judgment may be used to apply field duplicate actions to all samples of the same matrix.

E. EXAMPLES

Example #1: (Both field duplicate sample concentrations ≥ 2X QL; %RPD > 50%; Acceptable laboratory precision)

Soil samples SAA11 and SAA12 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 89% and 85% solids, respectively. Sample SAA11 has a detected concentration of Aroclor-1254 of 100 ug/kg. Sample SAA12 has a detected concentration of Aroclor-1254 of 250 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 86%. The validator notes that both results are greater than twice the sample Quantitation Limit (QL). The QL for Aroclor-1254 in sample SAA11 is 37 ug/kg and the QL for Aroclor-1254 in SAA12 is 39 ug/kg. The validator reviews the MS/MSD data and determines that laboratory precision was acceptable. As a result, the validator estimates (J) the positive Aroclor-1254 detects in the field duplicate samples only, on the Data Summary Table, and notes the qualification and justification in the Data Validation Memorandum. The validator also notes that poor field precision may be due to a heterogenous matrix or a result of sampling error.

Analyte	SAA11		SAA12		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
Aroclor 1254	100	37	250	39	86

Example #2: (QL ≤ both field duplicate sample concentrations < 2X QL; RPD > 50%; Acceptable laboratory precision)

Soil samples SAA21 and SAA22 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 51% and 50% solids, respectively. Sample SAA21 has a detected concentration of alpha-Chlordane of 3.8 ug/kg. Sample SAA22 has a detected concentration of alpha-Chlordane of 6.5 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 52%. The sample QL for alpha-Chlordane in sample SAA21 is 3.3 ug/kg based on 51% solids sample and the sample QL for alpha-Chlordane in sample SAA22 is 3.4 ug/kg based on 50% solids. The validator reviews the MS/MSD results and determines that laboratory precision is acceptable. The validator notes that there were no blank actions applicable for alpha-Chlordane to the samples arising from blank contamination. The validator notes that both field duplicate results are between the sample QL and twice the sample QL. As a result the validator uses professional judgment to accept the alpha-Chlordane results in the field duplicate samples taking into consideration the increased variability of data near the quantitation limit. The validator notes in the Data Validation Memorandum that field duplicate precision was acceptable.

Analyte	SAA21		SAA22		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
alpha-chlordane	3.8	3.3	6.5	3.4	52

E. EXAMPLES

Example #3: (One sample concentration = ND; One sample concentration ≥ 2X QL; Acceptable laboratory precision)

Aqueous samples SAA31 and SAA32 are field duplicates, analyzed under CLP SOW OLM04.3. Sample SAA31 has a detected concentration of endrin ketone of 8.0 ug/L. Endrin ketone was not detected in sample SAA32. The validator notes that the positive endrin ketone detect in sample SAA31 is greater than twice the sample QL of 0.1 ug/L. The validator reviews the MS/MSD data and determines that laboratory precision was acceptable. The validator reviews the preceding blank and sample runs for potential contribution of endrin ketone to the sample, and determines that there was no apparent endrin ketone contamination. The validator estimates (J) the positive endrin ketone detect in sample SAA31 and estimates (UJ) the quantitation limit of the endrin ketone non-detect in sample SAA32 on the Data Summary Table based on poor field precision. The validator notes the qualification in the Data Validation Memorandum and also suggests that poor field precision may be due to sampling error.

Analyte	SAA31		SAA32		RPD
	Sample Conc. (ug/L)	Sample QL (ug/L)	Sample Conc. (ug/L)	Sample QL (ug/L)	
endrin ketone	8.0	0.1	ND	0.1	NA

Example #4: (One sample concentration = ND; One sample concentration < 2X QL; Acceptable laboratory precision)

Soil samples SAA41 and SAA42 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 90% and 85% solids, respectively. Sample SAA41 has a detected concentration of Aroclor-1260 of 65 ug/kg. Aroclor-1260 was not detected in sample SAA42. The validator notes that the positive Aroclor-1260 detect is between the sample QL and twice the sample QL. The sample QL for Aroclor-1260 in sample SAA41 is 37 ug/kg and the sample QL for Aroclor-1260 in sample is SAA42 is 39 ug/kg. The validator reviews the MS/MSD results and determines that RPD criteria were met for the pesticides indicating acceptable laboratory precision. The validator verifies the identification of the multi-component analyte in the samples and determines that the results were correct as reported. The validator reviews the preceding blank and sample runs for potential contribution of Aroclor-1260 to the sample, and determines that there was no apparent Aroclor-1260 contamination. As a result, the validator uses professional judgment to accept the positive Aroclor-1260 detect in SAA41 and to accept the Aroclor-1260 non-detect in sample SAA42, taking into consideration the increased variability of data near the quantitation limit. The validator reports the results on the Data Summary Table and notes this in the Data Validation Memorandum.

Analyte	SAA41		SAA42		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
Aroclor 1260	65	37	ND	39	NA

E. EXAMPLES

Example #5: (Both duplicate sample concentrations ≥ 2X QL; Poor field and laboratory precision)

Soil samples SAA34 and SAA35 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 90% and 95% solids, respectively. Sample SAA34 has a detected concentration of aldrin of 10 ug/kg. Sample SAA35 has a detected concentration of aldrin of 40 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 120%. The validator notes that both results are greater than twice the sample QL. The sample QL for aldrin in sample SAA34 is 1.9 ug/kg and the sample QL for aldrin in sample SAA35 is 1.8 ug/kg. The validator reviews the MS/MSD data for samples SAA34MS/MSD and determines that the RPD for aldrin equals 60% which is outside the criteria. The validator is unable to determine the source of the imprecision since both the lab and field precision were poor; therefore, the validator uses professional judgment to estimate (J) the aldrin positive detects in all samples associated with the sample delivery group and estimate (UJ) the quantitation limits for aldrin non-detects in all samples associated with the sample delivery group. The validator reports the qualified data on the Data Summary Table and justifies the qualification in the Data Validation Memorandum. The validator notes that the source of the imprecision cannot be determined.

Analyte	SAA34		SAA35		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
aldrin	10.0	1.9	40	1.8	120

X. SENSITIVITY CHECK

A. OBJECTIVE

Although most CLP SOWs do not incorporate the analysis of sensitivity checks, many EPA methods do require that a Method Detection Limit (MDL) study be performed prior to sample analysis and/or that a Laboratory Fortified Blank (LFB) be analyzed at the time of sample analysis. The MDL study generates statistically-based detection limits and can be used to assess method sensitivity, laboratory precision, and method bias for specific analytes within an analytical method on a specific instrument and column. An LFB, a type of Laboratory Control Sample, is a reagent blank spiked with several or all of the target analytes at or below their quantitation limits. LFB data can be used to assess laboratory sensitivity and bias for specific analytes at the quantitation limit within an analytical method on a specific instrument and column at the time of sample preparation and analysis. To determine sample qualification, the MDL study is evaluated prior to the LFB data.

Region I routinely uses MDL studies as a pre-qualification check to verify the laboratory's ability to meet the technical specification/method requirements prior to contract award and field sample receipt. Region I also routinely includes LFB analyses to document the method sensitivity and bias associated with the day-to-day preparation and analysis of field samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the Pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. Method Detection Limit (MDL) Study

a. The method detection limit (MDL) for each target analyte must be established in accordance with the specified method and the Code of Federal Regulations (40 CFR Part 136, App.B). A minimum of seven replicates must be analyzed for each matrix of interest.

b. Surrogates must be spiked into each MDL sample as specified in the method. Recoveries and %RSDs for surrogates and target analytes must meet the criteria specified in the method. If the method does not specify recovery and/or replicate %RSD criteria, then the %RSD for the seven replicates should be less than or equal to 25% and the mean recovery for target analytes and surrogates should be between 80-120%.

c. Samples must be analyzed on the same instrument under the same conditions as was used for the MDL study.

d. The MDL study must be performed within one year prior to the start of the preparation and/or analysis of the samples.

e. The MDL for each target analyte must be less than one half the target analyte method-required quantitation limit.

2. Laboratory Fortified Blank (LFB)

a. Verification of laboratory accuracy at the quantitation level requires the routine analysis of an LFB spiked with target analytes at the quantitation limit and surrogate analytes spiked at the concentrations specified in the method. The stock solution used for spiking the LFB must be prepared from a source other than the source used for preparing the initial and continuing calibration standards.

b. One LFB containing all the target analytes at the quantitation limit must be analyzed immediately prior to sample analysis but after instrument calibration. Subsequently, an LFB must be analyzed every 12 hours. One LFB must be extracted with each sample delivery group of pesticide/PCB samples, or whenever pesticide/PCB samples are extracted, whichever is more frequent.

c. Method QC acceptance criteria must be met for surrogates and target analytes. If the method does not specify recovery QC acceptance criteria for the LFB, then the recovery for target analytes should be between 60-140%. Surrogate analytes for the LFB must meet validation criteria as per Section VI of this document.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
Qualification of data should be based on a combined evaluation of both the MDL study and LFB results. To determine appropriate sample qualification, the MDL Study should be evaluated first and then the LFB results.	All potential impacts on the sample data resulting from MDL and/or LFB study anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Method Detection Limit (MDL) Study	**1. Method Detection Limit (MDL) Study**
a. Verify that the MDL study was generated in accordance with the method and 40 CFR Part 136 App. B, and that a minimum of seven replicates for each matrix of interest were prepared and analyzed.	a. If the required MDL study was not performed at all or was not performed according to the 40 CFR Part 136 App. B criteria, then the validator should evaluate the LFB data, if available, to determine the action to be taken. See Tables Pest/PCB-X-1, Pest/PCB-X-2, and Pest/PCB-X-3 for the appropriate action. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.

C. EVALUATION	D. ACTION
1. b. Verify that the %RSD for each target analyte and surrogate is less than or equal to 25% for all seven replicates of the MDL study.	1. b. If the MDL target analyte and surrogate%RSD criteria are exceeded, then the validator should evaluate initial calibration %RSDs to assess instrument precision and linearity. The validator should use professional judgment to assess the impact of laboratory precision on analytical sensitivity and data quality.
c. Compare all seven replicates of the MDL study to verify that the mean recovery for each target analyte and surrogate is within the 80-120% criteria for each replicate in the MDL Study.	c. If the mean percent recovery for a target analyte and/or surrogate is greater than 120%, then the validator should: - Use professional judgment to estimate (J) positive detects for that analyte in all samples associated with that MDL study, taking into consideration the LFB results. - Accept the non-detects. If the mean percent recovery for a target analyte and/or surrogate is less than 80% but greater than or equal to 10%, then the validator should: - Use professional judgment to estimate (J) positive detects for that analyte in all samples associated with that MDL study, taking into consideration the LFB results. - Use professional judgment to estimate (UJ) the non-detects for that analyte in all samples associated with that MDL study taking into consideration the LFB results. If the mean percent recovery for a target analyte and/or surrogate is less than 10%, then the validator should estimate (J) positive detects for that analyte and reject (R) the non-detects for that analyte in all samples associated with that MDL study.

C.	EVALUATION	D.	ACTION
*1. d.	Check and recalculate the %RSDs and % recoveries for at least three analytes per MDL study. Verify that the recalculated values agree within ± 10% of the reported results.	1. d.	If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
e.	Verify that the samples were analyzed on the same instruments and under the same conditions as was used for the MDL study.	e.	If the samples were not analyzed on the same instruments or under the same conditions as the MDL study, then the validator should contact the laboratory to obtain a correct MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.
f.	Verify that the matrix for the MDL Study is the same as that of the samples.	f.	If the MDL Study is not the same matrix as the samples, then the validator should contact the laboratory to obtain a correct MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.

C. EVALUATION	D. ACTION
1. g. Compare the date of the MDL study to the dates of all associated sample analyses to verify that the MDL study was performed within one year prior to the start of the first sample prepared and/or analyzed in the sample delivery group.	1. g. If the MDL study was not submitted or was not performed within one year of the start of preparation and/or analysis of the first sample in the SDG, then the validator should contact the laboratory to obtain a current MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should evaluate the lowest standard of the initial calibration and the daily continuing calibration standard data and use professional judgment to assess the impact of analytical sensitivity on data quality.
h. Verify that all MDLs are not more than one half of the method-required quantitation limits.	h. If the MDL study reveals that a target analyte has a detection limit greater than one half the method-required quantitation limit, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should: i. Elevate the quantitation limit for that target analyte in all samples associated with that MDL study to the lowest concentration calibration standard analyzed or to the laboratory reported MDL, whichever is higher. ii. Estimate (J) positive detects which were below the elevated quantitation limit for that target analyte and/or in all samples associated with that MDL study. iii. The validator should evaluate the elevated quantitation limits in relation to the required quantitation limits in the site DQO's. The validator should discuss the impact of the elevated quantitation limits on the site objectives and whether or not the data are usable for the site objectives in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
1. i. Check and recalculate the MDL value for at least one analyte per MDL Study. Verify that the recalculated values agree within ± 10% of the reported results.	1. i. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
If the LFB criteria are not met, then laboratory performance related to method bias and method/instrument sensitivity is questionable. **2. Laboratory Fortified Blank (LFB)** * a. Check the standards preparation logs to verify that the stock standard used to prepare the LFB was from a source independent from the initial and continuing calibration standards. b. Verify that an LFB was prepared and/or analyzed at the proper frequency and that it was spiked with the correct analytes at their quantitation limits. c. Verify that the reported recoveries for all LFB spike analytes are within the method QC acceptance criteria.	**2. Laboratory Fortified Blank (LFB)** a. If the LFB was not prepared from a source independent from the initial and continuing calibration standards, then the laboratory performance related to method bias and method/instrument sensitivity is questionable. The validator should review other calibration verification checks, i.e., Performance Evaluation Sample (PES) analyses to ensure the calibration accuracy. Professional judgment should be used to qualify sample quantitation limits. b. If an LFB analysis was not performed or the LFB was not analyzed for the correct analytes at the proper frequency and concentration, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality. c. Sample data should be qualified based on the number and type of analytes that are recovered outside the method QC acceptance criteria and based on the degree that analyte recoveries exceed the criteria.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. i. If any of the LFB analyte recoveries are outside the method QC acceptance criteria, then the LFB results should be used to qualify sample data for the specific analytes that are included in the LFB solution. The validator should use professional judgment to qualify sample data for analytes not included in the LFB,taking into account the analyte's chemical class, analyte recovery efficiency, and any analytical problems historically associated with the analyte or that were encountered by the laboratory. ii. If an LFB analyte recovery is greater than 140%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LFB to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that LFB. iii. If more than half of the LFB analyte recoveries are greater than 140%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LFB to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that LFB.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. iv. If an LFB analyte recovery is less than 60% but greater than or equal to 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LFB to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that LFB to indicate potential low bias. v. If more than half of the LFB analyte recoveries are less than 60% but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LFB to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that LFB to indicate potential low bias. vi. If an LFB analyte recovery is less than 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LFB to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. vii. If more than half of the LFB analyte recoveries less than 10%, then the validator should: - Estimate (J) all positive detects in all samples associated with that LFB to indicate potential low bias. - Reject (R) the quantitation limits for all non-detects in all samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives. viii. If more than half of the LFB analyte recoveries are outside the method QC acceptance limits in one LFB with some low recoveries and some high recoveries, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes or the entire fraction for samples associated with that LFB. ix. Action on non-compliant surrogate recoveries should follow the guidance provided in Section VI. Professional judgment should be used to evaluate the impact that a non-compliant LFB surrogate recovery has on the sample data.

C. EVALUATION	D. ACTION
*2. d. Check and recalculate the % recovery for at least one target analyte per LFB. Verify that the recalculated value agrees within ± 10% of the reported result.	2 d. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.d, C.2.a, C.2.d

Table Pest/PCB-X-1:

QUALIFICATION OF ORGANIC ANALYTES BASED ON MDL STUDY RESULTS

Sample Results	Mean % Recovery			
	%Rec < 10%	10% ≤ %Rec < 80%	80% ≤ %Rec ≤ 120%	%Rec > 120%
Detects	J	Professional Judgment*	A	Professional Judgment*
Non-Detects	R	Professional Judgment*	A	A

Sample Results	% RSD	
	> 25%	≤ 25%
Detects	Professional Judgment**	A
Non-detects	Professional Judgment**	A

* Taking into consideration LFB results.
** Taking into consideration initial calibration %RSDs.

Table Pest/PCB-X-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE:
≤ ONE-HALF OF LFB ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target analytes and/or Aroclors at or below the quantitation limit.

Table Pest/PCB-X-3:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE:
> ONE-HALF OF LFB ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target analytes and/or Aroclors at or below the quantitation limit.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

E. EXAMPLES

Example #1: (Low LFB recoveries for several analytes)

Low concentration water samples were analyzed under CLP SOW OLC03.2, however, no MDL study data was available. LFB analytes dieldrin, endrin, and endosulfan sulfate recovered below QC acceptance criteria but greater than 10%, i.e., 25%, 30%, and 18%, respectively. The validator estimates (J) the positive detects for dieldrin, endrin, and endosulfan sulfate in all the field samples associated with the LFB to indicate potential low bias and estimates (UJ) the quantitation limits for the dieldrin, endrin, and endosulfan sulfate non-detects in all the field samples associated with the LFB to indicate a decrease in sensitivity and the possibility of false negatives. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #2: (High LFB recoveries for more than 50% of pesticide analytes)

Low concentration water samples were analyzed under CLP SOW OLC03.2, however no MDL study data was available. LFB analytes gamma-chlordane, endrin, dieldrin, and heptachlor epoxide recovered above the QC acceptance criteria, i.e., 150%, 160%, 135%, and 173%, respectively. The validator notes that more than 50% of the LFB analytes exceeded the QC criteria. Therefore, the validator estimates (J) all positive detects in all samples associated with the LFB and all non-detects were accepted. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #3: (Low MDL recoveries for non-LFB analytes; Acceptable LFB results)

The analytical method used did not specify QC acceptance criteria for the MDL study. The validator uses the default criteria for mean % recoveries (80-120%) to evaluate the MDL data. The MDL study submitted by the laboratory did not meet the default criteria for mean % recovery for 4,4'-DDD and methoxychlor (45% and 9%, respectively). The validator examines the LFB results submitted with the sample results and determines that QC acceptance criteria were met for all LFB analytes. However, the validator notes that 4,4'-DDD and methoxychlor were not LFB analytes. The validator uses professional judgment to estimate (J) the positive 4,4'-DDD detects, estimate (UJ) the 4,4'-DDD non-detects, estimate (J) the positive methoxychlor detects, and reject the methoxychlor non-detects. The validator reports the qualified results on the Data Summary Table and notes the sample qualifications in the Data Validation Memorandum.

Example #4: (High LFB recovery for two analytes; High MDL %RSDs for two analytes)

The analytical method used for sample analysis did not specify QC acceptance criteria for the MDL study. The validator uses the default criteria for mean % recoveries (80-120%) and % RSDs to evaluate the MDL data. The MDL study submitted by the laboratory did not meet default (25%) %RSD criteria for endrin and dieldrin (32% and 29%, respectively). The validator reviews the initial calibration %RSDs and determines that endrin and dieldrin met the initial calibration %RSD acceptance criteria. In addition, the analytical method used did not specify QC acceptance criteria for the LFB. The validator uses the default recovery criteria of 60-140% to evaluate LFB results. The validator examines the LFB submitted with the analytical results and determines that dieldrin and endrin also exceeded the LFB % recovery criteria of 140% (159% and 160%, respectively). Since the initial calibration %RSDs were acceptable, the high MDL %RSDs were not utilized to qualify sample data. Based upon the LFB recoveries, the validator uses professional judgment to estimate (J) the positive dieldrin and endrin detects to indicate potential high bias for these two analytes and accept the quantitation limits for dieldrin and endrin non-detects in all field samples

associated with the LFB. The validator reports the qualified results on the Data Summary Table and notes the sample qualifications in the Data Validation Memorandum.

XI. PERFORMANCE EVALUATION SAMPLES/ACCURACY CHECK

A. OBJECTIVE

Performance Evaluation Samples (PESs) are analyzed to provide information on the overall accuracy and bias of the analytical method and on laboratory performance. PESs are evaluated for false negatives, false positives, and inaccurate target analyte quantitation. In general, the most serious problem a PES can expose is the failure of the laboratory to properly detect and identify a PES analyte. This failure is known as a false negative. False negatives significantly increase the "uncertainty" of any site decisions concerning the "cleanliness" or contamination level of a site. Also, the analysis of PE samples can reveal the laboratory's erroneous detection of unspiked analytes (false positives). False positives should always be evaluated in conjunction with blank data to determine the probable source(s) of contamination.

Finally, the PES provides information on the magnitude and direction of quantitative bias for the entire laboratory method, including sample preparation (extraction and cleanup) and analysis (chromatography and calibration). Sample data that are biased high or low can potentially impact site decisions, especially when sample data have target analyte concentrations at or near action levels.

Ideally, a PES is comprised of the same matrix as the field samples being evaluated. However, for some matrices (i.e., soil) PESs are not available. In these situations, a PES of another matrix (i.e., water) may be analyzed with the field samples to assess laboratory performance on the "analysis" portion, even though laboratory performance on the "sample preparation" portion cannot be assessed. The validator should use professional judgment when evaluating samples of one matrix using PES data from another matrix.

B. CRITERIA

1. Zero Blind Performance Evaluation Samples

A Zero Blind PES is a quality control sample when the composition and concentration are known to the laboratory.

A Laboratory Control Sample (LCS) is a Zero Blind PES sample which is often used by the laboratory as an internal quality control check of analytical accuracy and method bias.

a. An LCS is required by some EPA methods and certain CLP SOWs. The frequency, concentration, acceptance criteria, and corrective actions for LCS analysis should be stated in the method, Sampling and Analysis Plan (SAP), or the Quality Assurance Project Plan (QAPP) and should support the DQOs of the project. The LCS should be prepared in the proper matrix for each parameter at the concentration level and frequency required in the EPA-approved project SAP, QAPP, and/or method. The LCS must contain one or more target analytes. The LCS must be prepared and analyzed concurrently with field samples contained in the sample delivery group (SDG).

b. The percent recoveries for LCS analytes must be within the method QC acceptance criteria.

c. Surrogate analytes for the LCS must meet validation criteria as per Section VI of this document.

2. Single Blind Performance Evaluation Samples

A Single Blind PES is a quality control sample whose composition and concentration is not known to the laboratory, but the sample is identified to the laboratory as a PES.

A Single Blind PES may be submitted with a sample delivery group to assess method bias, laboratory performance and to evaluate data quality. A Single Blind PES may also be submitted for analysis prior to sample shipment to prequalify a laboratory for a specific matrix and/or parameter.

a. EPA Region I Performance Evaluation Program Guidance, July 1996, Revision, requires that a Single Blind or Double Blind PES be sent with each sample delivery group (20 samples or less) that is sent to a laboratory. A PES is required for each matrix, parameter, and concentration level unless an EPA and/or non-EPA PES does not currently exist for that particular matrix, parameter, and concentration level.

The PE Program applies to the Superfund program including EPA Fund-lead and PRP/Federal Facility Oversight Projects. In addition, the PE Program applies to Fund-lead projects performed by States under Cooperative Agreements and other Federal Agencies under Interagency Agreements. The PE Program also applies to Non-Fund-lead Superfund projects undertaken by potentially responsible parties. The PE Program also applies to Non-Superfund Programs.

EPA-provided PE samples are available for certain categories of Superfund work as specified in the EPA Region I Performance Evaluation Program Guidance, July 1996, Revision. The EPA Performance Evaluation Chemist provides the current list of EPA-provided PE samples upon request. For those categories of Superfund work that do not have access to EPA-provided PE samples and for all Non-Superfund program work scientifically defensible PE samples should be obtained from commercial vendors.

b. Acceptance criteria for EPA PESs are statistically-derived by the Analytical Operations Center under the QATS contract. Tabulated report forms for EPA PESs must be submitted to the Region I OEME-QA Unit for scoring at the time of data validation, in accordance with the EPA Region I Performance Evaluation Program Guidance, July 1996, Revision. This guidance is found in Attachment H of the Part I - Data Validation Manual: The Data Quality System.

c. True values and QC acceptance criteria for all non-EPA PESs should be provided by the manufacturer and these acceptance criteria must be fully documented and must be scientifically defensible.

d. Surrogate analytes for EPA and non-EPA Single Blind PE samples must meet validation criteria as per Section VI of this document.

3. Double Blind Performance Evaluation Samples

A Double Blind PES is a quality control sample whose composition and concentration is not known to the laboratory and the sample is **not** identifiable as a PES nor is it identified to the laboratory as a PES.

A Double Blind PES may be submitted with a sample delivery group in lieu of a Single Blind PES, to assess method bias, laboratory performance and to evaluate data quality.

a. The use of Double Blind PESs is dictated by the project DQOs and should be documented in the EPA-approved SAP and/or QAPP.

b. True values and acceptance criteria for Double Blind PESs must be fully documented and must be scientifically defensible.

c. Surrogate analytes for EPA and non-EPA Double Blind PE samples must meet validation criteria as per Section VI of this document.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from performance evaluation sample anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Zero Blind PES - LCS	**1. Zero Blind PES - LCS**
a. Verify that an appropriate LCS sample (correct parameter, concentration level, target analytes, and matrix) was prepared and analyzed at the required frequency for each sample delivery group in accordance with the EPA approved project SAP, QAPP, and/or method.	a. If an appropriate LCS was not analyzed at the required frequency for the correct parameters, concentration levels, target analytes, or matrices, then the validator should use professional judgment to determine if the sample data should be qualified or rejected.
b. Verify that the required LCS results are provided for each sample delivery group.	b. If the required LCS results were not submitted for each sample delivery group, then the validator should contact the laboratory to obtain raw data and/or tabulated results.

C. EVALUATION	D. ACTION
1. c. Verify that the reported recoveries for all LCS spike analytes are within the method QC acceptance criteria.	1. c. Sample data should be qualified based on the number and type of analytes that recover outside the method QC acceptance criteria and based on the degree that analyte recoveries exceed the criteria. i. If any of the LCS analyte recoveries are outside the method QC acceptance criteria, then the LCS results should be used to qualify sample data for the specific analytes that are included in the LCS solution. Professional judgment should be used to qualify sample data for non-LCS analytes, taking into account the analyte's chemical class, analyte recovery efficiency, and any analytical problems historically associated with the analyte or that were encountered by the laboratory. ii. If an LCS analyte recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LCS to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that LCS. iii. If more than half of the LCS analyte recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LCS to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that LCS.

C. EVALUATION	D. ACTION
1. c. Continued from above.	1. c. iv. If an LCS analyte recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LCS to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that LCS to indicate potential low bias. v. If more than half of the LCS analyte recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LCS to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that LCS to indicate potential low bias. vi. If an LCS analyte recovery is less than 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LCS to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any samples associated with that LCS to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
1. c. Continued from above	1. c. vii. If more than half of the LCS analyte recoveries are less than 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LCS to indicate potential low bias. - Reject (R) quantitation limits for <u>all</u> non-detects in all samples associated with that LCS to indicate that the data are unusable due to the possibility of false negatives. viii. If more than half of the LCS analyte recoveries are outside the method QC acceptance limits in one LCS, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes, or the entire fraction for samples associated with that LCS. ix. Based upon the number and type of analytes misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied. See Attachment I of Part I - Data Validation Manual: The Data Quality System.
d. Evaluate the surrogate analytes for the LCS.	d. Action on non-compliant surrogate recoveries should follow the guidance provided in Section VI. Professional judgment should be used to evaluate the impact that non-compliant LCS surrogate recoveries have on the sample data.

C. EVALUATION	D. ACTION
*1. e. Check and recalculate the percent recovery for at least one analyte per LCS fraction. Verify that the recalculated value agrees within ±10% of the reported result.	1. e. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Single Blind <u>and</u> Double Blind PESs a. Verify that an appropriate Single Blind or Double Blind PES (correct parameter, concentration level, target analytes, and matrix) was analyzed at the required frequency for each sample delivery group in accordance with Region I PE policy and/or the EPA approved SAP and/or QAPP. b. Verify that Single Blind PES results are provided for each sample delivery group in accordance with Region I PE policy. The PE policy is found in Attachment H of Part I - Data Validation Manual: The Data Quality System. c. **EPA PESs** If the PES was supplied and scored by Region I OEME-QA, then the Region I PES Score Report must be evaluated to determine how many of the analytes met or exceeded PES acceptance criteria.	**2. Single Blind <u>and</u> Double Blind PESs** a. If a required Single Blind or Double Blind PES was not analyzed at the required frequency for the correct parameters, concentration levels, target analytes, or matrices, then the validator should use professional judgment to determine if the sample data should be qualified or rejected. b. If the PES results were not submitted for each sample delivery group, then the validator should contact the laboratory to obtain raw data and/or tabulated results. If a PES was not submitted to the laboratory by the sampler, then the validator should contact the sampler to confirm the PES omission and document that fact on the worksheet and discuss the impact of the omission on the results in the Data Validation Memorandum. c. **EPA PESs** Note: PES results should not be qualified based on QC sample data and should not be reported on the Data Summary Table. Rather, PES results should be discussed in the Data Validation Memorandum or Tier I Validation Cover Letter and PES Score Reports should be attached as supporting documentation.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. Continued from above.
• Evaluate the "PES ANALYTES MISSED" to assess the potential for low bias and false negative sample results.	• Sample data should be qualified based on the number and type of "PES ANALYTES MISSED" identified on the PES Score Report. i. If a PES analyte is not identified in the PES, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives. ii. Based upon the chemical class, the number of analytes that were not identified, and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied. (See Attachment I of Part I - Data Validation Manual: The Data Quality System.)
• Evaluate "PES CONTAMINANTS" in conjunction with blank data to asses the potential for high bias and false positive sample results.	• Sample data should not be qualified based on the number and type of "PES CONTAMINANTS" identified on the PES Score Report alone. i. If a TCL contaminant is detected in the PES and is also found in a blank, then the validator should evaluate and qualify sample data based upon blank contamination in accordance with Section V. ii. If a TCL contaminant is detected in the PES but is not present in any blank, then that interference is specific to the PES and does not impact sample data.

C. EVALUATION	D. ACTION
2. c. Continued from above. ● Evaluate "PES ANALYTES REPORTED" that were misquantified to assess the potential for high and/or low bias in sample data.	2. c. Continued from above. ● Sample data should be qualified based on the number and type of misquantified (Action High/Action Low "PES ANALYTES REPORTED") identified on the PES Score Report. **Sample data should not be qualified based on "Warning Low/Warning High" scores for "PES ANALYTES REPORTED".** i. If any of the PES analytes do not meet PES acceptance criteria, then the PES results should be used to qualify sample data for the specific analytes that are included in the PES sample. Professional judgment should be used to qualify sample data for non-PES analytes taking into account the analyte's chemical class, analyte recovery efficiency, and analytical problems historically associated with the analyte or that were encountered by the laboratory. ii. If a PES analyte is scored in the "Action High" category, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that PES to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that PES. iii. If more than half of the PES analytes are scored in the "Action High" category, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that PES.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. iv. If a PES analyte is scored in the "Action Low" category, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that PES to indicate that data are unusable due to the possibility of false negatives. v. If more than half of the PES analytes are scored in the "Action Low" category, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential low bias. - Reject (R) <u>all</u> quantitation limits for non-detects in all samples associated with that PES to indicate that data are unusable due to the possibility of false negatives. vi. If more than half of the PES analytes are scored in the "Action" levels in one PES, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes, or the entire fraction for samples associated with that PES. vii. Based upon the number and type of analytes misquantified, and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied. (See Attachment I of Part I - Data Validation Manual: The Data Quality System.)

C. EVALUATION	D. ACTION
2. c. Continued from above. ● Evaluate the surrogate analytes for the EPA PES.	2. c. Continued from above. ● Action on non-compliant surrogate recoveries should follow the guidance provided in Section VI. Professional judgment should be used to evaluate the impact that a non-compliant EPA PES surrogate recoveries would have on the sample data.
d. **Non-EPA PESs** If the PES was obtained from a source other than Region I OEME-QA, then the validator should use the vendor's criteria to evaluate the PES results. Confirm that PES acceptance criteria are fully documented and scientifically defensible. ● Evaluate the "PES ANALYTES MISSED" to assess the potential for low bias and false negative sample results.	d. **Non-EPA PESs** If the non-EPA PES acceptance criteria are not fully documented and/or scientifically defensible, then the validator should use professional judgment to qualify or reject the sample data. ● Sample data should be qualified based on the number and type of "PES ANALYTES MISSED" identified from the vendor's acceptance criteria. i. If a PES analyte is not identified in the PES, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives. ii. Based upon the chemical class, the number of analytes that were not identified, and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied. (See Attachment I of Part I - Data Validation Manual: The Data Quality System.)

C. EVALUATION	D. ACTION
2. d. Continued from above. ● Evaluate "PES CONTAMINANTS" in conjunction with blank data to assess the potential for high bias and false positive sample results.	2. d. Continued from above. ● Sample data should not be qualified based on the number and type of "PES CONTAMINANTS" identified from the vendor's acceptance criteria alone. i. If PES CONTAMINANTS are detected in the PES and is also found in a blank, then the validator should evaluate and qualify sample data based upon blank contamination in accordance with Section V. ii. If PES CONTAMINANTS are detected in the PES but is not present in any blank, then that interference is specific to the PES and does not impact sample data.
● Evaluate "PES ANALYTES REPORTED" that were misquantified to assess the potential for high and/or low bias in sample results.	● Sample data should be qualified based on the number and type of misquantified "PES ANALYTES REPORTED" identified from the vendor's acceptance criteria. i. If any of the PES analytes do not meet acceptance criteria, then the validator should use the PES results to qualify sample data for the specific analytes that are included in the PES sample. Professional judgment should be used to qualify sample data for non-PES analytes, taking into account the analyte's chemical class, analyte recovery efficiency, and analytical problems associated with the analyte either historically or that were encountered by the laboratory.

C. EVALUATION	D. ACTION
2. d. Continued from above.	2. d. Continued from above. ii. If a PES analyte recovery is outside the upper limit of the vendor's documented acceptance limits, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that PES to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that PES. (Note: The validator should confirm that the vendor's acceptance limits are calculated as plus and minus three standard deviations from the mean, similar to EPA-PES "Action Limits".) iii. If more than half of the PES analyte recoveries are outside the upper limit of the vendor's documented acceptance limits (See note above, Section 2.d.ii), then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that PES.

C. EVALUATION	D. ACTION
2. d. Continued from above.	2. d. Continued from above. iv. If a PES analyte recovery is outside the lower limit of the vendor's documented acceptance limits (See note above, Section 2.d.ii), then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that PES to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that PES to indicate that the data are unusable due to the possibility of false negatives. v. If more than half of the PES analyte recoveries are outside the lower limit of the vendor's documented acceptance limits (See note above, Section 2.d.ii), then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that PES to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. d. Continued from above.	2. d. Continued from above vi. If more than half of the PES analyte recoveries are outside the vendor's documented acceptance limits in one PES (See note above, Section 2.d.ii), where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes or the entire fraction for samples associated with that PES. vii. Based upon the number and type of analytes misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied. (See Attachment I of Part I - Data Validation Manual: The Data Quality System.)
● Evaluate surrogate analytes for the non-EPA PES.	● Action on non-compliant surrogate recoveries should follow the guidance provided in Section VI. Professional judgment should be used to evaluate the impact that non-compliant non-EPA PES surrogate recoveries would have on the sample data.
* e. Determine what percentage of PES analytes were below or above PES acceptance criteria.	e. If more than half of the PES analytes are high or low, then the validator should check the raw data and/or contact the laboratory to verify that the PE sample was prepared according to the PE instructions (if applicable). Check also that the appropriate PE instructions (for the PE concentration) were sent to the laboratory.

C. EVALUATION	D. ACTION
*2 f. Check and recalculate the analytical concentrations of at least one analyte per PES fraction. Verify that the recalculated value agrees within ± 10% of the reported result.	2. f. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum. i. If corrected data reports affect the original results reported on the initial EPA PES score report, then the validator should resubmit the corrected PES results to Region I OEME-QA for a PES rescore. Sample data should be reevaluated and requalified based on the corrected PES data. ii. If corrected data reports affect the original results reported for the initial non-EPA PES, then the validator should reevaluate and requalify sample data based on the corrected PES data.

* **Note:** **The following subsections are applicable only to a Tier III data validation:**

C.1.e, C.2.e, C.2.f

Table Pest/PCB-XI-1:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON LCS* RECOVERIES WHERE: ≤ ONE-HALF OF LCS ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LCS = Laboratory Control Sample (LCS) is an internal laboratory quality control sample designed to assess analytical accuracy and method bias. The LCS is spiked with several or all of the method target analytes and/or Aroclors.

LL - Lower limit of method QC acceptance criteria
UL - Upper limit of method QC acceptance criteria

Table Pest/PCB-XI-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LCS* RECOVERIES WHERE: > ONE-HALF OF LCS ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* LCS = Laboratory Control Sample (LCS) is an internal laboratory quality control sample designed to assess analytical accuracy and method bias. The LCS is spiked with several or all of the method target analytes and/or Aroclors.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower limit of method QC acceptance criteria
UL - Upper limit of method QC acceptance criteria

Table Pest/PCB-XI-3:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON PES RESULTS WHERE: ≤ ONE-HALF OF PES ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	●Single Blind ●Double Blind PES < Lower Limit "Action Low"	●Single Blind ●Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	●Single Blind ●Double Blind PES > Upper Limit "Action High"
Detects	J	A	J
Non-Detects	R	A	A

Table Pest/PCB-XI-4:

QUALIFICATION OF ORGANIC ANALYTES BASED ON PES RESULTS WHERE: > ONE-HALF OF PES ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS *

Sample Results	●Single Blind ●Double Blind PES < Lower Limit "Action Low"	●Single Blind ●Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	●Single Blind ●Double Blind PES > Upper Limit "Action High"
All Detects	J	A	J
All Non-Detects	R	A	A

* Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

E. EXAMPLES

Example #1: (One LCS analyte < lower limit; One LCS analyte > upper limit)

A Laboratory Control Sample (LCS) containing six pesticides spiked at three times the quantitation limit is found to have 4,4'-DDT with a % recovery of 45% and 4,4'-DDE with a % recovery of 165%. Less than one-half of the spiked LCS analytes were outside the LCS acceptance criteria (60-140%). The validator notes that the PEM preceding the LCS analysis had a DDT breakdown of 35.0%. The validator estimates (J) positive detects for 4,4'-DDT and 4,4'-DDE in all field samples associated with that LCS. The validator accepts the 4,4'-DDE non-detects and estimates (UJ) the 4,4'-DDT non-detects in all field samples associated with that LCS. The validator reports qualified data on the Data Summary Table and notes in the Data Validation Memorandum that DDT results are biased low and DDE results are biased high in all field samples associated with that LCS.

Example #2: (One Single Blind PES analyte < lower PES acceptance limit)

A Single Blind Performance Evaluation Sample (PES) is found to have an endrin ketone result that scored below the PES acceptance criteria. The validator determines that less than one-half of the spike PES analytes are outside the PES acceptance criteria. Therefore, the validator estimates (J) positive endrin ketone detects and rejects (R) the quantitation limits for endrin ketone non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and notes the positive endrin ketone detects are biased low and the non-detects are rejected due to the possibility of false negatives in the Data Validation Memorandum.

Example #3: (More than one-half of the PES analytes greater than the upper acceptance limits)

A Single Blind PES is found to have more than one-half of the spike pesticide PES analytes with % recoveries above the upper acceptance limits. The validator estimates (J) all positive detects in all field samples associated with that PES and accepts (A) all the quantitation limits for non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and notes the positive pesticide results are biased high in the Data Validation Memorandum.

Example #4: (More than one-half of PES analytes "Action High" or "Action Low")

A Single Blind PES is found to have more than one-half of the spike pesticide PES analytes with results that do not meet PES acceptance criteria. Some of the PES analytes are flagged "Action Low" and some flagged "Action High". The site DQOs are to determine whether or not cleanup levels were achieved. The validator determines that analytical error yields uncertainty in quantitative accuracy which may adversely affect site decisions. Therefore, the validator uses professional judgment to estimate (J) all positive detects in all field samples associated with that PES and reject (R) all quantitation limits in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and discusses the limited use of the data in the Data Validation Memorandum.

Example #5: (One "PES ANALYTES MISSED")

A Single Blind PES is found to have one "PES ANALYTES MISSED" for aldrin which is a contaminant of concern at the site. The validator estimates (J) all positive aldrin detects and rejects (R) all aldrin quantitation limits in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

E. EXAMPLES

Example #6: (One "PES CONTAMINANTS", also in blank)

A Single Blind PES is found to have one "PES CONTAMINANTS", gamma-BHC, at 1.2 ppb. The method blank contained 0.20 ppb of gamma-BHC, resulting in a Blank Action Level of 1.0 ppb. The validator uses the gamma-BHC Blank Action Level to evaluate the sample data and reports qualified data on the Data Summary Table. The validator suspects that the gamma-BHC false positive PES analyte is a result of laboratory contamination and discusses this in the Data Validation Memorandum. PES results are not reported on the Data Summary Table.

Example #7: (One "PES CONTAMINANTS", not in blank)

A Single Blind PES is found to have one "PES CONTAMINANTS", 4,4'-DDE, which is not detected in any of the blanks but is detected in two samples. The validator determines that the 4,4'-DDE is an interference specific to the PES because it was not detected in any of the method blanks or instrument blanks. The validator notes that 4,4'-DDD and 4,4'-DDT are present in each of the two samples in addition to 4,4'-DDE, therefore, the validator uses professional judgment to accept the positive 4,4'-DDE detects in the field samples and reports the data unqualified on the Data Summary Table and discusses this in the Data Validation Memorandum.

XII. TARGET ANALYTE IDENTIFICATION

A. OBJECTIVE

Qualitative criteria for analyte identification have been established to minimize the number of erroneous analyte identifications. An erroneous identification can be either a false positive (reporting an analyte that it is not present) or a false negative (not reporting an analyte that is present).

The identification criteria can be applied more easily in detecting false positives than false negatives (non-detects) since each positively identified analyte must be labeled on the chromatogram or on the printout of retention times from the data system. False negatives represent an absence of data and therefore, are more difficult to assess.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. **Retention Times:** Initially, a target analyte is identified when its retention time falls within the established retention time window for that analyte on a particular column. Analyte identification is confirmed when the sample is analyzed on a dissimilar GC column and the analyte falls within the retention time window established for that analyte on the second column. Occasionally, when a sample matrix interferes with positive analyte identification, or analyte coelution occurs on one or both columns, a third alternate GC column is used to provide necessary confirmation data.

    a. Retention times of all surrogates and target analytes reported in the samples must fall within the standard retention time windows established for all GC columns used for sample analyses.

2. **Analyte Chromatographic Criteria:** Analyte chromatographic criteria are critical to ensure that coelution does not interfere with analyte identification and quantitation.

    a. Target analytes should display symmetrical peak shapes.

    b. Target analytes should be adequately resolved. Analyte resolution should be evaluated using professional judgement.

    c. Target analyte peaks should return to baseline after elution.

    d. For multicomponent analytes: When more than one multicomponent analyte is observed in a sample, none of the peaks chosen to quantitate either analyte should be peaks common to both analytes.

3. **Chromatographic Plotting Criteria:** Criteria for chromatographic plotting are critical to ensure that chromatographic peaks can be visually and electronically evaluated to correctly identify and accurately quantitate target analytes.

a. The same scaling factor must be used for the field samples and the calibration standards. It is critical that the chromatograms for the initial calibration low point standard and the field sample with no target analytes reported can be compared by using the same scaling factor.

b. Chromatograms must display single component pesticides detected in the sample at less than full scale, and the largest peak of any multicomponent analyte detected in the sample at less than full scale.

c. If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale, and multicomponent analytes between 25 and 100 percent of full scale.

d. For any sample, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC.

e. For any sample, the baseline of the chromatogram must return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

f. If a chromatogram is replotted electronically to meet any of the above requirements, the scaling factor must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

4. **GC/MS Confirmation:** GC/MS confirmation is required if the concentration of a target analyte exceeds 10 ng/uL in the final pesticide/PCB sample extract. **Note: A standard GC/quadrupole mass spectrometer can achieve 10 ng/uL single component pesticide confirmation.** Alternate GC/MS technology may achieve analyte identifications at less than 10 ng/uL. Mass spectra of the analyte in the sample and a current laboratory generated reference standard (10 ng/uL single component pesticides, 50 ng/uL Aroclors, and 125 ng/uL Toxaphene) must match according to the following criteria:

a. All ions present in the standard mass spectrum at a relative intensity greater than 10 percent <u>must</u> be present in the sample spectrum.

b. The relative intensities of these ions must agree within ± 20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance must be between 30 percent and 70 percent.)

c. Ions greater than 10 percent of the <u>sample</u> mass spectrum but not present in the <u>standard</u> spectrum must be considered and accounted for.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from target analyte identification anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Compare Form Is and the Pesticide/PCB Identification Summary (Form X, PEST-1, and PEST-2) and verify that reported retention times for all positive detects are within the calculated retention time windows reported on Pesticide Initial Calibration Forms (Form VI PEST-1 and -3).	1. a. If sample results have been reported on the Form Is for target analytes (single or multicomponent) with retention times which are not within the calculated retention time windows, then the validator should review the raw data to determine whether or not the laboratory has made any analytical and/or transcription errors. The validator should have the laboratory resubmit all corrected forms.
* b. Verify that all analytes reported on Forms I and X display chromatographic peaks within the calculated retention time windows on two or more analytical columns.	b. If the retention time criteria for two (or more) column confirmations are not achieved, then affected target analytes that are reported as positive detects should be considered non-detects. The reported value is considered a false positive and it should be replaced on the Data Summary Table with the target pesticide/PCB sample quantitation limit. The validator should have the laboratory resubmit all corrected forms and raw data if necessary. For multicomponent analytes, if the retention time criteria for one or more peaks chosen for quantitation are not met, then the validator should review the raw data to determine whether or not the laboratory has made any analytical, identification, and/or transcription errors. The validator should have the laboratory resubmit all corrected forms and raw data if necessary.

C. EVALUATION	D. ACTION
*1. c. Verify that all analytes reported on Form Is as "not detected" show no chromatographic peaks within the calculated retention time windows on two or more analytical columns. Review the sample chromatograms to determine whether or not false negatives have been reported.	1. c. If interferences obscure a target analyte's retention time window, then the validator should use professional judgment to elevate the target analyte's quantitation limit to the concentration of the interferent or reject (R) the sample data, in accordance with D.2.a.i. All technical decisions should be justified and documented in the Data Validation Memorandum. A peak that meets retention time criteria on both analytical columns but was reported as a non-detect may be a false negative. The validator should review the relevant sample chromatograms for false negatives. If false negatives are found, the laboratory should be contacted and asked to re-evaluate the false negative peak and report it, if necessary. The validator must evaluate whether significant matrix interference and/or coelution exists, and evaluate retention time data in conjunction with the sample chromatograms. The validator should use professional judgment when deciding if an analyte should be reported. All conclusions made regarding target analyte identification should be documented and justified in the Data Validation Memorandum.
* d. While reviewing the raw data, the validator should check for analyte retention time shifts which could lead to the reporting of false positives and/or false negatives.	d. If analyte retention times have shifted, then the validator should follow the chromatographic and resolution check guidance in Section II C and D 2.a-c. to ensure that false negatives and/or false positives have not been reported.

C. EVALUATION	D. ACTION
After a validator has confirmed that all reported pesticides/PCBs meet analyte retention time criteria, one must next confirm that the chromatographic criteria are achieved in order to ensure positive analyte identification.	
*2. a. Confirm that analyte chromatographic criteria are achieved for each positive detect. Failure to meet chromatographic criteria may indicate the presence of target and/or non-target interferences, or analytical system issues (i.e. column degradation) that prohibit the positive identification and accurate quantitation of target analytes.	2. a. Review raw chromatographic data to evaluate peak shape, resolution, and baseline.
* i. Evaluate peak shape.	i. The validator should evaluate whether or not symmetrical peaks were used for quantitating the results. If the peaks were not symmetrical, the validator should evaluate all confirmatory data, including GC/MS results, and use professional judgement to assess the impact of the unsymmetrical peaks on the report results and should justify all technical decisions in the Data Validation Memorandum. For aqueous samples, the validator should determine if an appropriate cleanup procedure was performed on the extracts. If the required cleanup procedure was not performed, then the validator should request reextraction and reanalysis by the laboratory with appropriate cleanup procedures. If cleanup procedures do not reduce interferences, the validator should note this in the Data Validation Memorandum and recommend that a more selective and/or analyte-specific analytical procedure be employed.

C. EVALUATION	D. ACTION
*2. a. ii. Evaluate chromatographic resolution.	2. a. ii. If the chromatographic peak for a positive detect is not adequately resolved on both columns, then the validator should use estimate (J) the reported result and document it in the Data Validation Memorandum. If the interfering peak is a target analyte, evaluate target analyte resolution and refer to Section II GC/ECD Instrument Performance Check.
* iii. Evaluate chromatographic baseline.	iii. If the sample chromatogram does not return to baseline after the elution of a target analyte peak or if a peak elutes as a part of a "humpogram" or coelutes with a multicomponent analyte, then interference may impede positive analyte identification and/or quantitation. The validator should use professional judgment to assess the impact of baseline rise, humpograms, and the presence of other multicomponent analytes on the positive identification of target analytes and qualify the sample data, if appropriate, and justify all technical decisions in the Data Validation Memorandum.
* b. For multicomponent analytes, in addition to the above retention time and chromatographic evaluations, the validator should evaluate the overall similarity of chromatographic patterns between the samples and the standards, while also evaluating the relative peak height ratios.	b. If the chromatographic pattern of the multicomponent analyte in the sample does not match that of the standard, then professional judgment should be used to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks).

C. EVALUATION	D. ACTION
*2. c. Ascertain if samples contain any combination of single component, multicomponent analytes and/or non-target analytes.	2. c. If samples containing single component analytes, multicomponent analytes, and/or non-target analytes have complex chromatograms, then the validator must evaluate the sample data carefully for false positives and/or false negatives as described above in C.1.a-c.
* i. Determine if target and/or non-target interferences have caused a multicomponent analyte to be incorrectly identified as another multicomponent analyte.	i. If a multicomponent analyte has been misidentified, then the validator should have the laboratory resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which identification is more accurate. The validator may qualify or reject the data. A discussion of the reasons for data qualification and the qualifiers used must be documented in the Data Validation Memorandum.
* ii. Determine if multiple multicomponent analytes are present.	ii. If an observed pattern closely matches more than one Aroclor or other multicomponent analyte, then professional judgment should be used to decide whether the other possible Aroclor is a better match, or if multiple Aroclors (multicomponent analytes) are present. If discrepancies between the raw data and reported data are found, then the validator should have the laboratory resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which identification is more accurate. The validator may qualify or reject the data. A discussion of the reasons for data qualification and the qualifiers used must be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*2. d. Review the PES data, refer to Section XI to assess the laboratory's ability to accurately identify and quantitate target analytes.	2. d. If the laboratory failed to accurately identify and/or quantitate PE samples, then sample data are suspect and sample results should be qualified according to section XI.
* e. Review the sample chromatograms to determine if any analyses showed saturated peaks. If an instrument blank was not analyzed after a high concentration sample and the instrument is not proven to be free of contamination, then the possibility of sample carryover exists.	e. If carryover is a possibility, then the validator should use professional judgment to determine whether or not a reported target analyte is a native contaminant in the sample analyzed or an interferent from a previously analyzed sample. Refer to Section V for additional guidance.
* f. Review the continuing calibration analyses of the Standard Mixtures A and B and the Performance Evaluation Mixture to identify any potential analytical problems such as decreased column resolution or compromised instrument sensitivity that could affect the interpretation of sample chromatograms.	f. If analytical problems including decreased column resolution abilities or compromised instrument sensitivity are detected in the continuing calibration analyses of the Standard Mixtures A and B and/or the Performance Evaluation Mixtures, then the validator should use professional judgment to qualify or reject sample data.
* g. Review MS/MSD data to evaluate the effect of sample matrix on analyte identification and quantitation.	g. If matrix interferences exist that either suppress or enhance the signal of spiked matrix analytes, then the validator should consider the direction of the bias and use professional judgment to qualify the sample data.
*3. Review the sample chromatograms to verify that all chromatographic plotting criteria are achieved and that sample chromatograms are properly scaled to allow the validator to evaluate the presence/absence of pesticide/PCB peaks.	3. If sample chromatograms do not demonstrate acceptable chromatographic separation and resolution, then the validator may not be able to adequately assess the presence/absence of pesticide/PCBs. In this situation the validator should contact the laboratory to replot and rescale the affected sample chromatograms.

C. EVALUATION	D. ACTION
4. a. Verify from the Form Is that GC/MS confirmation was performed for pesticide/PCB concentrations in the final sample extract which exceeded 10 ng/uL.	4. a. If GC/MS confirmation was required but not performed, then the validator should request sample analysis by GC/MS. If GC/MS sample analysis data is not provided by the laboratory, then the validator should note this non-compliance in the Data Validation Memorandum.
* b. Review the GC/MS raw data. Compare the confirmatory sample analyte spectrum to the laboratory standard spectrum and verify the mass spectral match using the VOA/SV Target Analyte Identification criteria in Section XII.	b. The application of qualitative criteria for GC/MS analysis of target analytes requires professional judgment. It is left to the validator's discretion to obtain additional information from the laboratory if it is deemed necessary. If it is determined that the presence of a pesticide/PCB was not confirmed by GC/MS, then the validator should qualify all affected data as non-detected (U) or should reject (R) the sample data indicating unusable data and document the justification for this decision in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.1.c, C.1.d, C.2.a, C.2.a.i, C.2.a.ii, C.2.a.iii, C.2.b, C.2.c, C.2.c.i, C.2.c.ii, C.2.d, C.2.e, C.2.f, C.2.g, C.3, C.4.b

E. EXAMPLES

Example #1 (False negative)

The validator reviewed the sample chromatograms for pesticide/PCB soil sample SAA12 and noted that both GC column analyses had a chromatographic peak within the calculated retention time window for heptachlor. Heptachlor was reported as not detected in sample SAA12. The laboratory correctly identified heptachlor in the PE sample. No sample matrix interferences were noted in the MS/MSD analyzed for soil sample SAA11. Since no explanation was given in the SDG narrative for reporting heptachlor as a non-detect in sample SAA12, the laboratory was contacted and requested to justify their decision or requantitate the false negative result and report heptachlor as a positive detect. The laboratory requantitated the false negative result and reported heptachlor on the Form I. As a result of the heptachlor false negative, all field sample chromatograms were carefully reviewed for potentially positive results. The validator discusses all these actions in the Data Validation Memorandum.

Example #2 (Coeluting peaks)

Dieldrin is reported on the two GC columns at concentrations of 3.5 ug/kg and 4.3 ug/kg, respectively for pesticide/PCB soil sample SAA13. The validator reviews the sample chromatograms and determines that the peak identified as dieldrin was poorly resolved and emerged as a shoulder with its peak apex coeluting with another peak (non-target analyte) on the second column. Since this peak did not meet the required chromatographic criteria, it could not be positively identified as dieldrin. The validator determined that the laboratory correctly identified dieldrin in the PE sample. The validator also determined that dieldrin was well resolved in the standards and had a symmetrical peak shape. No other sample matrix interferences were noted in the MS/MSD analyzed for soil sample SAA10. Since the coeluting peak obscured the dieldrin peak apex on the second column, the presence/absence of dieldrin is unknown below a certain concentration (4.3 ug/kg). The validator reviewed the DQOs and determined that a higher detection limit provided usable data since the action limit for dieldrin is 10.0 ug/kg and the detection limit for dieldrin in sample SAA13 was elevated to 4.3 ug/kg, the highest potential concentration of dieldrin in that sample. The validator reports the value as an estimated non-detect at the level of the interferent concentration, 4.3 (UJ), on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #3 (Coeluting peaks)

Aroclor 1260 and 4,4'-DDT were reported in aqueous sample SAA14. Aroclor 1260 concentrations were 9.8 and 9.3 ug/L and 4,4'-DDT concentrations were 5.6 and 5.8 ug/L on the two GC columns, respectively. The validator examines the chromatograms and determines that 4,4'-DDT elutes at the same retention time as one of the non-quantifying Aroclor 1260 peaks on both columns. The validator confirms that no retention time shifts have occurred in either the bracketing calibration standards or the sample surrogates. The validator concludes that the reported presence of 4,4'-DDT is suspect. However, the site DQOs have an action limit for 4,4'-DDT of 4 ug/L. The validator determines that the maximum concentration of 4,4'-DDT that could be present in sample SAA14 is 5.8 ug/L. Therefore, the validator elevates the quantitation limit for the non-detected 4,4'-DDT in sample SAA14 to 5.8 ug/L. The validator reports the SAA14 4,4'-DDT value at 5.8 (UJ) and Aroclor 1260 as 9.3 ug/L on the Data Summary Table and notes in the Data Validation Memorandum that the detection limits for 4,4'-DDT were not achieved and, therefore, the site DQOs were not achieved.

E. EXAMPLES

Example #4 (False positive due to cross-contamination)

A large peak for endrin was noted in the initial analysis of sample SAX357. A 5X dilution was required to bring the peak into the linear calibration range. Sample SAX358, which was analyzed immediately following SAX357, reported endrin only slightly above the detection limit. The validator suspects that the endrin concentration reported in sample SAX358 is a false positive result due to carryover from the original SAX357 sample analysis. The laboratory did not report the analysis of an instrument blank following the high concentration sample in the analytical sequence. The validator reviews the site DQOs and endrin is not a contaminant of concern and, therefore, uses professional judgment to reject the positive endrin detect in sample SAX358 due to possible cross-contamination from the previously analyzed sample, SAX357. The validator reports endrin as rejected (R) in sample SAX358 on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #5 (Incorrect chromatographic plotting scale)

Sample SAE632 was analyzed undiluted. Review of the chromatograms for both columns indicated that the 4,4'-DDE peak was off scale although the concentration was within the linear calibration range. The apex of the peak was not visible on the chromatograms which prevents evaluation of retention times, peak shape, and peak resolution. Since 4,4'-DDE is a contaminant of concern at the site, the validator requested the laboratory to rescale the chromatograms or reanalyze the sample in order to plot the 4,4'-DDE peak on scale. The laboratory reanalyzed and rescaled the sample to get the 4,4'-DDE peak on scale. The validator reports the reanalysis 4,4'-DDE positive detect on the Data Summary Table without qualification.

Example #6 (Missing GC/MS Confirmation Data)

Toxaphene was reported in aqueous sample SET069 at 300 ug/L in the pesticide/PCB data package. The validator notes that GC/MS confirmation was not provided but was required under the method used for analysis since the Toxaphene concentration exceeds 125 ng/uL in the final sample extract. The validator contacts the laboratory to obtain the GC/MS confirmation data. The laboratory complies and sends the required GC/MS confirmation data which meet GC/MS confirmatory criteria; therefore, the validator reports Toxaphene at 300 ug/L in sample SET069 and notes this in the Data Validation Memorandum.

XIII. COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMITS

A. OBJECTIVE

The objective for the evaluation of analyte quantitation and reported quantitation limits is to ensure that reported quantitative results and quantitation limits are accurate. To this end, laboratory calculations proceeding from raw data to the final reported concentrations are evaluated for accuracy.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the Pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have been not specified. Deviations, modifications, or non-CLP method specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. a. Reported quantitation limits must meet projected-required DQOs.

   b. Reported concentrations for positive detects and analyte quantitation limits for non-detects and adjustments of those concentrations/analyte quantitation limits must be calculated according to the appropriate method requirements.

   c. Reported concentrations for positive detects and analyte quantitation limits for non-detects must be adjusted for percent solid results, sample dilutions, and concentrations and cleanup procedures that are not accounted for in the method.

2. a. Analyte Calibration Factors (CFs) for pesticides must be calculated based on the midpoint standard established during the initial three point calibration.

   b. Analyte Calibration Factors for multicomponent analytes must be calculated based on the single-point standard established during the initial calibration.

   c. Analyte quantitation must be calculated using the CF from the appropriate initial calibration.

3. a. The lower of the two concentrations obtained on the two dissimilar GC columns is reported as the sample result.

   b. The percent difference between the concentrations reported on the dissimilar columns must not exceed 25 percent.

4. All soil/sediment sample results must be adjusted for percent solid results, and must have percent solids greater than 30 percent.[1]

[1]U.S. EPA office of Water Regulations and Standards Industrial Technology Division - Method 1620, p.29, Section 14.16 Draft, September 1989.

Sediment samples are collected at CERCLA sites to establish whether or not the presence of hazardous chemicals has impacted the resident organisms and their natural environment. The data quality objectives for ecological risk assessment generally require that the analytical method used for sediment analysis achieve, at a minimum, the dry weight CLP SOW quantitation limits.

Most analytical methods established for soil-type matrices are applicable to both soils and sediments with no difference in how those two matrices are prepared and analyzed. Since a definition for soil and sediment matrices is not provided in the analytical methodology, Region I has adopted the definition for soil samples used by the Office of Water Regulations and Standards Industrial Technology Division (ITD). This definition states that soil samples are "soils, sediments, and sludge samples containing more than 30% solids".

High moisture sediments cannot be successfully analyzed by routine CLP analytical methods. Additional sampling and analytical preparation steps which are outside of the scope of a CLP method should be employed. For example, standing water may first be decanted, and then the sample may be centrifuged or filtered to remove excess water. To achieve the dry weight quantitation limits, the laboratory must perform a percent solids analysis prior to sample extraction and the initial weight of sample extracted must be increased accordingly. This presumes that the samplers have collected sufficient sample volume, above and beyond the normal volume requirements, so that additional sample can be extracted. As a last resort, the laboratory can decrease the final extract volume to a minimum of 0.5 milliliters.

Certain solid matrices, such as peat, are unusual in their reactive chemistry. Peat is a natural sink for organic analytes. It is composed of both a solid spongy matrix (which tightly binds organic analytes) and the interstitial pore water present therein.

Routine analytical methods underestimate the concentrations of organic analytes in peat matrices because the typical organic preparation and extraction techniques do not breach the matrix. In order for peat to successfully be analyzed, the matrix itself must be "sheared" into small pieces to increase surface area so that the extraction solvent can interact with the interstitial pore water and the spongy matrix to partition the target organic analytes into the solvent.

Sampling and analytical methodologies must be determined during project scoping processes and must be based on the project data quality objectives. For more information, see Attachment A of the Data Validation Manual.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from analyte quantitation anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the reported quantitation limits meet method requirements and support project DQOs.	1. If reported quantitation limits do not meet the method requirements and/or support project DQOs, then the validator must investigate the cause of the deficiency and use professional judgment to assess the impact on the sample data. The validator must discuss this in the Data Validation Memorandum.
*2. a. Verify that sample results reported by the laboratory were accurately calculated according to the method. Recalculate, from the raw data, the concentration for at least one positive detect and one sample quantitation limit (for a diluted sample or a soil sample) for a pesticide and an Aroclor, in every field sample.	2. a. If incorrect values, equations, or factors have been used to calculate sample results and/or sample quantitation limits, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* b. Verify that the concentration for positive detects and sample quantitation limits have been adjusted to reflect sample dilutions, concentration procedures, cleanup methods, and dry weight factors that are not accounted for in the method.	b. If the concentration for positive detects and/or sample quantitation limits were not correctly adjusted for sample dilutions, concentration procedures, cleanup methods, or dry weight factors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*2. c. Compare the raw data including quantitation reports, chromatograms, and sample preparation logs to the reported positive sample results and quantitation limits on Form I PEST and Form X PEST-1 and -2.	2. c. If discrepancies between the raw and reported data are found, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*3. Verify that the correct standard CFs from the analysis of the calibration standards were used to quantitate sample results.	3. If the laboratory utilized an incorrect CF to quantitate the value for any analyte, then the validator should have the laboratory requantitate and resubmit all corrected forms and quantitation reports. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
4. a. Verify that the lowest of the two reported sample concentrations from Form X PEST was reported on Form I PEST.	4. a. If the validator determines that the higher sample concentration is more accurate than the lower concentration, then the validator should report the higher value on the Data Summary Table and clearly justify the technical decision in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
4. b. Evaluate the percent difference (%D) obtained for positive results on the two GC columns. Verify that the percent difference between the calculated concentrations is less than or equal to 25.0 %.	4. b. If the % D for sample results on the two GC columns is > 25.0%, then the chromatograms should be reviewed for chromatography, resolution, and interferences etc. (See Section XII Target Analyte Identification) and use professional judgment to qualify the results based on the following: i. For **Single Component Pesticides**, if the %D for the sample results on the two columns is greater than 25.0% but less than 100%, then the validator should estimate (J) the positive detects for that analyte in the sample. ii. For **Single Component Pesticides**, if the %D for the sample results on the two columns exceeds 100%, then the validator should reject (R) positive detects for that analyte in the sample. However, professional judgement should be used to evaluate the chromatography before rejecting positive results. iii. For **Multicomponent Analytes**, if the %D for the sample results on the two columns is greater than 25.0% but less than 500%, then the validator should estimate (J) positive detects for that analyte in the sample. iv. For **Multicomponent Analytes**, if the %D for the sample results on the two columns exceeds 500%, then the validator should reject (R) positive detects for that analyte in the sample. v. If one sample concentration of either single component pesticides or multicomponent analytes is less than the quantitation limit and the other sample concentration is greater than or equal to the quantitation limit, then the validator should use professional judgment to determine which value to report and should qualify the reported positive value as estimated (J) or the reported quantitation limit as estimated (UJ).

C. EVALUATION	D. ACTION
5. Determine if any soil/sediment/solid sample has less than or equal to 30 percent solids.	5. a. If a soil/sediment/solid sample has greater than 30 percent solid results, then the validator should accept all sample data. b. If a soil/sediment/solid sample has percent solids of greater than or equal to 10 percent but less than or equal to 30 percent, then the validator should: ● Estimate (J) positive detects. ● Reject (R) non-detects. c. If a soil/sediment/solid sample has less than 10 percent solids, then the validator should reject (R) positive and non-detect sample results as unusable. d. The validator should include a discussion of the sample matrices having low percent solids in the Data Validation Memorandum. The validator may need to contact the field sampler to determine whether sampling techniques were appropriate for the sample matrix.
6. Determine whether or not the laboratory performed procedures to remove the aqueous medium and increased the sample size to account for low percent solids.	6. If the laboratory performed procedures to decrease the aqueous medium and increased the sample size, then the validator should check the chromatograms for interferences and possible false negatives and use professional judgement to accept, qualify, and/or reject data. The validator must also verify that the sample-specific quantitation limits were adjusted and that the adjusted levels meet the project DQOs.

Note: The following subsections are applicable only to a Tier III data validation:

C.2.a, C.2.b, C.2.c, C.3

Table Pest/PCB-XIII-1:

QUALIFICATION OF PESTICIDE/PCB ORGANIC ANALYTES BASED ON SAMPLE PERCENT SOLIDS

Sample Result	% Solids > 30%	10% ≤ % Solids ≤ 30%	% Solids < 10%
Detects	A	J	R
Non-detects	A	R	R

Table Pest/PCB-XIII-2:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON %D OF ANALYTES BETWEEN TWO QUANTITATION COLUMNS.

%D Between the Two Quantitation Columns	Sample Results	
	Detects	Non-Detects
25.0% < %D < 100% (single component pesticides)	J	N/A*
%D > 100% (single component pesticides)	R	N/A
25.0% < %D < 500% (Multicomponents)	J	N/A
%D > 500% (Multicomponents)	R	N/A
One Value < QL & One Value ≥ QL	J	UJ

* N/A - Not Applicable

E. EXAMPLES

Example #1: (25% < %D ≤ 100%)

The laboratory detected endosulfan sulfate in soil sample SAA12 on column 1 at 5.6 ug/kg and on column 2 at 8.2 ug/kg. The %D between the two quantitated endosulfan sulfate results is 46.4%. Since the %D is greater than 25% but less than 100%, the validator estimates (J) the positive detect for endosulfan sulfate on the Data Summary Table and reports this in the Data Validation Memorandum.

Example #2: (%D > 500%)

The laboratory detected Aroclor 1262 in soil sample SAA56 on column 1 at 110 ug/kg and on column 2 at 15 ug/kg. The %D between the two columns for this analyte is 630%. Since the %D is greater than 500%, the validator rejects (R) the positive detect for Aroclor 1262 as unusable in sample SAA56. The validator reports Aroclor 1262 as rejected (R) on the Data Summary Table and discusses this in the Data Validation Memorandum.

Example #3: (%D > 25%)

A positive result for dieldrin was reported in sample SAA13. The analyte concentrations were 1.8 and 3.0 ug/L on the first and second column (66 %D), respectively. The percent difference is great enough that the validator suspects a potential interference on one of the columns. The validator reviews the sample chromatograms and determines that chromatographic resolution criteria on the second column for the dieldrin peak have not been met. Dieldrin is not resolved from an interferant, resulting in an elevated concentration quantitated from the second column. The validator uses professional judgment to estimate the lower concentration and reports it on the Data Summary Table as 1.8J and discusses this in the Data Validation Memorandum.

Example #4: (10% ≤ % Solids < 30%)

DQOs for the Maple Street Site call for low PCB detection limits to assess human health risk posed by the site contamination. Soil sample SAA100 had 15% solids and a positive detect for Aroclor 1254. Due to low percent solids, the Aroclor 1254 positive detect in sample SAA100 is estimated (J) and all PCB non-detects are rejected (R) in sample SAA100 due to percent solids less than 30%. The validator reports the qualified data on the Data Summary Table and notes that the non-detected data are unusable and do not meet project DQOs in the Data Validation Memorandum.

Example #5: (% Solids < 10%)

Sediment sample SAA90 had 8% solids and positive detects for 4,4'-DDT and 4,4'-DDE. As a result of the extremely low percent solids (< 10%), the validator rejects (R) all positive detects and non-detects for this sample as unusable. The validator contacts the sampler to determine if the sampling techniques which were used accommodated the high moisture samples. The validator reports the data as rejected (R) on the Data Summary Table and discusses the high moisture content of the sample and the need to perform sampling techniques specific to samples with high moisture content with analytical method alternatives to accommodate the high moisture samples in the Data Validation Memorandum.

XIV. SYSTEM PERFORMANCE

A. OBJECTIVE

The objective of assessing overall system performance is to determine if any method preparatory and/or analytical procedures result in qualitative and/or quantitative system error or bias. All sample, QC sample, and blank results are reviewed for accuracy, chromatography, precision, sensitivity, and contamination to ascertain if there are any general trends in data quality.

B. CRITERIA

Since there are no specific criteria for system performance, professional judgment should be used to assess the overall performance.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
*1. The results of Zero, Single, and Double Blind PESs, MDL Study, LFB, Performance Evaluation Mixture (PEM), calibration standards, MS/MSD, surrogate spike analytes, GPC calibration verification, and Florisil cartridge check analyses may be used to assess the overall system accuracy including extraction efficiency and instrument response. * a. Evaluate all PES and other relevant QC data to determine if any analytical trends exist over the sample analysis period. * b. The validator should ascertain from the PES and other relevant QC data if there is a high or low quantitative bias for a particular analyte or group of analytes. * c. The validator should also determine from the PES and other relevant QC data if there is a potential for false negatives and/or false positives to be reported. * d. The validator should ascertain from the MS/MSD and surrogate spike analyte analyses if the sample matrix effects impact analyte recovery, thus indicating a method bias outside the control of the laboratory. * e. The validator should ascertain from the GPC calibration verifications and Florisil cartridge check analyses whether sample cleanup techniques impact analyte recovery.	1. The validator should refer to the previous sections for specific guidance in evaluating accuracy using PES, MDL study, LFB, PEM, calibration standard, MS/MSD, surrogate data, GPC calibration verification, and Florisil cartridge check. If the validator determines that analytical trends indicate a qualitative and/or quantitative systematic bias, then the validator should use professional judgment to determine whether or not to qualify or reject the sample data based on the extent of the impact. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should differentiate between sample matrix-related preparatory and analysis problems that are outside the laboratory's control and those within the laboratory's control.

C. EVALUATION	D. ACTION
*2. The results of the Resolution Check Mixture (RCM), PEM, PES, LFB, and calibration standard analyses as well as field samples may be used to assess the overall performance of the chromatographic system. * a. Evaluate sample and QC sample chromatograms analyzed on all columns to determine if the column chromatography, peak shape, resolution, and baseline drift has either deteriorated or improved over the sample analysis period. * b. The validator should determine from the raw data if unacceptable chromatography, e.g., baseline drift, high background noise, loss of resolution, peak tailing, or peak splitting, may contribute to a high or a low quantitative bias for a particular analyte or group of analytes. * c. The validator should also determine from the raw data if unacceptable chromatography, e.g., baseline drift, high background noise, loss of resolution, peak tailing, or peak splitting, may result in a potential for false negative and/or false positive identifications.	2. The validator should refer to the previous sections for specific guidance on evaluating analyte identification and quantitation. If the validator determines that chromatographic trends indicate a qualitative and/or quantitative systematic bias, then professional judgment should be used to determine whether or not to qualify or reject the sample data based on the extent of the impact. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should especially note when chromatography problems and column degradations are caused by severe matrix interferences. The validator should recommend additional cleanup procedures and/or alternate analytical methods for future site work.

C. EVALUATION	D. ACTION
*3. The results of the PEM, calibration standard, MDL Study, surrogate spike analyte, MS/MSD, and field duplicate analyses may be used to assess overall system precision. * a. Compare all of the the daily standard calibrations and PEMs area counts throughout analytical sequence to ascertain if the instrument had consistent detector response over the sample analysis period. * b. Review the size of the solvent peak and the area counts of the surrogate analytes for each sample to ascertain if there is a change in detector response. * c. The validator should evaluate the MS/MSD RPDs in conjunction with field duplicate RPDs to identify any analytical trends, ascertain if sample matrices were homogeneous or heterogeneous, and determine if sampling error may have contributed to field imprecision. * d. Verify that samples were analyzed on the same instrument and under the same conditions as were used for the MDL Study analyses.	3. The validator should refer to the previous sections for specific guidance on evaluating laboratory and field precision and surrogate analyte analyses. If the validator determines that an instrument produces erratic detector responses, then the validator should use professional judgment to qualify or reject sample data. If MS/MSD RPDs indicate laboratory imprecision, then the validator should suspect laboratory technique and take into consideration the results of the field duplicate RPDs when using professional judgment to qualify sample data. If field duplicate RPDs indicate field imprecision resulting from heterogeneous sample matrices or field sampling error, then the validator should use professional judgment to qualify sample data based on the extent of impact. The validator should differentiate between lack of precision due to instrument performance problems and ones caused by matrix effects or sampling error.

C. EVALUATION	D. ACTION
*4. The results of the LFB, PES, calibration standards, and PEM analyses may be used to assess the overall system sensitivity. * a. Review all daily LFBs, low level calibration standards, and PES data to evaluate sensitivity for each instrument to verify that the target analytes can be identified and accurately quantitated at the quantitation limit over the sample analysis period. These problems could potentially result in false negatives and low biased results. * b. Check the area counts of the daily PEM and calibration standards to monitor changes in instrument sensitivity. * c. Compare the area counts of surrogates in each sample throughout the analytical sequence to determine if any samples show unacceptably low counts. * d. Review the sample chromatograms for abrupt, discrete shifts in the chromatographic baseline which may indicate a change in the instrument's sensitivity or the zero setting. A baseline "decline" could indicate a decrease in sensitivity in the instrument or an increase in the instrument zero, possibly causing target analytes, at or near the detection limit, to be missed (false negatives). Additionally, a decline in the baseline may result in incorrect peak integration and produce inaccurate quantitation. A sudden baseline shift could indicate problems such as a change in the instrument zero, a leak, degradation of the column, or the formation of matrix degradation products. The validator should check for any abrupt shift in the zero setting which may cause a false positive to be reported. Additionally, a rise in the baseline may result in incorrect peak integration and subsequent misquantitation. * e. The validator may determine that instrument sensitivity is adequate but sample matrix effects may preclude the project required detection limits from being obtained while using the current analytical methods.	4. The validator should refer to the previous sections for specific guidance on evaluating sensitivity, accuracy, analyte identification, and quantitation. If the validator determines that instrument sensitivity is unacceptable, then the validator should use professional judgment to qualify or reject the affected sample data. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should also note if sample matrix interferences prohibited the quantitation limits from being achieved and should recommend additional cleanup procedures and/or alternate analytical methods for future site work.

C. EVALUATION	D. ACTION
*5. The results of the PES method, instrument, equipment/rinsate, trip, and bottle blank analyses may be used to assess overall system contamination. * a. Review all blank and sample results to evaluate the possibility that sample contamination was introduced via cross-contamination from either a previously analyzed sample, or from field or laboratory general contamination. * b. Compare blank analysis on two different instruments to determine if the contamination is instrument related or the interferents are present in the blank as a result of sample processing activities.	5. The validator should refer to the previous sections for specific guidance on evaluating blank contamination. If the validator determines that there is a systematic blank error introduced during sample collection or processing (extraction or analysis), then the data should be qualified according to Section V. However, if the validator suspects intermittent or sporadic introduction of interferents during analysis, then the validator should use professional judgment to qualify or reject sample data and document and justify all technical decisions in the Data Validation Memorandum.

* **Note:** **This section is only applicable to a Tier III data validation - If a validator suspects system performance has degraded to the degree that data are affected and only a Tier II validation has been requested, then the validator should contact the Site Manager to approve the necessary Tier III validation.**

E. EXAMPLES

Example #1: (Low LFB recoveries; Decreasing counts of surrogate analytes)

During the review of pesticide/PCB data, the validator notices a trend of decreasing external standard areas over a discrete analytical time period on the instrument. The validator notes that the majority of LFB analyte recoveries are below 60%. The validator also notes that surrogate analyte area counts show a decreasing trend throughout the analytical sequence. The validator uses professional judgment to estimate (J) all positive detects and estimate (UJ) the quantitation limits of all non-detects for all samples analyzed during the analytical sequence. The validator reports the qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

Example #2: (Abrupt decreasing baseline)

The validator notices an abrupt decrease in the baseline the instrument during the analysis of sample SAP01. The validator notes that the area of the last eluting surrogate analyte in sample SAP01 differs significantly from the areas of the same surrogate from the previous sample runs. The validator inspects the chromatogram and observes abrupt decrease baseline in the middle of sample SAP01 run. The validator suspects that target analytes eluting after the abrupt baseline shift which are at or near the detection limit may not be detected. Therefore, the validator estimates (J) the positive detects and rejects (R) the non-detects that elute after the abrupt baseline shift. The validator reports the qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

Example #3: (Several PES analytes show low bias; %D of continuing calibration unacceptable; Several LCS analytes show low bias)

The validator reviews the results of the Region I EPA PES sample analyzed by the laboratory and discovers that over half of the PES analytes were scored "Action Low". The validator determines that a low bias for the detected analytes in all samples has occurred. In addition to the low bias problem, the validator discovers that several of the same analytes in the PEM standards had greater than 25% Difference in the continuing calibration. The validator also notices that the LCS analyte recoveries for over half of the analytes were less than 60% indicating a potentially low bias for all positive detects. As a result of the analytical trend identified above, the validator estimates (J) the positive detects and rejects (R) the non-detects in all field samples. The validator reports the qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

Example #4: (Corruption of analytical system by oily sample)

The validator reviews the PEM analyzed during the continuing calibration and discovers that the 4,4'-DDT and Endrin breakdown criteria were not met for either chromatographic column after the analysis of sample SAA746. However, the sample results for sample SAA746 were acceptable as all QC criteria were met. The validator noted several other problems which occurred after the analysis of sample SAA746, e.g., dissimilar column %Ds for field samples did not meet criteria, peak tailing, and an upward drift in the baseline. The validator proceeded to check the preparatory log sheets and notes that the sample has a thick and oily consistency which would contribute to the unacceptable chromatography in the samples and standards analyzed after SAA746. The validator uses professional judgment to qualify sample data resulting from poor chromatography caused by the analysis of an oily matrix. The validator reports the qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

XV. OVERALL EVALUATION OF DATA

A. OBJECTIVE

The objective of the final evaluation of a data package is to identify the "analytical error" and any "sampling error" associated with the data. The sum of the "analytical error" and the "sampling error" equals the "measurement error". "Measurement error" will then be used by the end user in conjunction with sampling variability (spatial variations in pollutant concentrations) to determine "total error" (total uncertainty) associated with the data. Ultimately, the end data user will assess data usability in the context of the pre-determined Data Quality Objectives (DQOs) and resultant "total error" of the data.

B. CRITERIA

The Sampling and Analysis Plan (SAP) or Quality Assurance Project Plan (QAPP) and DQO Summary Form should specify the site specific DQOs and acceptable levels of uncertainty or "total error".

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
1. Obtain the SAP, QAPP, or DQO Summary Form for the project. Thoroughly review and understand the DQOs for the sampling event.	1. In the first section of the Data Validation Memorandum, entitled Overall Evaluation of Data, summarize the appropriate project DQOs. The summary should be in a bulleted format.
2. Evaluate the appropriateness of the analytical method chosen by reviewing all the discussions in the previous sections. For example, was the method capable of achieving quantitation limits sufficiently low to meet DQOs for risk assessment? Was the method capable of successfully analyzing each particular matrix sampled? Was a “low” method chosen when the samples were likely to contain concentrations well above the upper calibration range?	2. If an inappropriate method was chosen for sample analysis, then the validator should discuss the method deficiencies and identify more appropriate methods or modifications for use in subsequent sampling rounds. The validator should include this discussion in the Overall Evaluation of Data section of the Data Validation Memorandum.
3. Evaluate any analytical problems that were identified by reviewing all the discussions in the previous sections.	3. Estimate and describe the "analytical error" that contributes to the "measurement error" associated with the data package in the Overall Evaluation of Data section of the Data Validation Memorandum. a. If "analytical error" results in unusable data, then the validator should reject the data and return it to the laboratory and deny payment as per Attachment I. b. If "analytical error" results in data of reduced worth to the Region, then the validator should recommend a reduction in the laboratory’s payment. See Attachment I.

C. EVALUATION	D. ACTION
4. Evaluate any sampling issues that were identified in the previous sections. Note: The validator is only responsible for evaluating those "sampling errors" that are identified during the routine data validation process. Other "sampling errors" may have occurred and they should be assessed by the end user prior to data use.	4. Estimate and describe the "sampling error" that contributes to the "measurement error" associated with the data package in the Overall Evaluation of Data section of the Data Validation Memorandum. Examples of "sampling error" for which the validator would have information include highly contaminated equipment blanks as well as delayed sample shipment that caused holding time violations; elevated moisture content, etc. a. If "sampling error" severely impacts potential data usability, then the validator should note this in the Data Validation Memorandum and contact the EPA project manager. b. The end user should review the results of the sampler's field notes/trip report to determine additional "sampling error" issues with which to fully assess "measurement error".
5. Evaluate data quality in terms of "measurement error" as a combination of "analytical error" and "sampling error".	5. Discuss data quality in terms of "measurement error" as the sum of "analytical error" and "sampling error". All discussions should be included in the Overall Evaluation of Data section of the Data Validation Memorandum.
6. Identify potential usability issues raised by an unacceptable degree of "measurement error".	6. If data usability is potentially compromised by a high degree of "measurement error", then the validator should note this in the Overall Evaluation of Data section of the Data Validation Memorandum. If data quality impacts the use of those data by the end user, then the validator should detail in the Overall Evaluation of Data section of the Data Validation Memorandum how data use will be limited and for which end user, i.e., risk assessor, hydrogeologist, etc.
7. Sampling variability is not assessed during data validation, and therefore, should be assessed by the end user prior to data use.	7. The end user should review the results of the Data Validation Memorandum in conjunction with the sampler's field notes/trip report to assess the impact of sampling variability issues on data usability.

APPENDIX F

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM04.3, Exhibit D Pesticides/Aroclors**.

SECTION I: PRESERVATION & HOLDING TIME CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-I-B for preservation and holding time data validation criteria.

SECTION II: GC/ECD INSTRUMENT PERFORMANCE CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-II-B for GC/ECD Instrument Performance Check data validation criteria and the following method GC/ECD instrument performance check QC criteria:

Resolution Check Mixture

A Resolution Check Mixture containing the following analytes must be analyzed at the beginning of the initial calibration sequence. The resolution between two adjacent peaks in the Resolution Check mixture must be greater than or equal to 60.0%.

Analyte	Concentration (ng/mL)
gamma-Chlordane	10.0
Endosulfan I	10.0
4,4'-DDE	20.0
Dieldrin	20.0
Endosulfan sulfate	20.0
Endrin ketone	20.0
Methoxychlor	100.0
Tetrachloro-m-xylene (surrogate)	20.0
Decachlorobiphenyl (surrogate)	20.0

Resolution Check - The % Resolution is calculated using the following equation:

$$\% \ Resolution = \frac{V}{H} \ x \ 100$$

Where,

V = Depth of valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.
H = Height of the shorter peak.

Example

IF:
Height of Peak A = 2560
Height of Peak B = 1435

The resolution (depth of the valley) between the two peaks must be at least 60.0% of the shorter peak or Peak B in this situation (0.6 x 1435 = 861). If the resolution between Peak A and Peak B is less than 861, the data will need to be qualified as indicated in Tables Pest/PCB-II-1 and Pest/PCB-II-2.

Performance Evaluation Mixture

A Performance Evaluation Mixture (PEM) containing the analytes listed below must be analyzed at the beginning of the initial calibration sequence, immediately after the Resolution Check mixture and at the end of the initial calibration sequence. It must also be analyzed once during every 24 hours of the analytical sequence as part of the calibration verification. The resolution between two adjacent peaks in the PEM must be greater than or equal to 90.0%. The percent breakdown for both DDT and Endrin in each PEM must be less than or equal to 20.0% for both GC columns. The combined percent breakdown for DDT and Endrin in each PEM must be less than or equal to 30.0% for both GC columns.

Analyte	Concentration (ng/mL)
gamma-BHC	10.0
alpha-BHC	10.0
4,4'-DDT	100.0
beta-BHC	10.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene (surrogate)	20.0
Decachlorobiphenyl (surrogate)	20.0

Percent Difference (% D) - The % D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM analysis of the initial calibration sequence on each GC column must be less than or equal to ±25.0%. The % D is calculated using the following equation:

$$\% \ Difference = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

Where,

C_{calc} = Calculated concentration of each analyte from the analyses of the calibration standards.
C_{nom} = Nominal concentration of each analyte.

4,4'-DDT/ENDRIN BREAKDOWN CALCULATIONS

$$\% \text{ Breakdown DDT} = \frac{\text{Amount found (ng) (DDD + DDE)}}{\text{Amount (ng) of DDT injected}} \times 100$$

$$\% \text{ Breakdown Endrin} = \frac{\text{Amount found(ng) (Endrin Aldehyde + Endrin Ketone)}}{\text{Amount (ng) of Endrin injected}} \times 100$$

$$\text{Combined \% Breakdown} = \text{\% Breakdown DDT} + \text{\% Breakdown Endrin}$$

SECTION III: INITIAL CALIBRATION CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

Each GC/ECD system must be initially calibrated using the following sequence:

INITIAL CALIBRATION SEQUENCE

1. Resolution Check
2. Performance Evaluation Mixture
3. Aroclor 1016/1260
4. Aroclor 1221
5. Aroclor 1232
6. Aroclor 1242
7. Aroclor 1248
8. Aroclor 1254
9. Toxaphene
10. Low Point Standard A
11. Low Point Standard B
12. Midpoint Standard A
13. Midpoint Standard B
14. High Point Standard A
15. High Point Standard B
16. Instrument Blank
17. Performance Evaluation Mixture

Table App.F.III-1 - ANALYTES CONTAINED IN INDIVIDUAL STANDARD MIXTURE A AND INDIVIDUAL STANDARD MIXTURE B

IND STD MIXTURE A	LOW POINT CONCENTRATION (ng/mL)
alpha-BHC	5.0
Heptachlor	5.0
gamma-BHC	5.0
Endosulfan I	5.0

Dieldrin	10.0
Endrin	10.0
4,4'-DDD	10.0
4,4'-DDT	10.0
Methoxychlor	50.0
Tetrachloro-m-xylene (surrogate)	5.0
Decachlorobiphenyl (surrogate)	10.0

IND STD MIXTURE B	LOW POINT CONCENTRATION (ng/mL)
beta-BHC	5.0
delta-BHC	5.0
Aldrin	5.0
Heptachlor epoxide	5.0
alpha-Chlordane	5.0
gamma-Chlordane	5.0
4,4'-DDE	10.0
Endosulfan sulfate	10.0
Endrin aldehyde	10.0
Endrin ketone	10.0
Endosulfan II	10.0
Tetrachloro-m-xylene (surrogate)	5.0
Decachlorobiphenyl (surrogate)	10.0

Table App.F.III-2 - MULTICOMPONENT ANALYTE LOW POINT CONCENTRATIONS

MULTICOMPONENT ANALYTES	LOW POINT CONCENTRATIONS (ng/mL)
Aroclor 1016	100
Aroclor 1221	200
Aroclor 1232	100
Aroclor 1242	100
Aroclor 1248	100
Aroclor 1254	100
Aroclor 1260	100
Toxaphene	500

Multicomponent standards including the Aroclors and Toxaphene must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture.

RETENTION TIME WINDOW CALCULATION

In the initial calibration (low, mid, and high point) the absolute retention times (RTs) are measured for all single component pesticides, the surrogates, and at least three major peaks of each multicomponent analyte. The mean RTs for single component standards and surrogates are calculated as the average of the three values and the RTs for multicomponent analytes are based on one value.

A retention time window for each single component pesticide and surrogate and for three to five major peaks of each multicomponent analyte are calculated using the values in Table App.F.III-3.

The following equation is used when calculating a mean absolute retention time for each single component pesticide and surrogate:

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where,

$\overline{RT}$ = Mean absolute retention time of analyte.

RT_i = Absolute retention time of analyte.

n = Number of measurements (3).

Example

The retention time window is calculated by first taking the mean of the retention times from the low, mid, and high concentration of the individual standards in the initial calibration. For example the retention times for Endrin are:

Low - 9.86 Mid - 9.85 High - 9.86

Mean = 9.86

Since we know from Table App.F.III-3 that the retention time window for Endrin is ±0.07, we add and subtract 0.07 to and from the Mean to calculate the retention time window for Endrin from the initial calibration.

RT Window for Endrin = 9.79 - 9.93

Table App.F.III-3 - RETENTION TIME WINDOWS FOR ANALYTES AND SURROGATES

Analyte	Retention Time Window in Minutes
alpha-BHC	±0.05
beta-BHC	±0.05
gamma-BHC	±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin	±0.05
alpha-Chlordane	±0.07
gamma-Chlordane	±0.07
Heptachlor Epoxide	±0.07

Analyte	Retention Time Window in Minutes
Dieldrin	±0.07
Endrin	±0.07
Endrin Aldehyde	±0.07
Endrin Ketone	±0.07
DDD	±0.07
DDE	±0.07
DDT	±0.07
Endosulfan I	±0.07
Endosulfan II	±0.07
Endosulfan Sulfate	±0.07
Methoxychlor	±0.07
Aroclors	±0.07
Toxaphene	±0.07
Tetrachloro-m-xylene (surrogate)	±0.05
Decachlorobiphenyl (surrogate)	±0.10

CALIBRATION FACTOR CALCULATION

In the initial calibration, peak areas or peak heights are measured to determine the analyte Calibration Factor (CF). The Calibration Factor for each single component pesticide and surrogate and for each peak in the selected set of 3 to 5 major peaks for each multicomponent analyte is calculated using equation III-1. The mean CF for each single component pesticide and surrogate is calculated using equation III-2. **Note: The single component pesticide mean CF is only used in calculating the % RSD and not for quantifying sample results.**

Equation III-1 is as follows:

$$CF = \frac{\text{Peak Area (height) of the standard}}{\text{Mass injected (ng)}}$$

Equation III-2 is as follows:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where,

$\overline{CF}$ = Mean calibration factor.
CF_i = Calibration factor.
n = Total number of values (3).

% RSD CALCULATION

Initial calibration data is used to assess the linearity of each GC/ECD system used for sample analysis. The linearity of the GC is assessed by calculating a % RSD of the calibration factors for each single component pesticide and surrogate.

Equation III-3 is as follows:

$$\% \ RSD = \frac{SD_{CF}}{\overline{CF}} \ x \ 100$$

Where,

$$SD_{CF} = \sqrt{\sum_{i=1}^{n} \frac{(CF_i - \overline{CF})^2}{(n-1)}}$$

$\overline{CF}$ = Mean calibration factor.
CF = Each individual Calibration Factor used to calculate the mean.
n = Total number of values (3).
% RSD = Percent Relative Standard Deviation.
SD_{CF} = Standard Deviation of the calibration factors.

SECTION IV: CALIBRATION VERIFICATION CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IV-B for calibration verification data validation criteria and the following method calibration verification QC criteria:

The analyses of the instrument blanks, Performance Evaluation Mixtures (PEM), and the midpoint concentration of Individual Standard Mixtures A and B constitute the calibration verification. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

A valid analysis sequence is given below:

1. Sequence

Time	Injection #	Material Injected
	1 - 15	First 15 steps of the Initial Calibration
0 hr.	16	Instrument Blank* at the end of the Initial Calibration
	17	PEM at the end of the Initial Calibration
	18	First Sample
	o	
	o	Subsequent Samples
	o	
12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd injection past 12:00 hr.	Performance Evaluation Mixture
	o	Sample
	o	
	o	Subsequent Samples
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	etc.	

*The instrument blank contains only the surrogate analytes: Tetrachloro-m-xylene and Decachlorobiphenyl.

PERCENT DIFFERENCE (% D) - The % D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM and Individual Mixture runs of the calibration verification on each GC column must be less than or equal to ± 25.0%. The % D is calculated using the following equation:

$$\% D = \frac{C_{nom} - C_{calc}}{C_{nom}} \times 100$$

Where,

C_{calc} = Calculated concentration of each analyte from the analyses of the standards.
C_{nom} = Nominal concentration of each analyte.

Example

The nominal amount of gamma-BHC added to the PEM is 10 ng/mL. The calculated amount of gamma-BHC is found to be 12 ng/mL. Using the above equation, the percent difference is calculated as follows:

$$\% D = \frac{12 - 10}{10} \times 100 = 20\%$$

Example

The nominal amount of 4,4'-DDT added to the INDA is 10 ng/mL. The calculated amount of 4,4'-DDT is found to be 9.0 ng/mL. Using the above equation, the percent difference is calculated as follows:

$$\% D = \frac{9.0 - 10}{10} \times 100 = -10\%$$

Example

The nominal amount of Aldrin added to the INDB is 5.0 ng/mL. The calculated amount of Aldrin is found to be 7.0 ng/mL. Using the above equation, the percent difference is calculated as follows:

$$\% D = \frac{7.0 - 5.0}{5.0} \times 100 = 40\%$$

SECTION V: BLANK CRITERIA

Method Required Blanks

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-V-B for blank data calibration criteria and the following method QC criteria:

1. Method Blank - A volume of reagent water or purified solid matrix, approximate in weight or volume to the samples, which is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of the entire set of samples. Surrogate analytes must recover between 30 - 150%. A method blank must be extracted and analyzed once per each SDG, or each matrix within an SDG, or each extraction procedure within an SDG, or whenever samples are extracted, whichever is most frequent.

2. Sulfur Cleanup The sulfur cleanup blank is a modified form of the method blank. It is a volume of clean solvent spiked with the surrogates and carried through the sulfur cleanup and analysis procedures. This blank is used to determine the levels of contamination associated with the separate sulfur cleanup steps. Surrogate analytes must recover between 30 - 150%. The sulfur cleanup blank is prepared separately when only part of a set of samples

extracted together requires sulfur removal. If all of the samples associated with a given method blank require sulfur cleanup, then the method blank must be subjected to sulfur cleanup and no separate sulfur cleanup blank is required.

3. Instrument Blank - The instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrument analysis itself, such as the carry-over of analytes from standards or highly contaminated samples into other analyses. An instrument blank must be analyzed after a sample whose concentration exceeds the calibration range. Until an instrument blank meets the technical acceptance criteria, the system is considered contaminated.

   A GPC blank, a type of instrument blank, is analyzed after the initial calibration of the GPC. It is not spiked with surrogate analytes.

SECTION VI: SURROGATE ANALYTE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VI-B for the surrogate analyte data validation criteria and the following method surrogate analyte QC criteria:

Table App.F.VI-1 - SURROGATE RETENTION TIME WINDOWS

Surrogate	Retention Time Window in Minutes
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

The surrogate % recovery is calculated using the following equation:

$$Surrogate\ Percent\ Recovery = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of surrogate determined by analysis.

Q_a = Quantity of surrogate added to sample/blank.

Table App.F.VI-2 - SURROGATE SPIKE RECOVERIES

Surrogate	Percent Recovery* (Soil/Sediment and Water)
Tetrachloro-m-xylene	30-150
Decachlorobiphenyl	30-150

* Advisory for sample analysis. Mandatory for method blanks/sulfur cleanup blanks

SECTION VII: PESTICIDE CLEANUP CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST-VII-B for pesticide cleanup data validation criteria and the following method pesticide cleanup QC criteria:

GPC

The Initial GPC Calibration consists of analyzing the GPC Calibration and GPC Calibration Verification solutions to establish the correct "Collect" and "Dump" time periods and a GPC blank to ensure that the system is free of contaminants.

1. The GPC Calibration Solution contains the following analytes in methylene chloride:

   corn oil
   bis-(2-ethylhexyl)phthalate
   methoxychlor
   perylene
   sulfur

2. The GPC blank consists of methylene chloride.

Table App.F.VII-1 - GPC CALIBRATION ACCEPTANCE CRITERIA

Peak Resolution	Corn Oil and phthalate peaks must exhibit > 85% resolution. Phthalate and methoxychlor peaks must exhibit > 85% resolution. Methoxychlor and perylene peaks must exhibit > 85% resolution. Perylene and sulfur peaks must not be saturated and must exhibit > 90% baseline resolution.
Peak Shape	Peaks must be observed and symmetrical for all analytes in the calibration solution.
Retention Time	The retention times must not vary more than ± 5.0% between calibrations.
Blanks	A GPC blank must be analyzed after each initial GPC calibration and target analytes cannot be present at greater than the quantitation limit for any target analyte.

The Continuing GPC Calibration consists of analyzing the following two solutions in sequence every 7 days.

1. The Pesticide GPC Calibration Verification Solution contains the following 6 pesticide analytes (same as matrix spiking solution) in methylene chloride:

   gamma-BHC
   heptachlor
   aldrin
   dieldrin
   endrin
   4,4'-DDT

2. The PCB GPC Calibration Verification Solution contains 2 ug/mL each of Aroclors 1016 and 1260 in methylene chloride.

Table App.F.VII-2 - GPC CALIBRATION VERIFICATION CRITERIA

% Recovery of Single Component Pesticides	80 - 110%
Aroclor Patterns	Elution patterns must be the same as those from the Aroclor 1016 and Aroclor 1260 standards in the initial calibration sequence.

Florisil Cartridge

The Florisil Cartridge Performance Check consists of testing every lot of Florisil prior to use or every 6 months, whichever is more frequent, by analyzing the following two solutions in a mixture.

1. The Florisil Cartridge Check Solution contains 2,4,5-trichlorophenol in acetone, at a concentration of 0.1 ug/mL.

2. The Pesticide Spiking Solution contains the following Standard Mixture A analytes at the midpoint concentration:

alpha-BHC	4,4'-DDD
heptachlor	4,4'-DDT
gamma-BHC	methoxychlor
endosulfan I	tetrachloro-m-xylene
dieldrin	decachlorobiphenyl
endrin	

Table App.F.VII-3 - FLORISIL CARTRIDGE PERFORMANCE CHECK CRITERIA

INDA Analyte % Recovery	80% - 120%
2,4,5-TCP Recovery	2,4,5-Trichlorophenol must recover at < 5.0%
Other Target Analytes	No interfering peaks with the target analytes

SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VIII-B for MS/MSD data validation criteria and the following method MS/MSD QC criteria:

A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix for each SDG, or each matrix within an SDG, or each group of samples of a similar concentration level.

The following advisory matrix spike analyte recoveries and RPDs are listed below:

Table App.F.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Method QC Criteria			
	Water		Soil/Sediment	
	% Recovery*	RPD**	% Recovery	RPD
gamma-BHC (Lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

*The MS/MSD % recovery is calculated using the following equation:

$$Matrix\ Spike\ Recovery = \frac{SSR - SR}{SA} X\ 100$$

Where,

SSR = Spike Sample Result.
SR = Sample Result.
SA = Spike Added.

**The MS/MSD relative percent difference (RPD) is calculated using the following equation:

$$Relative\ Percent\ Difference = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)}\ x\ 100$$

Where,

MSR = Matrix Spike Recovery.
MSDR = Matrix Spike Duplicate Recovery.

Note: The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always positive.

SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IX-B for field duplicate data validation criteria.

SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-X-B for sensitivity check data validation criteria.

SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XI-B for accuracy check data validation criteria.

SECTION XII: TARGET ANALYTE IDENTIFICATION CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XII-B for target analyte identification data validation criteria.

SECTION XIII: ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XIII-B for analyte quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Pesticide/PCB analytes must be quantified using the external standard method. The midpoint calibration factor from the most recent compliant initial calibration must be used to quantify sample single component pesticides on both columns and the lower sample concentration must be reported. For multicomponent analytes, the calibration factors for three to five major peaks from the most recent compliant initial calibration are each used to quantify the multicomponent analyte in the sample. The three to five concentrations are then averaged and an analyte mean concentration for the sample is calculated for each column. The lower mean concentration is reported. Pesticides/PCBs must be reported to the CRQLs listed below:

Table App.F.XIII-1 - TARGET ANALYTE LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

	Quantitation Limits		
	Water	Soil	On Column
Pesticides/Aroclors	ug/L	ug/kg	(pg)
alpha–BHC	0.050	1.7	5
beta–BHC	0.050	1.7	5
delta–BHC	0.050	1.7	5
gamma–BHC	0.050	1.7	5
Heptachlor	0.050	1.7	5
Aldrin	0.050	1.7	5
Heptachlor epoxide	0.050	1.7	5
Endosulfan I	0.050	1.7	5
Dieldrin	0.10	3.3	10
4,4'–DDE	0.10	3.3	10

Endrin	0.10	3.3	10
Endosulfan II	0.10	3.3	10
4,4'–DDD	0.10	3.3	10
Endosulfan sulfate	0.10	3.3	10
4,4'–DDT	0.10	3.3	10
Methoxychlor	0.50	17	50
Endrin ketone	0.10	3.3	10
Endrin aldehyde	0.10	3.3	10
alpha–Chlordane	0.050	1.7	5
gamma–Chlordane	0.050	1.7	5
Toxaphene	5.0	170	500
Aroclor–1016	1.0	33	100
Aroclor–1221	2.0	67	200
Aroclor–1232	1.0	33	100
Aroclor–1242	1.0	33	100
Aroclor–1248	1.0	33	100
Aroclor–1254	1.0	33	100
Aroclor–1260	1.0	33	100

SAMPLE CONCENTRATION - Concentrations of single component pesticides and surrogates are calculated for both GC columns using the calibration factor from the initial calibration for the midpoint concentration of the external calibration standard in the following equations:

Sample concentrations for waters:

$$ug/L = \frac{(A_x)\ (DF)\ (V_t)\ (GPC)}{(CF)\ (V_o)\ (V_i)}$$

Sample concentration for soils:

$$ug/Kg = \frac{(A_x)\ (DF)\ (V_t)\ (GPC)}{(CF)\ (W_s)\ (D)\ (V_i)}$$

Where,

A_x = Area of peak for the analyte being measured.
CF = Calibration Factor for the midpoint concentration from the initial calibration (area per ng).
V_t = Volume of total extract (uL).
V_i = Volume injected (uL).
V_o = Volume of sample extracted (mL).
DF = Dilution Factor. The dilution factor for analysis of water and soil/sediment samples by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, DF = 1.
W_s = Weight of sample extracted.

D = $\frac{100 - \%\ Moisture}{100}$

GPC = GPC factor (If no GPC is performed, GPC = 1; if GPC is performed, then GPC = 2.0).

Adjusted CRQL = Non-adjusted CRQL x Sample Dilution Factor

Sample concentrations for waters:

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(V_x)\,(V_t)\,(V_y)\,(DF)}{(v_o)\,(V_c)\,(V_i)}$$

Where,

V_t, DF, V_o, and V_i are as given in the sample concentration equation above.

V_x = Contract sample volume (1000 mL).

V_y = Contract injection volume (1 uL or 2 uL).

V_c = Contract concentration extract volume (10,000 uL if GPC was not performed and 5,000 uL if GPC was performed).

Sample concentrations for soil/sediments:

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)\,(V_t)\,(V_y)\,(DF)}{(W_s)\,(V_c)\,(V_i)\,(D)}$$

Where,

V_t, DF, V_o, and V_i are as given in the sample concentration equation above.

W_x = Contract sample weight (30 g).

V_y = Contract injection volume (1 uL or 2 uL).

V_c = Contract concentrated extract volume (GPC is required: 5000 uL).

For example, the adjusted CRQL for a water sample with a 1.0U non-diluted CRQL and a 1 to 100 dilution (100 dilution factor) would be 100U, according to the following equation:

100U = 1.0U x 100

Adjusting for dry weight, the CRQL would be calculated as:

$$Dry\ Wt.\ CRQL = \frac{\dfrac{Non\text{-}adjusted\ Weight}{100 - \%\ Moisture}}{100}$$

For example, the dry weight CRQL for a soil sample with a 33U non-adjusted CRQL and 10% moisture would be 37U, according to the following equation:

$$37U = \frac{33U}{\dfrac{100 - 10}{100}}$$

The lower of the two concentrations calculated for each single component pesticide is reported on the tabulated Report Form I. For multicomponent analytes the lower mean concentration is reported. The percent difference between the two values is reported on Form X and calculated using the following equation:

PERCENT DIFFERENCE CALCULATION

$$\%\ D = \frac{C_h - C_l}{C_l} \times 100$$

Where,

C_h = The higher of the two concentrations for the target analyte in question.

C_l = The lower of the two concentrations for the target analyte in question.

SECTION XIV: SYSTEM PERFORMANCE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XIV-B for system performance data validation criteria.

SECTION XV: OVERALL ASSESSMENT CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XV-B for overall assessment data validation criteria.

APPENDIX G

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Organic Analysis, Low Concentration Water, OLC03.2 - 12/00, Exhibit F Pesticide/Aroclors**.

Capillary columns are mandatory. Packed columns cannot be used.

SECTION I: PRESERVATION & HOLDING TIME CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-I-B for preservation and holding time data validation criteria.

SECTION II: GC/ECD INSTRUMENT PERFORMANCE CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-II-B for GC/ECD Instrument Performance Check data validation criteria and the following method GC/ECD instrument performance QC criteria:

Resolution Check Mixture

A Resolution Check Mixture containing the following analytes must be analyzed at the beginning of the initial calibration sequence. The resolution between two adjacent peaks in the Resolution Check mixture must be greater than or equal to 60.0%.

Analyte	Concentration (ng/mL)
gamma-Chlordane	10.0
Endosulfan I	10.0
4,4'-DDE	20.0
Dieldrin	20.0
Endosulfan sulfate	20.0
Endrin ketone	20.0
Methoxychlor	100.0
Tetrachloro-m-xylene (surrogate)	20.0
Decachlorobiphenyl (surrogate)	20.0

Resolution Check - The % Resolution is calculated using the following equation:

$$\% \ Resolution = \frac{V}{H} \ x \ 100$$

Where,

V = Depth of valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.
H = Height of the shorter of the adjacent peaks.

Example

If:
Height of Peak A = 2560
Height of Peak B = 1435

The resolution (depth of the valley) between the two peaks must be at least 60.0% of the shorter peak or Peak B in this situation (0.6 x 1435 = 861). If the resolution between Peak A and Peak B is less than 861, the data will need to be qualified as indicated in Tables Pest/PCB-II-1 and Pest/PCB-II-2.

Performance Evaluation Mixture

A Performance Evaluation Mixture (PEM) containing the analytes listed below must be analyzed at the beginning of the initial calibration sequence, immediately after the Resolution Check Mixture and at the end of the initial calibration sequence. It must also be analyzed once during every 24 hours of the analytical sequence as part of the continuing calibration. The resolution between two adjacent peaks in the PEM must be greater than or equal to 90.0%. The percent breakdown for both DDT and Endrin in each PEM must be less than or equal to 20.0% for both GC columns. The combined percent breakdown for DDT and Endrin in each PEM must be less than or equal to 30.0% for both GC columns.

Analyte	Concentration (ng/mL)
gamma-BHC	10.0
alpha-BHC	10.0
4,4'-DDT	100.0
beta-BHC	10.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene (surrogate)	20.0
Decachlorobiphenyl (surrogate)	20.0

PERCENT DIFFERENCE (% D) - The % D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM runs of the initial calibration on each GC column must be less than or equal to ± 25.0%. The % D is calculated using the following equation:

$$\% \ Difference = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

Where,

C_{calc} = Calculated concentration of each analyte from the analyses of the calibration standard.
C_{nom} = Nominal concentration of each analyte.

4,4'-DDT/ENDRIN BREAKDOWN CALCULATIONS

$$\% \text{ Breakdown DDT} = \frac{\text{Amount found (ng) (DDD + DDE)}}{\text{Amount (ng) of DDT injected}} \times 100$$

$$\% \text{ Breakdown Endrin} = \frac{\text{Amount found (ng) (Endrin Aldehyde + Endrin Ketone)}}{\text{Amount (ng) of Endrin injected}} \times 100$$

$$\text{Combined \% Breakdown} = \text{\% Breakdown DDT} + \text{\% Breakdown Endrin}$$

SECTION III: INITIAL CALIBRATION CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

Each GC/ECD system must be initially calibrated using the following sequence.

INITIAL CALIBRATION SEQUENCE

1. Resolution Check
2. Performance Evaluation Mixture
3. Aroclor 1016/1260
4. Aroclor 1221
5. Aroclor 1232
6. Aroclor 1242
7. Aroclor 1248
8. Aroclor 1254
9. Toxaphene
10. Low Point Standard A
11. Low Point Standard B
12. Midpoint Standard A
13. Midpoint Standard B
14. High Point Standard A
15. High Point Standard B
16. Instrument Blank
17. Performance Evaluation Mixture

Table App.G.III-1 - ANALYTES CONTAINED IN INDIVIDUAL STANDARD MIXTURE A AND INDIVIDUAL STANDARD MIXTURE B

IND STD MIXTURE A	LOW POINT CONCENTRATION (ng/mL)
alpha-BHC	5.0
Heptachlor	5.0
gamma-BHC	5.0
Endosulfan I	5.0
Dieldrin	10.0

Endrin	10.0
4,4'-DDD	10.0
4,4'-DDT	10.0
Methoxychlor	50.0
Tetrachloro-m-xylene (surrogate)	5.0
Decachlorobiphenyl (surrogate)	10.0

IND STD MIXTURE B	LOW POINT CONCENTRATION (ng/mL)
beta-BHC	5.0
delta-BHC	5.0
Aldrin	5.0
Heptachlor epoxide*	5.0
alpha-Chlordane	5.0
gamma-Chlordane	5.0
4,4'-DDE	10.0
Endosulfan sulfate	10.0
Endrin aldehyde	10.0
Endrin ketone	10.0
Endosulfan II	10.0
Tetrachloro-m-xylene (surrogate)	5.0
Decachlorobiphenyl (surrogate)	10.0

*Note: Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is used as an analytical standard.

Table App.G.III-2 - MULTICOMPONENT ANALYTE LOW POINT CONCENTRATIONS

MULTICOMPONENT ANALYTES	LOW POINT CONCENTRATIONS (ng/mL)
Aroclor 1016	100
Aroclor 1221	200
Aroclor 1232	100
Aroclor 1242	100
Aroclor 1248	100
Aroclor 1254	100
Aroclor 1260	100
Toxaphene	500

Multicomponent standards including the Aroclors and Toxaphene must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture.

RETENTION TIME WINDOW CALCULATION

In the initial calibration the absolute retention times (RTs) are measured for all single component pesticides, the surrogates, and at least three major peaks of each multicomponent analyte. The mean RTs for single component standards and surrogates are calculated as the average of the three values and the RTs for multicomponent analytes are based on one value.

A retention time window for each single component pesticide and surrogate, and for the major peaks (3 to 5) of each multicomponent analyte are calculated using the values in Table App.G.III-3.

The mean absolute retention time ($\overline{RT}$) is calculated according to the following equation:

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where,

$\overline{RT}$ = Mean absolute retention time of analyte.
RT_i = Absolute retention time of analyte.
n = Number of measurements (3).

Example

The retention time window is calculated by first taking the mean of the retention times from the low, mid, and high concentration of the individual standards in the initial calibration. For example, the retention times for Endrin are:

Low - 9.86 minutes Mid - 9.85 minutes High - 9.86 minutes

Mean = 9.86 minutes

Since we know from Table App.G.III-3 that the retention time window for Endrin is ±0.07, we add and subtract 0.07 to and from the Mean to calculate the retention time window for Endrin from the initial calibration.

RT Window for Endrin = 9.79 - 9.93 minutes

Table App.G.III-3 - RETENTION TIME WINDOWS FOR ANALYTES AND SURROGATES

Analyte	Retention Time Window in Minutes
alpha-BHC	±0.05
beta-BHC	±0.05
gamma-BHC	±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin	±0.05
alpha-Chlordane	±0.07
gamma-Chlordane	±0.07

Analyte	Retention Time Window in Minutes
Heptachlor Epoxide	±0.07
Dieldrin	±0.07
Endrin	±0.07
Endrin Aldehyde	±0.07
Endrin Ketone	±0.07
DDD	±0.07
DDE	±0.07
DDT	±0.07
Endosulfan I	±0.07
Endosulfan II	±0.07
Endosulfan Sulfate	±0.07
Methoxychlor	±0.07
Aroclors	±0.07
Toxaphene	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

CALIBRATION FACTOR CALCULATION

In the initial calibration, peak areas or peak heights are measure to determine the analyte calibration factor (CF). The calibration factor for each single component pesticide and surrogate and for each peak in the selected set of 3 to 5 major peaks for each multicomponent analyte is calculated using equation III-1. The mean CF for each single component pesticide and surrogate is calculated using equation III-2. **Note: The single component pesticide mean CF is only used in calculating the % RSD and <u>not</u> for quantifying sample results.**

Equation III-1:

$$CF = \frac{\text{Peak Area (height) of the standard}}{\text{Mass injected (ng)}}$$

Equation III-2:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where,

CF_i = Calibration factor.

$\overline{CF}$ = Mean calibration factor.
n = Total number of values (3).

% RSD CALCULATION

Initial calibration data is used to assess the linearity of each GC/ECD system used for sample analysis. The linearity of the GC is assessed by calculating a % RSD of the calibration factors for each single component pesticide and surrogate using equation III-3.

Equation III-3:

The % RSD is calculated using the following equation:

$$\% \ RSD = \frac{SD_{CF}}{\overline{CF}} \times 100$$

Where,

$$SD_{CF} = \sqrt{\sum_{i=1}^{n} \frac{(CF_i - \overline{CF})^2}{(n-1)}}$$

% RSD = Percent relative standard deviation.
SD_{CF} = Standard deviation of calibration factors.
CF_i = Each individual Calibration Factor used to calculate the mean.

$\overline{CF}$ = Mean calibration factor.
n = Total number of values (3).

SECTION IV: CONTINUING CALIBRATION CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IV-B for continuing calibration data validation criteria and the following method continuing calibration QC criteria:

The analyses of the instrument blanks, Performance Evaluation Mixtures (PEM), and the midpoint concentration of Individual Standard Mixtures A and B constitute the continuing calibration. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B. A valid analysis sequence is given below:

1. Sequence

Time	Injection #	Material Injected
	1 - 15	First 15 steps of the Initial Calibration
0 hr.	16	Instrument Blank* at the end of the Initial Calibration
	17	PEM at the end of the Initial Calibration
	18	First Sample
	o	
	o	Subsequent Samples
	o	
12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd injection past 12:00 hr.	Performance Evaluation Mixture
	o	Sample
	o	
	o	Subsequent Samples
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	etc.	

*The instrument blank contains only the surrogate analytes: Tetrachloro-m-xylene and Decachlorobiphenyl.

PERCENT DIFFERENCE (%D) - The %D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM and Individual Mixture runs of the continuing calibration on each GC column must be less than or equal to $\pm$ 25.0%. The %D is calculated using the following equation:

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

Where,

C_{calc} = Calculated concentration of each analyte from the analyses of the standard.
C_{nom} = Nominal concentration of each analyte.

Example

The nominal amount of gamma-BHC added to the PEM is 10 ng/mL. The calculated amount of gamma-BHC is found to be 12 ng/mL. Using the above equation, the percent difference is calculated as follows:

$$\%D = \frac{12 - 10}{10} \times 100 = 20\%$$

Example

The nominal amount of 4,4'-DDT added to the INDA is 10 ng/mL. The calculated amount of 4,4'-DDT is found to be 9.0 ng/mL. Using the above equation, the percent difference is calculated as follows:

$$\%D = \frac{9.0 - 10}{10} \times 100 = -10\%$$

Example

The nominal amount of Aldrin added to the INDB is 5.0 ng/mL. The calculated amount of Aldrin is found to be 7.0 ng/mL. Using the above equation, the percent difference is calculated as follows:

$$\%D = \frac{7.0 - 5.0}{5.0} \times 100 = 40\%$$

SECTION V: BLANK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-V-B for blank data validation criteria and the following method QC criteria.

Method Required Blanks

1. Method Blank - A volume of reagent water or purified solid matrix, approximate in weight or volume to the samples, which is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of the entire set of samples. Surrogate analytes must recover between 30-150%. A method blank must be extracted and analyzed once per each SDG, or each extraction procedure within an SDG, or whenever samples are extracted, whichever is most frequent.

2. Sulfur Cleanup The sulfur cleanup blank is a modified form of the method blank. It is a volume of clean solvent spiked with the surrogates and carried through the sulfur cleanup and analysis

procedures. This blank is used to determine the levels of contamination associated with the separate sulfur cleanup steps. Surrogate analytes must recover between 30-150%. The sulfur cleanup blank is prepared separately when only part of a set of samples extracted together requires sulfur removal. If all of the samples associated with a given method blank require sulfur cleanup, then the method blank must be subjected to sulfur cleanup and no separate sulfur cleanup blank is required.

3. Instrument Blank - The instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrument analysis itself, such as the carry-over of analytes from standard or highly contaminated samples into other analyses. An instrument blank must be analyzed after a sample whose concentration exceeds the calibration range. Until an instrument blank meets the technical acceptance criteria, the system is considered contaminated.

   A GPC blank, a type of instrument blank, is analyzed after the initial calibration of GPC. It is not spiked with surrogate analytes.

SECTION VI: SURROGATE ANALYTE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VI-B for surrogate analyte data validation criteria and the following method surrogate analyte QC criteria:

Table App.G.VI-1 - SURROGATE RETENTION TIME WINDOWS

Surrogate	Retention Time Window in Minutes
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

The % surrogate recovery is calculated using the following equation:

$$\text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of surrogate determined by analysis.

Q_a = Quantity of surrogate added to sample/blank.

Table App.G.VI-2 - SURROGATE SPIKE RECOVERIES

Surrogate	Percent Recovery (Water)
Tetrachloro-m-xylene	30-150
Decachlorobiphenyl	30-150

*Pesticide surrogate analyte recoveries are mandatory for samples and blanks in the Low Concentration method.

SECTION VII: PESTICIDE CLEANUP CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VII-B for pesticide cleanup data validation criteria and the following method pesticide cleanup QC criteria:

Florisil Cartridge

The Florisil Cartridge Performance Check consists of testing every lot of Florisil prior to use by analyzing the following two solutions in a mixture.

1. The Florisil Cartridge Check Solution contains 2,4,5-trichlorophenol in acetone, at a concentration of 0.10 ug/mL.

2. The Pesticide Spiking Solution contains the following Standard Mixture A analytes at the midpoint concentration:

alpha-BHC	4,4'-DDD
heptachlor	4,4'-DDT
gamma-BHC	methoxychlor
endosulfan I	tetrachloro-m-xylene
dieldrin	decachlorobiphenyl
endrin	

Table App.G.VII-1 - FLORISIL CARTRIDGE PERFORMANCE CHECK CRITERIA

INDA Analyte % Recovery	80% - 120%
2,4,5-TCP Recovery	2,4,5-Trichlorophenol must recover at < 5.0%
Other Target Analytes	No interfering peaks with the target analytes

Sulfur Cleanup

Sulfur cleanup must be performed on all extracts containing sulfur at levels that interfere with GC/ECD analysis. Sulfur contamination in a sample analysis is unacceptable. A sulfur cleanup blank is required even if only a part of a set of samples requires sulfur cleanup.

1. Sulfur cleanup can be performed using two techniques: the Mercury Technique or the Copper Technique.

2. The method blank must be subjected to the same cleanup procedure as the samples.

SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VIII-B for MS/MSD data validation criteria and the following method MS/MSD QC criteria:

A matrix spike and matrix spike duplicate must be performed if requested on the traffic report, at a frequency of each group of 20 field samples in an SDG, or each SDG, whichever is more frequent.

The following advisory matrix spike analyte recoveries and RPDs are listed below:

Table App.G.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Method QC Criteria	
	% Recovery*	RPD**
gamma-BHC (Lindane)	56-123	15
Heptachlor	40-131	20
Aldrin	40-120	22
Dieldrin	52-126	18
Endrin	56-121	21
4,4'-DDT	38-127	27

*The MS/MSD % recovery is calculated using the following equation:

$$Matrix\ Spike\ Recovery = \frac{SSR - SR}{SA} X\ 100$$

Where,

SSR = Spike Sample Result.
SR = Sample Result.
SA = Spike Added.

**The MS/MSD relative percent difference (RPD) is calculated using the following equation:

$$\text{Relative Percent Difference} = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)} \times 100$$

Where,

MSR = Matrix Spike Recovery.
MSDR = Matrix Spike Duplicate Recovery.

Note: The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always positive.

SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IX-B for field duplicate data validation criteria.

SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-X-B for sensitivity check data validation criteria.

SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XI-B for accuracy check data validation criteria and the following method accuracy check QC criteria:

The Laboratory Control Sample is a method required internal laboratory quality control sample that must be prepared, analyzed, and reported once per SDG. It must be prepared and analyzed concurrently with the samples in the SDG using the same instrumentation as the samples.

Table App.G.XI-1 - LCS % RECOVERY LIMITS

Analyte	Method Required QC % Recovery Limits for LCS (Water)
gamma-BHC (Lindane)	50-120
Heptachlor epoxide	50-150
4,4'-DDE	50-150
Dieldrin	30-130
Endrin	50-120

Endosulfan sulfate	50-120
gamma-Chlordane	30-130

PERCENT RECOVERY CALCULATIONS

The LCS % recovery is calculated using the following equation:

$$LCS\ Percent\ Recovery = \frac{Q_d}{Q_a} \times 100\%$$

Q_d = Quantity of LCS determined by analysis.

Q_a = Quantity of LCS added to the blank.

SECTION XII: TARGET ANALYTE IDENTIFICATION CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XII-B for target analyte identification data validation criteria.

SECTION XIII: ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMIT CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XIII-B for analyte quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Pesticide/PCB analytes must be quantified using the external standard method. The midpoint Calibration Factor from the most recent compliant initial calibration must be used to quantify sample single component pesticides on both columns and the lowest sample concentration must be reported. For multicomponent analytes the calibration factors for three to five major peaks from the most recent compliant initial calibration are each used to quantify the multicomponent analyte in the sample. The three to five concentrations are then averaged and then a mean concentration for the sample analyte is determined on each column. The lowest mean concentration is reported. Pesticide/PCBs must be reported to the CRQLs listed below:

Table App.G.XIII-1 - TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

		Quantitation Limits
		Water
Pesticides/Aroclors	CAS Number	ug/L
alpha–BHC	319–84–6	0.01
beta–BHC	319–85–7	0.01
delta–BHC	319–86–8	0.01
gamma–BHC	58–89–9	0.01
Heptachlor	76–44–8	0.01

Aldrin	309–00–2	0.01
Heptachlor epoxide	1024–57–3	0.01
Endosulfan I	959–98–8	0.01
Dieldrin	60–57–1	0.02
4,4'–DDE	72–55–9	0.02
Endrin	72–20–8	0.02
Endosulfan II	33213–65–9	0.02
4,4'–DDD	72–54–8	0.02
Endosulfan sulfate	1031–07–8	0.02
4,4'–DDT	50–29–3	0.02
Methoxychlor	72–43–5	0.10
Endrin ketone	53494–70–5	0.02
Endrin aldehyde	7421-93-4	0.02
alpha–Chlordane	5103–71–9	0.01
gamma–Chlordane	5103–74–2	0.01
Toxaphene	8001–35–2	1.0
Aroclor–1016	12674–11–2	0.2
Aroclor–1221	11104–28–2	0.4
Aroclor–1232	11141–16–5	0.2
Aroclor–1242	53469–21–9	0.2
Aroclor–1248	12672–29–6	0.2
Aroclor–1254	11097–69–1	0.2
Aroclor–1260	11096–82–5	0.2

SAMPLE CONCENTRATION - Concentrations of single component pesticides and surrogates are calculated for both GC columns using the CF from the initial calibration for the midpoint concentration of the external calibration standard in the following equations:

Sample concentrations for waters:

$$ug/L = \frac{(A_x)\ (DF)\ (V_t)}{(CF)\ (V_i)\ (V_x)}$$

Where,

A_x = Area of peak for the analyte being measured.
CF = Calibration Factor for the midpoint concentration from the initial calibration (area per ng).
V_t = Volume of total extract in microliters (uL).
V_i = Volume of extract injected in microliters (uL).
V_x = Volume of sample extracted in milliliters (mL).
DF = Dilution Factor - The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, DF = 1.

Adjusted CRQL = Non-adjusted CRQL x Sample Dilution Factor

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{\text{Contract Sample Vol (1000mL)}}{V_x} \times DF \times \frac{V_t}{\text{Contract Ext. Vol (2000uL)}}$$

Where,

V_t = Volume of total extract in microliters (2000 uL).
DF = Dilution Factor.
V_x = Volume of water extracted in milliliters (mL).

For example, the adjusted CRQL for a water sample with a 1.0U non-diluted CRQL and a 1 to 100 dilution (100 dilution factor) would be 100U, according to the following equation:

100U = 1.0U x 100

PERCENT DIFFERENCE CALCULATION

The lower of the two concentrations calculated for each single component pesticide is reported on the tabulated report Form I. For multicomponent analytes the lower mean concentration is reported. The percent difference between the two values is reported on Form X and calculated using the following equation:

$$\%D = \frac{C_h - C_l}{C_l} \times 100$$

Where,

C_h = The higher of the two concentrations for the target analyte in question.

C_l = The lower of the two concentrations for the target analyte in question.

SECTION XIV: SYSTEM PERFORMANCE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XIV-B for system performance data validation criteria.

SECTION XV: OVERALL ASSESSMENT CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XV-B for overall assessment data validation criteria.

Table Pest/PCB-I-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION		TECHNICAL HOLDING TIMES	
Matrix	Refrig.& Light Protected	Extracted and/or Analyzed Within H.T.	Extracted and/or Analyzed Outside H.T.
AQ and S/S	Yes	A - acceptable results	J - detects UJ - non-detects R* - non-detects if Extraction HT>28 days and/or Analytical HT>60 days
AQ and S/S	No	J - detects UJ - non-detects	J - detects UJ - non-detects R* - non-detects if Extraction HT>28 days and/or Analytical HT>60 days

Note: AQ = Aqueous, S/S = Soil/Sediment

* The validator may use professional judgment to qualify or reject non-detected pesticides and multicomponent analytes based on their environmental stability.

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify (UJ) or reject (R) non-detects when Region I preservation and/or technical holding time criteria are not met. The results are acceptable (A) when the criteria are met.

APPENDIX H

Table Pest/PCB-II-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON THE RESOLUTION CHECK MIXTURE (RCM) - Resolution Check

Sample Results	Resolution ≥ 60.0%	Resolution < 60.0%
Detects	A	Professional judgment
Non-Detects	A	Professional judgment

Table Pest/PCB-II-2:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON THE PERFORMANCE EVALUATION MIXTURE (PEM) - Resolution Check

Sample Results	Resolution ≥ 90.0%	Resolution < 90.0%
Detects	A	J
Non-detects	A	Professional judgment

Table Pest/PCB-II-3:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON THE PERFORMANCE EVALUATION MIXTURE (PEM) - CALIBRATION CHECK - Accuracy Check

Sample Results	%D ≤ ±25.0%	%D > ±25.0%	One column meets criteria but the other exceeds
Detects	A	J	Professional judgement
Non-Detects	A	UJ	Professional judgement

Table Pest/PCB-II-4:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON 4,4'-DDT/ENDRIN BREAKDOWN - PESTICIDE DEGRADATION CHECK

Sample Results	4,4'-DDT Breakdown ≤ 20.0%	4,4'-DDT Breakdown > 20.0% and 4,4'-DDT detected	4,4'-DDT Breakdown > 20.0% and 4,4'-DDT not detected	Endrin Breakdown ≤ 20.0%	Endrin Breakdown > 20.0% and Endrin detected	Endrin Breakdown > 20.0% and Endrin not detected	Combined Breakdown ≤ 30.0%	Combined Breakdown > 30.0% and 4,4'-DDT and/or Endrin detected	Combined Breakdown > 30.0% and 4,4'-DDT and/or Endrin not detected
4,4'-DDT	A	J	R (NDs)	N/A	N/A	N/A	A	J	R (NDs)
DDD	A	J (detects) A (NDs)	J (detects) A (NDs)	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)
DDE	A	J (detects) A (NDs)	J (detects) A (NDs)	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)
Endrin	N/A	N/A	N/A	A	J	R (NDs)	A	J	R (NDs)
Endrin Aldehyde	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)	A	J (detects) A (NDs)	J (detects) A (NDs)
Endrin Ketone	N/A	N/A	N/A	A	J (detects) A (NDs)	J (detects) A (NDs)	A	J (detects) A (NDs)	J (detects) A (NDs)

N/A = Not Applicable
J = Estimate result
R (NDs) = Reject non-detects
A (NDs) = Accept non-detects

Note: The validator must always discuss negative and positive bias in sample data in the Data Validation Memorandum.

Table Pest/PCB-III-1:

QUALIFICATION OF PESTICIDE/MULTICOMPONENT* ANALYTES BASED ON THE INITIAL CALIBRATION

Sample Results	%RSD ≤ 20.0% (alpha-BHC & delta-BHC %RSD ≤ 25.0%)	%RSD > 20.0% (alpha-BHC & delta-BHC %RSD > 25.0%)	If applicable, multicomponent analyte %RSD ≤ 25.0%	If applicable, multicomponent analyte %RSD > 25.0%	One column meets criteria but the other exceeds
Detects	A	J	A	J	Professional judgement
Non-detects	A	UJ	A	UJ	Professional judgement

* OLM04.3 does not require analysis of more than one initial calibration standard concentration for multicomponent analytes.

Table Pest/PCB-IV-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON CALIBRATION VERIFICATION

Sample Results	%D ≤ ±25.0%	%D > ±25.0%	One column meets criteria but the other exceeds
Detects	A	J	Professional judgement
Non-detects	A	UJ	Professional judgement

Table Pest/PCB-VI-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON SURROGATE ANALYTE RECOVERIES

Surrogate Analyte Recovery				
Sample Results	One or more surrogates %Rec < 10%	One or more surrogates 10% ≤ %Rec < LL	All surrogates LL ≤ %Rec ≤ UL	One or more surrogates %Rec > UL
Detects	J	Professional Judgment	A	Professional Judgment
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Note: The surrogate recoveries in the method blank and the instrument blank must be within criteria for the analytical sequence to be valid.

Table Pest/PCB-VII-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON GPC CALIBRATION QUALITY CONTROL

	Criteria	Action
Peak Resolution	As per method QC acceptance criteria.	Professional Judgment
Peak Shape	Peak shapes must be symmetrical.	Professional Judgment
Aroclor Pattern	After GPC is performed, Aroclor 1016 and 1260 standard patterns must be similar to Aroclor patterns in the Initial Calibration sequence.	Professional Judgment
Retention Time	Retention time shifts between GPC calibrations for bis(2-ethylhexyl)phthalate and perylene must not exceed ± 5%.	Professional Judgment
GPC Instrument Blank	Target analytes must be less than the quantitation limit.	Refer to Section V for Blank Actions

Table Pest/PCB-VII-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON GPC CALIBRATION VERIFICATION QUALITY CONTROL

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < Lower Limit	Lower Limit ≤ %Rec ≤ Upper Limit	%Rec > Upper Limit
Detects	J	J	A	J
Non-detects	R	UJ	A	A

Note: Professional judgment should be used in applying the guidance above to qualify or reject sample data.

Table Pest/PCB-VII-3:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON FLORISIL CARTRIDGE CLEANUP QUALITY CONTROL

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < Lower Limit	Lower Limit ≤ %Rec ≤ Upper Limit	%Rec > Upper Limit
Detects	J	J	A	J
Non-detects	R	UJ	A	A
2,4,5-TCP Recovery Criterion	If 2,4,5-Trichlorophenol recovers at ≥ 5%, then the Florisil is not working properly and the data must be evaluated for potential interferences.			

Note: Professional judgment should be used to qualify the data when a combination of low recoveries and high recoveries are obtained.

Table Pest/PCB-VII-4:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON SULFUR CLEANUP QUALITY CONTROL

Sample Results	Degree of Sulfur Interference		
	Minor	Limited to discrete part of the sample chromatogram	Major
Detects	Estimate (J) positive detects for the affected analytes.	Accept positive detects that are not impacted by sulfur interference. Reject (R) positive detects for those analytes coeluting with the sulfur peak.	Reject (R) all detects for the affected sample and request sample reanalysis that includes sulfur cleanup.
Non-detects	Use professional judgement to evaluate the non-detects.	Accept non-detects that are not impacted by sulfur interference. Reject (R) non-detects for those analytes coeluting with the sulfur peak.	Reject (R) all non-detects for the affected sample and request sample reanalysis that includes sulfur cleanup.

Note: Professional judgment should be used in applying the above guidance to qualify or reject sample data.

Table Pest/PCB-VIII-1:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MATRIX SPIKE RECOVERIES AND RPDS **

Sample Results	Recovery < 10%	10% ≤ Recovery < Lower QC Limit	Lower QC Limit ≤ Recovery ≤ Upper QC Limit	Recovery > Upper QC Limit	RPD > QC Limit
Detects	J	J	A	J	J
Non-detects	R	UJ	A	A	UJ

** Note that qualification and rejection generally are limited to the spiking analytes, however, the validator may use professional judgment to qualify or reject all positive detects or non-detects in the unspiked sample, or even all results of a particular matrix, if the majority of spike analyte recoveries and/or RPDs are outside the method QC acceptance criteria.

Table Pest/PCB-VIII-2:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MS, MSD, AND UNSPIKED SAMPLE %RSD

Sample Results	%RSD ≤ 50%*	%RSD > 50%*	Two out of three sample results reported as non-detects
Detects	A	J	Professional Judgment
Non-detects	A	Professional Judgment	Professional Judgment

* If a non-detect is reported for an analyte in only one of the samples in the MS, MSD, or unspiked sample set, then the validator should use the sample quantitation limit value for that analyte to calculate the %RSD.

Table Pest/PCB-IX-1:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES
SITUATION 1: POSITIVE DETECTS IN BOTH FIELD DUPLICATES

Relative Percent Difference	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30 Non-Aqueous > 50%
Sample Results	**Both duplicate sample concs. ≥ 2 X QL**	**QL ≤ both duplicate samples concs. < 2 X QL**	**One sample conc. ≥ 2 X QL QL ≤ One sample conc. < 2 X QL**
Detects	J	Professional Judgment	Professional Judgment
Non-detects	N/A	N/A	Professional Judgment

* QL = Sample Quantitation Limit
N/A = Not Applicable

Note: Qualification refers to field duplicate sample results only. Professional judgment may be used to apply field duplicate actions to all samples of the same matrix.

Table Pest/PCB-IX-2:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES
SITUATION 2: POSITIVE DETECT IN ONLY ONE FIELD DUPLICATE**

Aqueous and Non-Aqueous		
Sample Results	**One Sample Conc. = ND (or values reported as less than the QL) QL ≤ One Sample Conc. < 2 X QL**	**One sample conc. = ND (or values reported as less than the QL) One sample conc. ≥ 2 X QL**
Detects	Professional Judgment	J
Non-detects	Professional Judgment	UJ

* QL = Sample Quantitation Limit

** RPD should not be evaluated for these duplicate pairs

Note: Qualification refers to field duplicate sample results only. Professional judgment may be used to apply field duplicate actions to all samples of the same matrix.

Table Pest/PCB-X-1:

QUALIFICATION OF ORGANIC ANALYTES BASED ON MDL STUDY RESULTS

Sample Results	Mean % Recovery			
	%Rec < 10%	10% ≤ %Rec < 80%	80% ≤ %Rec ≤ 120%	%Rec > 120%
Detects	J	Professional Judgment*	A	Professional Judgment*
Non-Detects	R	Professional Judgment*	A	A
Sample Results	**% RSD**			
	> 25%		≤ 25%	
Detects	Professional Judgment**		A	
Non-detects	Professional Judgment**		A	

* Taking into consideration LFB results.
** Taking into consideration initial calibration %RSDs.

Table Pest/PCB-X-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE:
≤ ONE-HALF OF LFB ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target analytes and/or Aroclors at or below the quantitation limit.

Table Pest/PCB-X-3:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE:
> ONE-HALF OF LFB ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target analytes and/or Aroclors at or below the quantitation limit.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table Pest/PCB-XI-1:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LCS* RECOVERIES WHERE:
≤ ONE-HALF OF LCS ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LCS = Laboratory Control Sample (LCS) is an internal laboratory quality control sample designed to assess analytical accuracy and method bias. The LCS is spiked with several or all of the method target analytes and /or Aroclors.

LL - Lower limit of method QC acceptance criteria
UL - Upper limit of method QC acceptance criteria

Table Pest/PCB-XI-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LCS* RECOVERIES WHERE:
> ONE-HALF OF LCS ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < LL	LL ≤ %Rec ≤ UL	%Rec > UL
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* LCS = Laboratory Control Sample (LCS) is an internal laboratory quality control sample designed to assess analytical accuracy and method bias. The LCS is spiked with several or all of the method target analytes and /or Aroclors.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

LL - Lower limit of method QC acceptance criteria
UL - Upper limit of method QC acceptance criteria

Table Pest/PCB-XI-3:

QUALIFICATION OF INDIVIDUAL ORGANIC ANALYTES BASED ON PES RESULTS WHERE:
≤ ONE-HALF OF PES ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	●Single Blind ●Double Blind PES < Lower Limit "Action Low"	●Single Blind ●Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	●Single Blind ●Double Blind PES > Upper Limit "Action High"
Detects	J	A	J
Non-Detects	R	A	A

Table Pest/PCB-XI-4:

QUALIFICATION OF ORGANIC ANALYTES BASED ON PES RESULTS WHERE:
> ONE-HALF OF PES ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS *

Sample Results	●Single Blind ●Double Blind PES < Lower Limit "Action Low"	●Single Blind ●Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	●Single Blind ●Double Blind PES > Upper Limit "Action High"
All Detects	J	A	J
All Non-Detects	R	A	A

* Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table Pest/PCB-XIII-1:

QUALIFICATION OF PESTICIDE/PCB ORGANIC ANALYTES BASED ON SAMPLE PERCENT SOLIDS

Sample Result	% Solids > 30%	10% ≤ % Solids ≤ 30%	% Solids < 10%
Detects	A	J	R
Non-detects	A	R	R

Table Pest/PCB-XIII-2:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON %D OF ANALYTES BETWEEN TWO QUANTITATION COLUMNS

%D Between the Two Quantitation Columns	Sample Results	
	Detects	Non-Detects
25.0% < %D < 100% (single component pesticides)	J	N/A*
%D > 100% (single component pesticides)	R	N/A
25.0% < %D < 500% (Multicomponents)	J	N/A
%D > 500% (Multicomponents)	R	N/A
One Value < QL & One Value ≥ QL	J	UJ

* N/A - Not Applicable

PART IV

Inorganic Data Validation Functional Guidelines

November 2008

INORGANIC DATA VALIDATION FUNCTIONAL GUIDELINES - PART IV

The requirements to be checked in validation are listed below. "CCS" indicates that some of the contractual requirements for these items will also be checked by Contract Compliance Screening (CCS). CCS requirements are not always the same as data validation criteria.

I. Preservation and Technical Holding Times ..... (CCS) INORG-I-1
II. ICP-MS Tune ..... (CCS) INORG-II-1
III. Calibrations ..... (CCS) INORG-III-1
IV. Blanks ..... (CCS) INORG-IV-1
V. ICP-AES Interference Check Sample (ICS) ..... (CCS) INORG-V-1
VI. ICP-MS Interference Check Sample (ICS) ..... (CCS) INORG-VI-1
VII. ICP-MS Internal Standards ..... (CCS) INORG-VII-1
VIII. Matrix Spikes ..... (CCS) INORG-VIII-1
IX. Laboratory Duplicate Samples ..... (CCS) INORG-IX-1
X. Field Duplicates INORG-X-1
XI. ICP Serial Dilutions ..... (CCS) INORG-XI-1
XII. Sensitivity Check ..... (CCS) INORG-XII-1
XIII. Performance Evaluation Samples/Accuracy Check ..... (CCS) INORG-XIII-1
XIV. Analyte Quantitation and Reported Quantitation Limits INORG-XIV-1
XV. System Performance INORG-XV-1
XVI. Overall Evaluation of Data INORG-XVI-1

Appendices

Appendix I CLP SOW ILM05.4/Inorganic Analysis
Appendix J Inorganic Functional Guidelines Action Tables
Appendix K Inorganic Data Validation Worksheets

I. PRESERVATION AND TECHNICAL HOLDING TIMES

A. OBJECTIVE

The objective is to ascertain the validity of the analytical results based on the preservation techniques which were used and the holding time of the sample from time of collection to time of sample preparation and sample analysis.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. REGION I PRESERVATION CRITERIA

SAMPLE TYPE	PRESERVATION CODE
Metals Aqueous [a]	2
Cyanide Aqueous [a]	1, 3
Metals (no Hg) Soil/Sediment/Sludge/Oily Waste/Wipe/Ash [b]	4
Mercury Soil/Sediment/Sludge/Oily Waste/Ash [b]	1
Cyanide Soil/Sediment/Sludge/Oily Waste [b]	1
Metals Biological Tissue [c]	5
Metals (no Hg) Air Filters [d]	4

Preservation Code:
1. Cool ($\leq 6°C$)
2. Preserve with HNO_3 to pH less than 2
3. Preserve with NaOH to pH more than 12; add reducing agent in the presence of oxidants (e.g., chlorine); remove sulfides as required by the method
4. Room temperature
5. Freeze

REFERENCES

a. 40 CFR, Part 136, Appendix C
b. SW-846: Chapter 3; 3000, 6000, 7000, 9000 Series
c. Evaluation of Dredged Material for Discharge in Waters of the U.S. - Testing Manual, EPA 823-B-97-001, February 1997, and QA/QC Guidance for Sampling and Analysis of Sediments, Waters, and Tissue for Dredged Material Evaluations, Chemical Evaluation, EPA 823-B-95-001, April 1995
d. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1, EPA/625/R-96/010a, June 1999.

2. REGION I TECHNICAL HOLDING TIME CRITERIA

PARAMETER*	CRITERIA
Metals (except mercury)	Properly preserved samples must be analyzed within 6 months of sample collection.
Mercury[a]	Properly preserved samples must be analyzed within 28 days of sample collection.
Cyanide	Properly preserved samples must be analyzed within 14 days of sample collection.

* See Section B.1 above for applicable sample types.
[a] Mercury by cold vapor AA.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from preservation and/or holding time anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Preservation a. Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form), laboratory tracking/storage forms, sample preparation records, and the data package narrative to verify that samples were properly preserved and maintained by the sampler and/or the laboratory according to Region I preservation criteria. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information.	1. Preservation a. If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume that the samples were not preserved and should document on the preservation and holding times worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify, or reject the sample data.

C. EVALUATION	D. ACTION
1. a. Continued from above.	1. a. Continued from above. If the samples were not preserved properly in the field and/or if the laboratory failed to properly preserve or maintain sample preservation, then the validator should take the following actions:
i. Verify that aqueous metals samples were preserved with HNO_3 according to Region I preservation criteria.	i. If data package documentation does not list the pH of each aqueous metals sample, then the validator should contact the laboratory to obtain any omitted information. - If aqueous metals samples were not preserved with HNO_3 according to Region I preservation criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples. Professional judgment should be used to reject (R) non-detects based on the pH of the sample and chemistry of the analytes of interest. - If the sample pH was adjusted with HNO_3 according to Region I preservation criteria upon laboratory receipt of samples for metals which either were not acid-preserved in the field or were received at an elevated pH (e.g., due to high sample alkalinity), then the validator may use professional judgment to accept the positive detects and non-detects for that sample. The validator should take into consideration the length of time that the acid resided in the sample prior to sample preparation and the analyte's stability. The acid should be in the sample at least 24 hours prior to sample preparation.

C. EVALUATION	D. ACTION
1. a. ii. Verify that aqueous cyanide samples were preserved with NaOH according to Region I preservation criteria.	1. a. ii. If data package documentation does not list the pH of each aqueous cyanide sample, then the validator should contact the laboratory to obtain any omitted information. If aqueous cyanide samples were not preserved with NaOH according to Region preservation criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples. Professional judgment should be used to reject (R) non-detects based on the sample pH and chemistry.
iii. Verify that aqueous cyanide samples were tested and treated, if needed, for oxidizing agents (e.g., chlorine) and sulfides, according to method requirements.	iii. If field or data package documentation does not indicate that a check for the presence of oxidants or sulfides was performed, or that samples were not treated for these interferences, then the validator should contact the sampler and/or laboratory to obtain any omitted information. Professional judgment should be used if a check was not performed or if samples were not treated in the presence of oxidants or sulfides. In this case, the validator should document in the Data Validation Memorandum all justifications for qualifying or not qualifying data, taking into consideration all available information about the sample matrix constituents, including any historical information that may exist for the site.

C. EVALUATION	D. ACTION
1. a. iv. Verify that inorganic samples were refrigerated or frozen (as required) according to Region I preservation criteria.	1. a. iv. For all matrices, if the Region I temperature preservation criteria were not met, then the validator should use professional judgment to accept, qualify, or reject the positive detects and non-detects for the affected samples. The validator should document all justifications for qualifying or not qualifying sample data in the Data Validation Memorandum. Professional judgment should be used when the laboratory has reported transportation cooler temperatures that slightly exceed the upper limits of the preservation criteria (> 6°C). In this case, the laboratory procedure for monitoring cooler temperature may be in question. In this event, all justifications for qualifying or not qualifying sample data should be documented in the Data Validation Memorandum.
2. Technical Holding Times a. Verify that inorganic samples were analyzed within the technical holding time criteria. Establish technical holding times by comparing sampling dates reported on the EPA Traffic Report and/or COC Forms with dates of analysis reported on tabulated result forms. • Verify that aqueous and soil/sediment metals (excluding mercury) samples were analyzed within 6 months of sample collection.	2. Technical Holding Times a. If aqueous and soil/sediment metals and cyanide samples were not analyzed within the technical analytical holding time criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects. Professional judgment should be used to reject (R) non-detects based on the magnitude of the holding time exceedance and the stability of the analyte.

C. EVALUATION	D. ACTION
2. a. Continued from above. • Verify that aqueous and soil/sediment mercury samples were analyzed within 28 days of sample collection. • Verify that aqueous and soil/sediment cyanide samples were analyzed within 14 days of sample collection. Note: Due to limited information concerning holding times for non-aqueous matrices, the holding times for water matrices should be applied to non-aqueous matrices.	2. a. Continued from above. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to estimate (UJ) or reject (R) non-detects when technical holding time criteria are exceeded. For all matrices, if analytical technical holding time criteria were grossly exceeded, then the validator should use professional judgment to estimate (J) positive detects and estimate (UJ) or reject (R) non-detects, taking into consideration the analyte's stability and the effects of additional storage on the sample results. The validator should use professional judgment to determine the reliability of the data.
* b. Check the raw data including digestion/distillation logs and instrument run logs to verify reported sample digestion/distillation and analysis dates.	b. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.2.b

Table INORG-I-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON PRESERVATION

Sample Results	Aq. Metals: HNO_3 to pH < 2? Aq. Cyanide[1]: NaOH to pH > 12?		Temperature Criteria Met?	
	Y	N	Y	N
Detects	A	J	A	Professional Judgment
Non-detects	A	UJ or R*	A	Professional Judgment

* Professional judgment may be used to estimate (UJ) or reject (R) non-detects based on the analyte's stability and magnitude of exceedance.

[1] Estimate (J) positive detects and use professional judgment to estimate (UJ) or reject (R) non-detects when cyanide samples are not tested nor treated, if needed, for oxidants or sulfides.

Table INORG-I-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON TECHNICAL HOLDING TIMES

Sample Results	Technical Holding Time (Aqueous/Soil/Sediment)	
	Metals: HT ≤ 6 Months	**Metals: HT > 6 Months**
	Mercury: HT ≤ 28 Days	**Mercury: HT > 28 Days**
	Cyanide: HT ≤ 14 Days	**Cyanide: HT > 14 Days**
Detects	A	J
Non-detects	A	UJ or R*

* Professional judgment may be used to estimate or reject non-detects based on the analyte's stability, magnitude of exceedance, and the effects of additional storage on the sample results.

- For other matrices, estimate (J) positive detects and use professional judgment to estimate (UJ) or reject (R) non-detects when Region I technical holding time criteria are not met.

E. EXAMPLES

Example #1: (Proper preservation; Analysis holding time exceeded)

Mercury soil sample MADD09 was sampled on 3/1/07 and was received at the laboratory on 3/2/07. Upon examination of the Traffic Report and the laboratory sample receipt and tracking information, the validator determines that the sample was shipped and stored at 4°C. As noted in the data package narrative, due to a laboratory tracking error, the laboratory analyzed the sample on 3/31/07, 30 days from the sampling date. The validator estimates (J) the positive detects and estimates (UJ) the non-detects for mercury in sample MADD09 on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

Example #2: (Improper preservation; Analysis holding time exceeded)

Aqueous mercury samples MAED54 and MAED55 were analyzed by CLP SOW ILM05.4. The validator examines the data package and determines that the laboratory did not report the pH. The validator contacts the laboratory to determine whether the pH was checked by the laboratory and notes that it was not checked. The validator then examines the Traffic Report contained in the data package and notes that the sampler failed to record what, if any, preservation techniques were utilized. The validator contacts the sampler who has no record of the samples being preserved with HNO_3.

The sampling date for MAED54 and MAED55 was 6/1/07 and the analysis date was 6/30/07, 29 days from sampling. The aqueous mercury sample exceeded the technical holding time criteria by one day. The validator examines the Form I and notes that mercury is reported at 2 ug/L for MAED54 and non-detected for MAED55. The validator reports the mercury positive detect as estimated (J) and uses professional judgment to reject (R) the non-detect on the Data Summary Table since there is no record of samples being preserved with acid. The validator notes in the Data Validation Memorandum that the sample data are qualified based on improper preservation (without acid) and exceeded technical holding times.

Example #3: (Proper preservation; Analysis holding time grossly exceeded)

Cyanide soil samples were sampled on 8/1/07 and received at the laboratory on 8/2/07. Upon examination of the Traffic Report, laboratory receipt information, and sample tracking records, the validator determines that the samples were properly preserved at 4°C. All samples were not analyzed until 9/1/07, 31 days from the sampling date, due to a laboratory tracking error, and the analysis holding time was grossly exceeded. The validator estimates (J) positive detects and uses professional judgment to reject (R) non-detects for cyanide in all samples on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

II. ICP-MS TUNE

A. OBJECTIVE

Inductively coupled plasma-mass spectrometer (ICP-MS) tunes are performed to verify proper mass calibration and resolution and serves as an initial demonstration of instrument stability and precision.

B. CRITERIA

ICP-MS tuning (instrument performance) criteria are not sample-specific. Since conformance is determined using standard materials, these criteria should be met under all circumstances. The CLP ICP-MS method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the ICP-MS analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from tuning anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Verify that the ICP-MS instrument was tuned prior to instrument calibration.	1. a. If the ICP-MS instrument was not tuned prior to calibration, then the validator should reject (R) all data not associated with a tune. The validator may need to obtain additional information from the laboratory. Rejected data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
1. b. i. Verify that the ICP-MS tuning solution contained the method-required analytes representing the mass regions of interest. * ii. Verify that the method-required number of analyses or scans of the ICP-MS tuning solution was performed.	1. b. If the laboratory did not use the required analytes spanning the analytical range to tune the instrument or if the tuning solution was not analyzed at the required frequency, then the validator must use professional judgment to determine whether the associated sample data should be qualified. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Verify from the reported results that the mass calibration, spectrometer resolution (peak width) and %RSD of the absolute signals for the analytes in the tuning solution are within the method QC acceptance criteria.	2. a. If tabulated results forms are not present for each ICP-MS tune under which samples are analyzed, then the validator should contact the laboratory to obtain the tabulated forms. b. If the mass calibration is not within the method-required mass range of the true mass for any isotope in the tuning solution, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects in all samples associated with that tune. c. If the mass resolution (peak width) is not within the method QC acceptance criteria at the specified peak height, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects in all samples associated with that tune. d. If the %RSD of the absolute signals of any analyte in the tuning solution is not within the method QC acceptance criteria, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects in all samples associated with that tune to indicate possible instrument instability.

C. EVALUATION	D. ACTION
*3. Check raw data to verify that the tune data are accurately reported on the tabulated forms.	3. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*4. Review standard preparation logs (if available in the data package) to verify that the analytes in the tuning solution were at the method-required concentrations.	4. If standards preparation data are not included in the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to validate sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b.ii, C.3, C.4

Table INORG-II-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON THE ICP-MS TUNE

Sample Results	Mass Calibration (amu) > QC Limit	Mass Resolution/ Peak Width (amu) > QC Limit	%RSD > QC Limit
Detects	J	J	J
Non-detects	UJ	UJ	UJ

E. EXAMPLES

Example #1: (Mass calibration outside criteria)

Tabulated tuning data generated under CLP SOW ILM05.4 show a measured mass of 24.16 amu which exceeds the method QC criteria of ≤ 0.1 amu from the true mass of 24 amu for magnesium. The validator estimates (J) all positive detects and estimates (UJ) all non-detects in all samples associated with that tune on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

Example #2: (Mass resolution/peak width outside criteria)

The validator examines the tabulated tune data generated under EPA Method 6020. The peak width for cobalt is 1.3 amu at 10% peak height which is outside the method criteria of less than 0.9 amu full width at 10% peak height. The validator estimates (J) all positive detects and estimates (UJ) all non-detects in all samples associated with that tune on the Data Summary Table. The validator discusses the instrument performance and the resulting qualifications in the Data Validation Memorandum.

Example #3: (ICP-MS tune %RSD outside criteria)

The validator examines the tabulated tune data generated under CLP SOW ILM05.4. The %RSD is 7.5% for beryllium and 6.3% for indium, which exceed the method QC criteria of 5% RSD. The validator estimates (J) all positive detects and estimates (UJ) all non-detects in all samples associated with that tune on the Data Summary Table. The validator discusses the instrument instability and precision problem and the resulting qualifications in the Data Validation Memorandum.

III. CALIBRATIONS

A. OBJECTIVE

Compliance requirements for instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the instrument calibration is still valid by checking the performance of the instrument on a continual basis. The Quantitation Limit Check Standard verifies that the instrument is capable of producing acceptable quantitative data at the low end of the calibration curve.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. Initial Calibration

   a. Initial calibration standards containing target analytes at method-specified concentrations are analyzed daily, each time the instrument is set up, and prior to the analysis of any field samples, QC samples, and blanks. The method-required number of calibration standards and replicates must be analyzed for each initial calibration using the same instrumental conditions that are used to analyze field samples, QC samples, and blanks.

   b. For methods which require the determination of correlation coefficients for the calibration curve, correlation coefficients must be within the method QC acceptance criteria.

2. Initial and Continuing Calibration Verifications

   a. Initial and continuing calibration verification standards must contain target analytes at method-specified concentrations and must be from a source different from that of the initial calibration standards. The ICV is analyzed immediately after initial calibration and the CCV is analyzed at a frequency of every ten samples or every two hours during an analytical run, whichever is more frequent, and at the end of the run after the last sample.

   b. The initial and continuing calibration verification recoveries for all target analytes must be within the QC acceptance criteria specified in the method.

   c. For methods which require replicate analyses (i.e., replicate integrations), the percent relative standard deviation (%RSD) must be within the method QC acceptance criteria.

3. Quantitation Limit Check Standard

a. The Quantitation Limit Check Standard must contain target analytes in the reagent blank at or near the quantitation limit according to the method requirements and is analyzed after the initial calibration is verified. Some methods may require the analysis of a Laboratory Fortified Blank in place of or in addition to the QL Check Standard. (See Section XII, Sensitivity Check.)

b. The Quantitation Limit Check Standard recoveries must be within the method QC acceptance criteria for all target analytes.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from initial and continuing calibration anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Initial Calibration a. Verify that the correct number of instrument calibration standards were prepared and analyzed at the method-required concentrations and frequency, and that the correct number of replicate analyses was performed.	1. Initial Calibration a. If the laboratory did not use the required number of standards at the correct concentrations, frequency, and number of replicates when analyzing the initial calibration standards, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.

C. EVALUATION	D. ACTION
*1. b. For methods that require determination of correlation coefficients for the calibration curve, verify that the method-required correlation coefficient criteria were met.	1. b. If the correlation coefficient for any target analyte did not meet the method QC acceptance criteria, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples associated with the initial calibration. ii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the initial calibration. iii. Depending on the degree of the deviation from linearity, the validator may use professional judgment to reject (R) all positive detects and/or all non-detects. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Initial and Continuing Calibration Verifications a. Verify that the ICV and CCV standards were analyzed at the method-required concentrations and frequency. Verify that the source of the ICV and CCV is different from that of the initial calibration standards.	2. Initial and Continuing Calibration Verifications Qualification is based on samples associated with a particular ICV or CCV. For an ICV not meeting criteria, action is applied to the affected analyte in all samples reported from the same analytical run. For a CCV not meeting criteria, generally, action is applied to the affected analyte in all samples bracketed by that CCV, that is, in all sample analyzed between the previous acceptable CCV analysis and the subsequent acceptable CCV analysis in the same analytical run. a. If the laboratory did not analyze the ICV and CCV standards at the required concentrations and frequency and did not use a separate source, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.

C. EVALUATION	D. ACTION
2. b. Verify that all ICV and CCV recoveries are within the QC acceptance criteria specified in the method.	2. b. i. If the ICV or CCV percent recovery for any target analyte is greater than the upper limit of the method QC acceptance criteria but within the following ranges: - ICP-AES/MS: upper limit < %R ≤ 125% - Hg, CN: upper limit < %R ≤ 130% then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that ICV or CCV. - Accept non-detects for the affected analyte in all samples associated with that ICV or CCV. ii. If the ICV or CCV percent recovery is greater than the following limits: - ICP-AES/MS: %R > 125% - Hg, CN: %R > 130% then the validator should: - Reject (R) positive detects for the affected analyte in all samples associated with that ICV or CCV. - Accept non-detects for the affected analyte in all samples associated with that ICV or CCV.

C. EVALUATION	D. ACTION
2. b. Continued from above.	2. b. iii. If the ICV or CCV percent recovery is less than the lower limit of the method QC acceptance criteria but within the following ranges: - ICP-AES/MS: 75% ≤ %R < lower limit - Hg, CN: 70% ≤ %R < lower limit then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that ICV or CCV. - Estimate (UJ) non-detects for the affected analyte in all samples associated with that ICV or CCV. iv. If the ICV or CCV percent recovery is less than the following limits: - ICP-AES/MS: %R < 75% - Hg, CN: %R < 70% then the validator should: - Reject (R) positive detects and non-detects for the affected analyte in all samples associated with that ICV or CCV.

C. EVALUATION	D. ACTION
2. c. Evaluate the appropriateness of qualifying the affected analyte in all samples which are reported from the same analytical run.	2. c. Generally, action applies to the affected analyte in samples associated with the specific CCV. However, professional judgment may be used to qualify the affected analyte in all samples reported from the same analytical run. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
d. Evaluate the appropriateness of qualifying additional analytes when the majority of analytes have ICV or CCV recoveries outside the QC criteria specified in the method.	d. For ICP-AES or ICP-MS multi-analyte analysis, if the majority of the ICV or CCV recoveries are outside method QC acceptance criteria, this may indicate a more serious problem with the instrument's stability. The validator should use professional judgment to qualify or reject all analytes in all samples associated with that ICV or CCV based on the number of analytes with recoveries outside QC limits and the direction and degree of deviation. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* e. Verify that, for methods which require replicate analyses (i.e., replicate integrations), the %RSD is within the method QC acceptance criteria.	e. If any %RSD for replicate analyses in the ICV or CCV is outside the method QC acceptance criteria, then the validator should use professional judgment to either accept or qualify associated sample data. The validator should evaluate whether the instrument consistently generates erratic responses or whether the imprecision is isolated to a specific ICV or CCV. The %RSDs of other QC samples should also be evaluated. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. Quantitation Limit Check Standard	3. Quantitation Limit Check Standard Qualification is based on samples associated with a particular QL Check Standard. For a QL Check Standard not meeting criteria, generally, action is applied to the affected analyte in all samples bracketed by that QL Check Standard, that is, in all sample analyzed between the previous acceptable QL Check Standard analysis and the subsequent acceptable QL Check Standard analysis from the same analytical run.
a. Verify that the Quantitation Limit Check Standard contains all target analytes at concentrations equal to or near their quantitation limits, as required by the method, and that the QL Check Standard was analyzed at the required frequency.	a. If a QL Check Standard was not analyzed for all target analytes at the required concentration and frequency, the validator should use professional judgment to assess the impact of analytical sensitivity on data quality. The validator should review any other low level QC data to determine the action to be taken.
b. Verify that all QL Check Standard recoveries are within the method QC acceptance criteria.	b. If any of the QL Check Standard recoveries are outside the method QC acceptance criteria, then the QL Check Standard results should be used to qualify sample data for the affected analytes that are included in the solution. The validator should use professional judgment to qualify sample data for analytes not present in the check standard, taking into account information that may exist in the sample delivery group for other low level standards. Note: If the QL Check Standard was spiked at low level concentrations other than the quantitation limit, then qualifications for positive detects should be based on 2x the true value of the low level check standard rather than on 2x the QL.

C. EVALUATION	D. ACTION
3. b. Continued from above.	3. b. i. If a QL Check Standard recovery for any target analyte is greater than the upper limit of the method QC acceptance criteria, but less than or equal to 180%, then the validator should: - Estimate (J) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard to indicate potential high bias. - Accept non-detects for the affected analyte in all samples associated with that QL Check Standard. ii. If a QL Check Standard analyte recovery is greater than 180%, then the validator should: - Use professional judgment to estimate (J) or reject (R) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard. - Use professional judgment to accept or estimate positive detects greater than or equal to 2x the QL Check Standard true value but less than the true value of the next highest concentration QC sample to indicate potential high bias. - Accept non-detects for the affected analyte in all samples associated with that QL Check Standard.

C. EVALUATION	D. ACTION
3. b. Continued from above.	3. b. iii. If a QL Check Standard analyte recovery is less than the lower limit of the method QC acceptance criteria, but greater than or equal to 50%, then the validator should: - Estimate (J) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard to indicate potential low bias. - Estimate (UJ) non-detects for the affected analyte in all samples associated with that QL Check Standard to indicate potential low bias. iv. If a QL Check Standard analyte recovery is less than 50%, then the validator should: - Use professional judgment to estimate (J) or reject (R) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard. - Use professional judgment to accept or estimate positive detects greater than or equal to 2x the QL Check Standard true value but less than the true value of the next highest concentration QC sample to indicate potential low bias. - Reject (R) non-detects for the affected analyte in all samples associated with that QL Check Standard to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
3. b. Continued from above.	3. b. v. The validator should use professional judgment to estimate (J) or reject (R) low level sample results when the QL Check Standard exceeds the acceptance criteria, taking into consideration project DQOs. c. The validator should use professional judgment to take action on positive detects greater than 2x the true value of the low level check standard, taking into consideration the laboratory's accuracy for other QC samples and standards and project DQOs. The validator should evaluate all relevant QC data which may provide information on the laboratory's ability to accurately quantitate target analytes at concentration ranges greater than 2x the true value of the low level check standard. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum. d. For ICP-AES or ICP-MS multi-analyte analysis, if the majority of the Quantitation Limit Check Standard recoveries are outside method QC acceptance criteria, this may indicate a more serious problem with the instrument's stability and/or accuracy at the low end of the calibration curve. The validator may use professional judgment to qualify or reject all sample results associated with that QL Check Standard based on the number of analytes with recoveries outside QC limits and the degree of deviation. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*4. a. Review standard preparation logs (if provided in the data package) to ensure that all initial calibration, initial calibration verification, continuing calibration verification and Quantitation Limit Check Standard concentrations are accurate and traceable.	4. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to facilitate the validation of sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.
* b. i. Check and recalculate the initial calibration, initial calibration verification, continuing calibration verification, and QL Check Standard concentrations and percent recoveries for at least one analyte per method (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values. ii. For methods that require determination of calibration curve correlation coefficients, check and recalculate the correlation coefficient for at least one target analyte per method. Verify that the recalculated value agrees within ±10% of the laboratory reported value.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable to a Tier III data validation:**

C.1.b, C.2.e, C.4.a, C.4.b

Table INORG-III-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON ICV AND CCV PERCENT RECOVERIES

Sample Results	% Recovery				
	ICP: %R < 75% Hg/CN: %R < 70%	ICP: 75% ≤ %R < LL Hg/CN: 70% ≤ %R < LL	LL ≤ %R ≤ UL	ICP: UL < %R ≤ 125% Hg/CN: UL < %R ≤ 130%	ICP: %R > 125% Hg/CN: %R > 130%
Detects	R	J	A	J	R
Non-detects	R	UJ	A	A	A

LL = Lower limit of method QC acceptance criteria
UL = Upper limit of method QC acceptance criteria

Table INORG-III-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON QUANTITATION LIMIT CHECK STANDARD RECOVERIES

Sample Results	% Recovery				
	%R < 50%	50% ≤ %R < LL	LL ≤ %R ≤ UL	UL < %R ≤ 180%	%R > 180%
Detects*	J/R (< 2x TV) ** Prof. Judg. (≥ 2x TV)**	J (< 2x TV)	A	J (< 2x TV)	J/R (< 2x TV)** Prof. Judg. (≥ 2x TV)**
Non-detects	R	UJ	A	A	A

LL = Lower limit of method QC acceptance criteria
UL = Upper limit of method QC acceptance criteria
* Action is applied to positive detects less than 2x the true value of the QL Check Standard.
** Professional judgment may be used to estimate or reject positive detects less than 2x the true value taking into account project DQOs. Professional judgment should be used to accept or estimate positive detects greater than or equal to 2x the true value of the Check Standard but less than the next highest concentration QC sample or standard.

E. EXAMPLES

Example #1: (High ICV recovery for mercury)

Samples were analyzed for mercury by CLP SOW ILM05.4 in two analytical runs. The first run occurred on 8/1/07 and the second run occurred on 8/2/07. The ICV analyzed on 8/1/07 recovered at 118% and was within the method QC acceptance criteria of 80-120% recovery. The ICV analyzed on 8/2/07 recovered at 122%, above the upper QC limit. Therefore, only the samples analyzed and reported from the second run on 8/2/07 were affected. The validator estimates (J) the positive detects for mercury reported from the 8/2/07 run and accepts the mercury non-detects on the Data Summary Table. The validator notes in the Data Validation Memorandum that the qualified mercury results may be biased high.

Example #2: (Low CCV recovery for aluminum)

Samples were analyzed for aluminum by ICP-AES under CLP SOW ILM05.4. In the analytical run on 8/25/07, the CCV3 recovered at 82% for aluminum, below the method QC acceptance criteria of 90-110% recovery but above 75%. The validator notes that the other calibration verifications in the same run, ICV, CCV1, CCV2, and CCV4, all recovered within the method QC acceptance criteria. Samples MAED52 through MAED57, analyzed between CCV2 and CCV3, and samples MAED58 through MAED60, analyzed between CCV3 and CCV4, were associated with the low CCV3 recovery. As a result, samples MAED52 through MAED60 were not bracketed by acceptable CCVs. The validator estimates (J) the positive detects and estimates (UJ) the non-detects for aluminum in samples MAED52 through MAED60 on the Data Summary Table. The validator notes in the Data Validation Memorandum that the aluminum results in these samples may be biased low.

Example #3: (High QL Check Standard recovery for chromium)

Chromium was analyzed by ICP-AES under CLP SOW ILM05.4. The CRI standard, or QL Check Standard, recovered at 140% for chromium, above the method QC criteria of 70-130%. Therefore, the validator estimates (J) positive detects less than 2x the QL and accepts non-detects on the Data Summary Table for chromium results reported from that analytical run. The validator discusses the potential high bias in the low level results and notes the sample qualifications in the Data Validation Memorandum.

Example #4: (Low QL Check Standard recovery for lead)

Lead was analyzed by ICP-AES under CLP SOW ILM05.4. The CRI standard, or QL Check Standard, recovered at 40%, well below the method QC criteria of 70-130%. Therefore, the validator uses professional judgment to reject (R) positive detects less than 2x the QL since low level lead near the QL was of concern at the site. Professional judgment was used to estimate (J) positive detects greater than or equal to 2x the QL but less than the ICV true value since the ICV was the next highest concentration standard in the analytical run, and non-detects were accepted for lead on the Data Summary Table. The validator notes in the Data Validation Memorandum that accuracy at the low end of the calibration curve is questionable.

IV. BLANKS

A. OBJECTIVE

The purpose of blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory and/or field activities and to subsequently assess their contribution to measurement error. The criteria for evaluation of laboratory blanks (preparation/method blanks and calibration/instrument blanks) may be applied to any blank associated with the samples. If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent measurement error associated with the entire data set, or if the problem is an isolated occurrence limited to specific samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. The frequency and types of blanks collected and analyzed must support the site-specific Data Quality Objectives (DQOs) as documented in the EPA-approved QAPP or SAP. Different types of blanks may be used to identify the source of potential contamination resulting in analytical and/or sampling measurement error. The following table lists types of blanks, the environment of those blanks, and the possible sources of contamination associated with those blanks:

BLANK	LABORATORY/FIELD	IDENTIFIES CONTAMINATION FROM
Preparation (Method) Blank	Laboratory	Laboratory and Reagents
Calibration (Instrument) Blank	Laboratory	Instrumentation
Bottle Blank	Field	Sample Container
Equipment Blank (Rinsate)	Field	Sampling Equipment

Note: Aqueous equipment (rinsate) blank results and bottle blank results will be used to determine blank action levels for aqueous samples typically based on a volume of 1 liter of blank sample. Ideally, soil/sediment blanks should be used to determine soil/sediment blank actions for soil/sediment samples based on a known weight of blank sample. However, aqueous equipment blanks and bottle blanks are often collected to evaluate contamination associated with soil/sediment sampling. **Aqueous equipment (rinsate) blank results and bottle blank results will not be used to determine blank action levels for non-aqueous samples.** Analytes that are present in both the non-aqueous sample and the associated aqueous equipment blank or bottle blank will be flagged EB (Equipment Blank) or BB (Bottle Blank), respectively. The degree of "sampling error" that this flagged sample result represents will be left to the determination of the end user.

2. Preparation (Method) Blanks

   A preparation blank must be prepared (e.g., digested or distilled) and analyzed with each sample delivery group, with each batch of samples of similar matrix in each sample delivery group, or whenever a sample digestion or distillation is performed. The preparation blank must undergo all preparation and analysis procedures performed on samples. The preparation blank must be analyzed on the same instrument used to analyze the samples associated with the preparation blank.

3. Calibration (Instrument) Blanks

   An initial calibration blank must be analyzed following instrument calibration at the beginning of the run but after the initial calibration verification and prior to sample analysis. A continuing calibration blank must be analyzed after every 10 samples or every two hours during an analysis run, and at the end of the run for each analyte and on each instrument used to analyze samples.

4. For ICP-MS analysis, all blanks should be spiked with internal standards according to the method. Blank internal standards must meet method internal standard QC acceptance criteria.

5. No contaminants should be present in the blanks.

6. No negative results should be observed in the blanks.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from blank anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. **Qualification should be based upon a comparison of the sample concentration(s) with the highest blank concentration associated with the samples.** More than one blank action level for a given analyte may exist in a sample delivery group if samples are associated with different blanks. This may occur when samples are prepared in different batches, samples are analyzed in a separate analytical sequence, or if samples are associated with different equipment or bottle blanks. In cases of specific preparation and/or calibration blank contamination, the validator should use professional judgment to qualify only those samples associated with that isolated blank contamination. Likewise, the validator may need to apply blank qualifications to a sample delivery group based on associated equipment or bottle blank data that exist in another sample group data package. **Sample results must not be corrected by subtracting any blank values.**

C. EVALUATION	D. ACTION
1. a. Verify that the correct number and type of blanks have been collected and analyzed in accordance with the EPA-approved QAPP or SAP.	1. a. If the correct number and type of blanks have not been collected and analyzed, then the validator should note this deviation from the EPA-approved QAPP or SAP in the Data Validation Memorandum. The validator should use professional judgment to qualify sample data when blank data are absent. When required equipment (rinsate) or bottle blanks are not identified on the chain of custody, the validator must contact the sampler or site project manager to obtain this information and note this contact on the Blanks validation worksheet.
b. Ascertain if aqueous equipment (rinsate) blanks or aqueous bottle blanks have been collected with non-aqueous samples to identify sources of field contamination.	b. If positive results are detected in the aqueous equipment (rinsate) blanks and/or bottle blanks <u>and</u> the associated <u>non-aqueous</u> samples, then the validator should flag (EB or BB) those detected analytes in the associated non-aqueous samples to indicate to the end user that an indeterminate amount of sampling error has potentially affected the sample results. (See Example #4.)
2. a. Verify that a preparation blank analysis has been reported once per matrix, preparation and analytical method, batch of samples prepared, and SDG.	2. a. If preparation blanks were not analyzed at the required frequency and for each matrix, preparation and analytical method, preparation batch, and SDG, then the validator should use professional judgment to determine whether the associated sample data should be qualified.

C. EVALUATION	D. ACTION
*2. b. Verify from the raw data that the digestion/distillation and analysis dates and times, sample IDs, etc., are accurately reported on the tabulated result forms.	2. b. If review of the raw data reveals discrepancies and/or transcription errors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
3. a. Verify from the Blanks form and Analysis Run Log form that a calibration blank was analyzed after initial calibration of the instrument, at the beginning of the run but after the initial calibration verification, after every 10 samples or every two hours, and at the end of the run.	3. a. If calibration blanks were not analyzed at the required frequency, then the validator should use professional judgment to determine whether the associated sample data should be qualified.
* b. Verify from the raw data that the analysis dates and times, sample IDs, sequence of blank analyses, etc., are accurately reported on the tabulated result forms.	b. If review of the raw data reveals discrepancies and/or transcription errors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
4. a. For ICP-MS data, verify that the blank internal standard responses meet method QC acceptance criteria.	4. a. If blank internal standard responses do not meet method QC acceptance criteria, then the validator should use professional judgment in applying blank actions. The possibility of false positives or false negatives being incorrectly reported for the blank should be evaluated.
* b. Check 10% of the raw data for each blank to verify that internal standard responses and/or percent relative intensities have been correctly transcribed to tabulated forms and that percent relative intensities have been correctly calculated.	b. If the laboratory has incorrectly transcribed and/or calculated the data, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
5. Target Analyte Contaminants Detected in Blanks	**5. Target Analyte Contaminants Detected in Blanks** If a contaminant is found in a blank but not in the associated sample, then no action is taken. If a contaminant is found in both a blank and the associated sample, then the validator should note this problem in the Data Validation Memorandum and qualify the data according to the following guidance. **Blank Action Level:** The blank action level is determined for each contaminant detected in the blanks associated with each sample. The blank action level is determined by multiplying by five the highest concentration of each contaminant detected among the blanks associated with each sample. Note: **If the blank action level for an analyte is determined using the value from an equipment blank or bottle blank, then the positive values in the equipment or bottle blank should be reported unqualified on the Data Summary Tables. However, if the blank action level is determined using the value from a laboratory blank (e.g., preparation or calibration blank), then the positive values in the equipment or bottle blanks should be qualified. (See Example #6.)**

C. EVALUATION	D. ACTION
5. a. Determine if any target analytes are present at concentrations greater than or equal to the MDL in any of the blanks.	5. a. If any target analyte is present at a concentration greater than or equal to the MDL in any of the blanks, then the following actions are taken. i. Positive sample results greater than the blank action level: - If a positive sample result for an analyte is greater than the blank action level (5 times the highest concentration in any associated blank) and greater than or equal to the sample quantitation limit, then the analyte's concentration should be reported as unqualified. - If a positive sample result is greater than the blank action level but is less than the sample quantitation limit, then no further qualification is necessary and the estimated sample result should be reported on the Data Summary Table. ii. Positive sample results less than or equal to the blank action level: - If a positive sample result for an analyte is less than or equal to the blank action level but is greater than or equal to the sample quantitation limit, then the sample quantitation limit for that analyte should be elevated to the concentration found in the sample and reported as not detected (U). (See Example #2.) The validator may use professional judgment to determine if further elevation of the quantitation limit is required.

C. EVALUATION	D. ACTION
5. a. Continued from above.	5. a. ii. Continued from above. - If a positive sample result for an analyte is less than or equal to the blank action level and is also less than the sample quantitation limit, then the sample quantitation limit should be reported on the Data Summary Tables as a non-detect (U). (See Example #1.) Note: The validator should note that blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results may not be on the same basis (units, dilution) as the calibration blank data. These factors must be taken into consideration when applying the "5x" criteria. The reviewer may find it easier to work from the raw data when comparing soil sample data to blank data. (See Example #5.) Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample data is deemed necessary. If the validator determines that the contamination originates from a source other than the sample, the sample data should be qualified. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample result. Since both results may not be reported, it may be impossible to verify this source of contamination. In this case, the "5x" rule may not apply; the target analyte should be reported as not detected (U), and an explanation of the data qualification rationale should be provided in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
5. b. Determine if contamination greater than the CRQL for any analyte exists in any of the blanks.	5. b. If contamination greater than the CRQL for any analyte exists in any blank, depending on the concentration of the contaminant in the sample compared to the blank, as well as the project DQOs, the validator may use professional judgment to reject (R) positive results for the affected analyte in samples associated with that blank due to the interference. This serious problem should be discussed in the Data Validation Memorandum.
c. Determine if blank contaminants are consistently found in the laboratory blanks. Determine if instrument contamination is isolated to specific sample runs.	c. While preparation, equipment, and bottle blanks are associated with specific samples, the association of calibration blanks to samples analyzed within the same analytical run is not always straightforward. Generally, for ICBs not meeting blank criteria, action applies to all samples analyzed in the same analytical run as the ICB. For CCBs, the validator should review the blank data to determine whether the blank contamination is isolated to specific CCBs or if the blank contamination appears consistently throughout the analytical run. If the validator determines that instrument contamination is isolated to a specific CCB and is limited to a specific set of samples, then the validator may use professional judgment to apply blank actions only to those samples bracketed by the affected CCB. If the validator determines that contamination is not isolated to a specific set of samples within a sample run, then the validator may use professional judgment to apply blank actions to all samples analyzed in the same analytical run. If target analytes are consistently found in the laboratory blank(s), it may indicate a systematic problem in the laboratory and should be noted in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
5. d. Determine if blank contamination exists solely in field blanks.	5. d. If contamination exists solely in the equipment (rinsate) or bottle blank at levels greater than the CRQL, then the validator should notify the sampler. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.
e. Evaluate the overall contamination in each type of blank to ascertain probable source(s) of contamination. For example, a contaminated equipment blank might indicate decontamination problems if the preparation, calibration, and bottle blanks were all clean.	e. If a review of the various types of blanks identifies a potential source of blank contamination, then the validator should discuss this problem in the Data Validation Memorandum. The validator should identify whether the measurement error is a result of either sampling or analytical error or both (see the Data Validation Manual, p. 1).
6. **Negative Blank Results Reported For Target Analytes**	6. **Negative Blank Results Reported For Target Analytes** **Negative Blank Action Level:** The negative blank action level is determined for each target analyte reported at less than or equal to the negative MDL in the blanks associated with each sample. The negative blank action level is the absolute value of five times the lowest negative value for each analyte reported among all of the blanks associated with each sample.
a. Determine if any target analytes are reported at concentrations less than or equal to the negative MDL in any of the blanks. (Note: The tabulated forms may not report negative values. It may be necessary to review the raw data for this information.)	a. Any target analyte reported as a negative value less than or equal to the negative MDL in any blank should be carefully evaluated to determine its effect on the sample data. i. Positive sample results greater than the negative blank action level: - If a positive sample result for an analyte is greater than the negative blank action level and greater than or equal to the sample quantitation limit, then the sample result should be reported as unqualified.

C. EVALUATION	D. ACTION
6. a. Continued from above.	6. a. i. Continued from above. - If a positive sample result for an analyte is greater than the negative blank action level and is less than the sample quantitation limit, then no further qualification is necessary and the estimated sample result should be reported on the Data Summary Table. ii. Positive sample results less than or equal to the negative blank action level: - If a positive sample result for an analyte is less than or equal to the negative blank action level and greater than or equal to the sample quantitation limit, then estimate (J) the positive detect for the affected analyte to indicate potential low bias. The validator may use professional judgment to determine if further qualification of sample results greater than the negative blank action level is required. - If a positive sample result for an analyte is less than or equal to the negative blank action level and is less than the sample quantitation limit, then no further action is required and the estimated sample result should be reported on the Data Summary Table. iii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the negative blank to indicate potential low bias.

C. EVALUATION	D. ACTION
6. b. Determine if negative blank values less than the negative quantitation limit for any analyte exist in any of the blanks.	6. b. Any blank reported with a negative blank value should be carefully evaluated to determine its effect on the sample data. Negative calibration blank values may indicate problems with the initial calibration or instrument/baseline drift. Negative blank values less than the negative quantitation limit may indicate a major instrument problem or a major change in instrument conditions and should be noted in the Data Validation Memorandum. If negative blank values less than the negative quantitation limit exist in any blank, then the validator may use professional judgment to reject (R) non-detects or positive detects for the affected analyte in samples associated with the blank. This serious problem should be discussed in the Data Validation Memorandum.
c. Determine if both negative and positive blank values are associated with a particular analyte.	c. If both negative and positive blank results are reported for an analyte and are associated with the same sample, then the validator should use professional judgment when applying blank actions. The following general guidance should be used when the positive sample result is less than or equal to both the positive and negative blank action levels: - If a positive sample result is greater than or equal to the sample quantitation limit, then the sample quantitation limit should be elevated to the concentration found in the sample and reported as a non-detect (U) for positive blank actions. The sample quantitation limit is then estimated (UJ) for negative blank actions on the Data Summary Table.

C. EVALUATION	D. ACTION
6. c. Continued from above.	6. c. Continued from above. - If a positive sample result is less than the sample quantitation limit, then the sample quantitation limit is reported as a non-detect (U) due to positive blank actions. The sample quantitation limit is then estimated (UJ) due to negative blank actions on the Data Summary Table.
*7. Review the raw data to confirm the presence of target analytes in the blanks and to evaluate the presence of additional contaminants. Confirm any negative results reported for the blanks as well as any negative blank results in the raw data that were not reported on the tabulated forms.	7. If review of the raw data suggests that additional contaminants or additional negative results are present or, conversely, the review indicates false positive or false negative results have been reported, then the validator should contact the laboratory to obtain additional information and/or have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation.**

C.2.b, C.3.b, C.4.b, C.7

Table INORG-IV-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON POSITIVE BLANK RESULTS

Positive Blank Result	Sample Result		Action
≥ MDL	> Blank Action Level and	≥ QL	A
		< QL	No further action (report estimated sample result)
	≤ Blank Action Level and	≥ QL	U - Raise the QL to the sample result and report as a non-detect
		< QL	U - Report the QL
	Non-detect (U)		A

Blank Action Level = 5x the highest blank concentration associated with the sample
QL = Sample Quantitation Limit

Note: Aqueous equipment (rinsate) and bottle blank results are not used to determine blank action levels for non-aqueous samples. Analytes present in both the non-aqueous sample and the associated aqueous equipment or bottle blanks should be flagged EB or BB, respectively.

Table INORG-IV-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON NEGATIVE BLANK RESULTS

Negative Blank Result	Sample Result		Action
≤ (-MDL)	> Negative Blank Action Level and	≥ QL	A
		< QL	No further action (Report estimated sample result)
	≤ Negative Blank Action Level and	≥ QL	J - Estimate the sample result
		< QL	No further action (report estimated sample result)
	Non-detect (U)		UJ - Estimate the QL

Negative Blank Action Level = absolute value of 5x the lowest negative blank value associated with the sample
QL = Sample Quantitation Limit

E. EXAMPLES

Example #1: (Initial calibration blank target analyte contaminant; Sample result ≤ blank action level and < QL)

The initial calibration blank (ICB) contained the highest concentration of copper among all of the blanks analyzed. In addition, all field samples analyzed were associated with the same contaminated initial calibration blank. Copper is detected in an aqueous field sample at less than the CRQL and less than 5x the ICB concentration.

5x Rule

	ug/L
Initial Calibration Blank Result	8
CRQL	25
Copper Sample Result	21 J
Blank Action Level	40 (5x8)
Qualified Sample Result	25 U

In this case, the copper sample result is less than the blank action level of 40 ug/L and is, therefore, reported as a non-detect at the CRQL on the Data Summary Table. The validator notes this problem in the Data Validation Memorandum.

Example #2: (Equipment blank target analyte contaminant; Sample result ≤ blank action level and ≥ QL)

An equipment blank was associated with a sample delivery group of aqueous samples. The equipment blank contained the highest concentration of sodium among all of the blanks analyzed. In addition, all aqueous field samples analyzed were associated with the same contaminated equipment blank. Sodium is detected in an aqueous field sample at greater than the CRQL and less than 5x the equipment blank concentration.

5x Rule

	ug/L
Equipment Blank Result	6400
CRQL	5000
Sodium Sample Result	8700
Blank Action Level	32000 (5x6400)
Qualified Sample Result	8700 U

In this case, the sodium sample result is less than the blank action level of 32000 ug/L. The validator elevates the sample quantitation limit for sodium to the sample concentration on the Data Summary Table and reports the result as 8700 U since the result is less than the blank action level. This problem is noted in the Data Validation Memorandum.

E. **EXAMPLES** (continued)

Example #3: (Preparation blank contamination; Sample result > blank action level)

Calcium is detected in an aqueous field sample at greater than 5x the preparation blank concentration, the highest level of calcium detected among all of the blanks associated with the sample.

5x Rule

	ug/L
Preparation Blank Result	1300
CRQL	5000
Calcium Sample Result	18200
Blank Action Level	6500 (5x1300)

The calcium sample result is greater than the blank action level of 6500 ug/L and is reported unqualified on the Data Summary Table.

Example #4: (Target analyte contamination in aqueous equipment blank associated with soil samples)

An equipment blank (rinsate) was associated with a sample delivery group of soil samples. The validator examines the data and finds that the equipment blank contains 20 ug/L of zinc. The validator then reviews all other blank data and finds no further zinc contamination. Soil sample MACN40 contains 11 mg/kg of zinc.

The validator reports the zinc result for sample MACN40 as 11 (EB) on the Data Summary Table to indicate to the end user that sampling error has potentially affected the sample results and notes this information in the Data Validation Memorandum.

E. EXAMPLES (continued)

Example #5: (Preparation blank contaminant; Application of sample weights, % solids, and volumes)

Soil samples were analyzed under CLP SOW ILM05.4. The preparation blank contained the highest concentrations of nickel (3 mg/kg) and aluminum (5 mg/kg) among all of the blanks associated with the samples in this sample delivery group. Soil sample MACN42 contained nickel at 13 mg/kg and aluminum at 2 mg/kg.

The validator calculates the sample quantitation limits for nickel and aluminum in sample MACN42 for 1.0 g digested, and 80% solids.

$$\text{Nickel QL} = \frac{\text{CRQL}}{\text{\% solids}} = \frac{4\ \text{mg/kg}}{0.8} = 5\ \text{mg/kg}$$

$$\text{Aluminum QL} = \frac{\text{CRQL}}{\text{\% solids}} = \frac{20\ \text{mg/kg}}{0.8} = 25\ \text{mg/kg}$$

The validator applies the following actions to the nickel and aluminum results for sample MACN42:

5x Rule - Sample MACN42

	Nickel mg/kg (dry wt.)		Aluminum mg/kg (dry wt.)
Prep. Blank Result	3	Prep. Blank Result	5
Sample QL	5	Sample QL	25
Sample Result	13	Sample Result	2 J
Blank Action Level	15 (5x3)	Blank Action Level	25 (5x5)
Qualified Sample Result	13 U	Qualified Sample Result	25 U

- The sample quantitation limit for nickel is elevated to the sample result on the Data Summary Table and is reported as 13 U since the result is greater than the sample quantitation limit but less than the blank action level.

- The aluminum sample result is replaced with the sample quantitation limit and is reported as 25 U on the Data Summary Table since the positive sample result of 2 mg/kg was below both the sample quantitation limit and the blank action level.

The validator notes all actions taken in the Data Validation Memorandum.

E. **EXAMPLES** (continued)

Example #6: (Application of aqueous laboratory blank action levels to equipment blanks)

The preparation blank associated with a batch of aqueous samples was contaminated with 90 ug/L of iron and vanadium was not detected. The equipment blank associated with this batch of samples was contaminated with 80 ug/L of iron and 60 ug/L of vanadium. Since iron was detected in both the preparation blank and the equipment blank, the blank with the highest iron concentration is used to determine the blank action level. The preparation blank concentration is, therefore, used to determine the blank action level for iron..

5x Rule

	Iron ug/L		Vanadium ug/L
Preparation Blank Result	90	Preparation Blank Result	50 U
Equipment Blank Result	80	Equipment Blank Result	60
CRQL	100	CRQL	50
Blank Action Level	450 (5x90)	Blank Action Level	300 (5x60)
Qualified Equipment Blank	100 U	Qualified Equipment Blank	60

The iron positive detect in the equipment blank is reported as a non-detect at the CRQL, 100 U, on the Data Summary Table since the equipment blank result is less than the blank action level established by the preparation blank. The blank action level for vanadium is determined using the value from the equipment blank and, therefore, the vanadium positive detect in the equipment blank is reported unqualified as 60 ug/L on the Data Summary Table.

E. EXAMPLES (continued)

Example #7: (Negative target analyte blank result; One positive sample result < negative blank action level, one non-detect sample result)

A preparation blank had a reported negative arsenic result of -7 ug/L, less than the negative arsenic MDL of 3 ug/L and the lowest negative arsenic value among all of the associated blanks. Aqueous sample MA2CB6 was reported with a positive arsenic result of 18 ug/L and aqueous sample MA2CB7 was reported as a non-detect at 10 U ug/L for arsenic. The negative blank action level for arsenic was calculated as 35 ug/L (5x |-7 ug/L|).

5x Rule

	Arsenic ug/L
MDL	3
Preparation Blank Result	-7
CRQL	10
Sample MA2CB6 Result	18
Sample MA2CB7 Result	10 U
Negative Blank Action Level	35 (5x |-7|)
Qualified Sample MA2CB6 Result	18 J
Qualified Sample MA2CB7 Result	10 UJ

The positive arsenic result for sample MA2CB6 is less than the negative blank action level of 35 ug/L and is, therefore, reported as an estimated value of 18 J ug/L on the Data Summary Table. The non-detect arsenic result for sample MA2CB7 is reported as estimated at the sample quantitation limit of 10 UJ ug/L on the Data Summary Table. The validator notes all actions taken in the Data Validation Memorandum and discusses the possible low bias in the results.

E. EXAMPLES (continued)

Example #8: (Different blank actions for an aqueous equipment blank and a soil sample)

An SDG consisted of soil samples and an aqueous rinsate blank. The aqueous rinsate blank was prepared in a different batch from the soil samples. In the soil batch, the highest blank concentration for copper was 1.8 mg/kg in the preparation blank, and the copper result for soil sample MA1D7F was reported as 3.6 mg/kg. In the water batch, the highest blank concentration for copper was 14 ug/L in the aqueous preparation blank. The aqueous rinsate blank RB-04 was reported as 8 ug/L. The results and qualifications are summarized below.

5x Rule

Soil Sample	Copper mg/kg	Aqueous Rinsate Blank	Copper ug/L
Preparation Blank Result	1.8	Preparation Blank Result	14
Sample MA1DF7 Result	3.6	Rinsate Blank RB-04 Result	8
CRQL	2.5	CRQL	25
Blank Action Level (Soil)	9.0 (5x1.8)	Blank Action Level (Aq.)	70 (5x14)
Qualified MA1DF7 Result	3.6 U	Qualified RB-04 Result	25 U

The copper result in soil sample MA1D7F is reported on the Data Summary Table as a non-detect at 3.6 U mg/kg since it is less than the Blank Action Level (based on the associated soil preparation blank) and greater than the sample quantitation limit. The copper result in aqueous rinsate blank RB-04 is reported on the Data Summary Table as a non-detect at the quantitation limit of 25 U ug/L since it is less than the Blank Action Level (based on the associated aqueous preparation blank) and less than the quantitation limit. The validator notes all actions taken in the Data Validation Memorandum and discusses the copper contamination in the rinsate blank being attributed to contamination introduced in the laboratory.

E. **EXAMPLES** (continued)

Example #9: (Two equipment blanks with target analyte contaminants, each associated with different samples)

For a sample delivery group, the traffic report and field sampling notes indicate that eight aqueous samples were collected on sampling day three along with associated equipment blank EB-T03, and six aqueous samples were collected on sampling day four along with associated equipment blank EB-T04. The two equipment blanks contained the highest levels of zinc contamination for this SDG, 110 ug/L zinc in EB-T03 and 280 ug/L in EB-T04. Zinc was detected in two samples, MA14G7 (day three) and MA11A4 (day four).

5x Rule

Sample Associated with EB-T03		Sample Associated with EB-T04	
	Zinc ug/L		Zinc ug/L
Equipment Blank EB-T03 Result	110	Equipment Blank EB-T04 Result	280
Sample MA14G7 Result	720	Sample MA11A4 Result	1270
CRQL	60	CRQL	60
Blank Action Level	550 (5x110)	Blank Action Level	1400 (5x280)
Qualified MA14G7 Result	720	Qualified MA11A4 Result	1270 U

- Equipment blank EB-T03 (Blank Action Level = 550 ug/L) is associated with sample MA14G7. The validator reports the zinc result for sample MA14G7 as unqualified on the Data Summary Table since the result is greater than the blank action level and greater than the quantitation limit.

- Equipment blank EB-T04 (Blank Action Level = 1400 ug/L) is associated with sample MA11A4. The validator reports the zinc result for sample MA11A4 as a non-detect at the reported sample concentration on the Data Summary Table since the result is less than the blank action level and greater than the sample quantitation limit.

The validator notes all actions taken in the Data Validation Memorandum.

E. **EXAMPLES** (continued)

Example #10: (Negative and positive blank results for the same analyte in the same sample)

Barium was detected in the preparation blank at 4 ug/L. Barium was also reported at a negative concentration of -3 ug/L in the initial calibration blank (ICB), less than the negative MDL for barium. Both of these blanks were associated with samples MANN11 and MASS22.

5x Rule

	ug/L
Preparation Blank Result	4
Pos. Blank Action Level	20 (5x4)
ICB Result	-3
Neg. Blank Action Level	15 (5x | -3 |)
CRQL	10
Barium Sample MANN11 Result	12
Qualified Barium Sample MANN11 Result	12 UJ
Barium Sample MASS22 Result	9 J
Qualified Barium Sample MASS22 Result	10 UJ

- Sample MANN11: The barium sample quantitation limit is elevated to the sample concentration due to positive blank actions (12 U); and the sample quantitation limit is then estimated (UJ) due to negative blank actions. Therefore, professional judgment is used to report barium in sample MANN11 as an estimated non-detect, 12 UJ, on the Data Summary Table.

- Sample MASS22: Barium in sample MASS22, which was reported at less than the sample quantitation limit, is reported at the sample quantitation limit due to positive blank actions (10 U). The sample quantitation limit is then estimated (UJ) for negative blank actions. Therefore, professional judgment is used to report barium in sample MASS22 as estimated at the sample quantitation limit, 10 UJ, on the Data Summary Table.

V. ICP-AES INTERFERENCE CHECK SAMPLE (ICS)

A. OBJECTIVE

Interference Check Samples (ICSs) in ICP-AES are analyzed to verify the instrument's ability to correct for spectral interferences using correction factors that correct for interelement contributions. Interference Check Samples contain known concentrations of interferents that will provide a test of the interelement correction factors. These interelement correction factors are unique to each instrument and its operating conditions and are determined experimentally.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. Interelement correction factors must be determined for each ICP-AES instrument at the method-required frequency and, at a minimum, annually.

2. a. The ICS solutions must contain interferents and analytes specified in the method. Typically, the ICS consists of two solutions. The ICSA solution contains the interferents, and the ICSAB solution contains the analytes mixed with the interferents.

   b. The ICS solutions should be from a source providing certified solutions, or prepared by the laboratory at interferent and analyte concentrations specified in the method.

   c. The ICS solutions must be analyzed at the method-required frequency and, minimally, once within each analytical run after the initial calibration verification (ICV) but prior to sample analysis.

3. a. The results of all target analytes in the ICSAB solution must be within 80-120 percent of the ICSAB true value.

   b. The results of all target analytes in the ICSA solution must be within 80-120 percent of the ICSA true value or within the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from ICS anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that interelement correction factors were determined for each ICP-AES instrument at the frequency specified in the method and at least annually.	1. If interelement correction factors were not determined at the proper frequency, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected. The validator should take into consideration the results of the ICS.
2. a. Verify that the ICSA and ICSAB solutions contain the method-required target analytes and interferents. Common interferents aluminum, iron, calcium, and magnesium should be in the ICS solutions. Other interferents may be included in the ICS as required by the method. b. Verify that the ICS solutions were obtained from a source providing certified solutions. If the ICS solutions were prepared by the laboratory, verify that the method-required concentrations of analytes and interferents were used. c. Verify that the ICS solutions were analyzed at the method-required frequency and, minimally, once within each analytical run. The ICS solutions should be analyzed at the beginning of the run after the ICV but before sample analysis.	2. If the ICS solutions were not analyzed for the required analytes and interferents at the correct frequency or were not obtained from a certified source, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected. A discussion of the rationale for data qualification and qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
	Note: **In general, action regarding unacceptable ICSA and ICSAB results is taken when interferents in the ICS are found in the sample at concentrations greater than 50% of their respective concentrations in the ICS. Professional judgment should be used to apply actions when more than one interferent is present in the sample. Generally, the interferent yielding the greatest interference effect is used as the worst case scenario. Generally, no action is taken if the sample contains interferents at concentrations less than or equal to 50% of their respective concentrations in the ICS.**
3. Evaluate the recoveries of all analytes in the ICSAB solution. Verify that all ICSAB recoveries are within 80-120% of the ICSAB true value.	3. a. If any ICSAB percent recovery is greater than 120% but less than or equal to 150%, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS. ii. Accept non-detects for the affected analyte in all samples associated with the ICS.

C. EVALUATION	D. ACTION
3. Continued from above.	3. b. If any ICSAB percent recovery is greater than 150%, then the validator should reject (R) positive detects and accept non-detects for the affected analyte in all samples associated with the ICS. Professional judgment may be used to estimate (J) positive detects, taking into consideration project DQOs. c. If any ICSAB percent recovery is less than 80% but greater than or equal to 50%, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS. ii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICS. d. If any ICSAB percent recovery is less than 50%, then the validator should reject (R) positive detects and non-detects for the affected analyte in all samples associated with the ICS.

C. EVALUATION	D. ACTION
Note: **The CLP SOW ILM05.4 ICS method QC acceptance criteria differ from the Region I Functional Guidelines ICS criteria. If data quality objectives allow for greater variability of data, then expanded ICS validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP. If greater variability is allowed, then the validator should ensure that there is sufficient QC data to support the use of the expanded criteria.**	**Note:** **The validator should evaluate the magnitude of the interference effects caused by the levels of interferents in the ICS compared to the potential estimated interference effect which may be caused by the levels of interferents in the sample. In some cases, the validator may determine that the level of interferent present in the sample does not warrant data qualification if the respective estimated interference effect is deemed not to have an impact on sample results. Generally, if the estimated interference is less than 10% of the sample concentration of the affected analyte, then no action is applied and the sample result is accepted without qualification. If the estimated interference is greater than 10% of the reported sample concentration of the affected analyte, then validation actions apply. Certain circumstances may warrant rejection (R) of the data if it cannot be determined whether or not the sample result is due entirely to interferences or if the validator determines that estimated interferences comprise greater than 90% of the sample result. Professional judgment should be used to reject, qualify, or accept sample results. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.**
4. a. Evaluate the results of all analytes in the ICSA solution. Verify that the ICSA result is within 80-120% of the ICSA true value or within the ICSA true value ± 2x the MDL, whichever range of control limits is greater.	4. a. For any ICSA result, positive or negative, which is not within 80-120% if the ICSA true value or within the ICSA true value ± 2x the MDL, whichever range is greater, the following actions should be taken: i. If any ICSA result is greater than 120% of the ICSA true value or is greater than the ICSA true value plus 2x the MDL, whichever value is greater, then the validator should: • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential high bias.

C. EVALUATION	D. ACTION
4. a. Continued from above.	4. a. i Continued from above. • Accept non-detects for the affected analyte in all samples associated with the ICSA. ii. If any ICSA result (positive or negative) is less than 80% of the ICSA true value or is less than the ICSA true value minus 2x the MDL, whichever value is lower, then the validator should: • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential low bias. • Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICSA.
b. Evaluate the extent of the deviation of the ICSA result from the ICSA true value for the analyte.	b. Depending upon the extent of deviation of the ICSA result, the validator may use professional judgment to take further action, taking into consideration the project DQOs. If may be necessary to reject (R) positive detects and/or non-detects for the affected analyte in samples associated with the ICS.

C. EVALUATION	D. ACTION
4. c. For any ICSA analyte which does not meet criteria, evaluate whether or not the ICSAB criteria were met.	4. c. If any analyte does not meet acceptance criteria for the ICSA but meets ICSAB criteria, then the validator may use professional judgment to qualify only those sample results in the affected concentration range, taking into consideration the project DQOs. For example, if a false positive/high bias or false negative/low bias is observed in the ICSA, but the ICSAB result for the analyte is within 80-120% recovery criteria, then the validator may use professional judgment to qualify only those sample results less than the ICSAB true value and accept the sample results greater than or equal to the ICSAB true value. A discussion of the rationale for data qualification or data acceptance should be documented in the Data Validation Memorandum. d. The validator should review the sample data to determine if other analytes which may act as potential interferents are present in the sample at greater than 10 mg/L. Possible interference effects, as documented in EPA methods, including the CLP method, and as indicated by the laboratory's reported interelement correction factors for that particular instrument should be reviewed. The analyte concentration equivalents presented in the methods should only be used as estimated values since the exact value of any analytical system is instrument-specific. Professional judgment should be used to qualify sample data if the validator suspects interference effects other than those arising from the interferents in the ICS.

C. EVALUATION	D. ACTION
4. Continued from above.	4. e. Evaluating ICS data is not necessarily straightforward. When multiple ICS analyses exist within an analytical run, the validator may use professional judgment to apply actions only to those samples bracketed by a particular ICS or to apply actions from the worst case scenario to all samples within that run. If multiple ICS analyses within an analytical run exhibit both positive and negative values for a particular analyte, then the validator should use professional judgment in qualifying sample results. It may help to evaluate the results of blanks analyzed within the same run to determine whether the positive or negative ICS values may be due to erratic responses also exhibited in blanks. f. Actions regarding the interpretation and/or the qualification of sample results due to ICS results can be complex. The validator should use professional judgment in determining the need to accept, qualify or reject the associated sample data based on the ICS results and project DQOs. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*5. a. Check raw data to verify that ICS results are accurately reported on the tabulated forms. Confirm that results equal to or below the negative MDL are also reported on the forms. b. Check and recalculate the ICS percent recovery for at least one analyte per each pair of ICSA/ICSAB. Verify that the recalculated value agrees within ±10% of the reported value.	5. If there are any transcription and/or calculation errors, then the validator should contact the laboratory to obtain corrected raw data and forms. If errors greater than 10% are detected, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.5

Table INORG-V-1:

QUALIFICATION OF ICP-AES ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSAB RECOVERIES

Sample Results	ICSAB Recoveries				
	%R < 50%	50% ≤ %R < 80%	80% ≤ %R ≤ 120%	120% < R ≤ 150%	%R > 150%
Detects	R	J	A	J	R*
Non-detects	R	UJ	A	A	A

* Professional judgment may be used to estimate (J) positive detects, taking into consideration project DQOs.
Note: Generally, action is applied when interferents are present in samples at greater than 50% of their respective levels in the ICS.

Table INORG-V-2:

QUALIFICATION OF ICP-AES ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSA RESULTS

Sample Results	ICSA Concentration or % Recovery*		
	%R < 80% or Conc. < TV-(2xMDL)	%R = 80-120% or Conc. = TV±(2xMDL)	%R > 120% or Conc. > TV+(2xMDL)
Detects	J	A	J
Non-detects	UJ	A	A

TV = ICSA True Value
* ICSA criteria are based on either 80-120% of the ICSA true value or the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

Note: Generally, action is applied when interferents are present in samples at greater than 50% of their respective levels in the ICS. Generally, no action is taken when the estimated interference is less than 10% of the sample result of the affected analyte. Professional judgment may be used to reject the data if the estimated interference comprises greater than 90% of the sample result. If ICSAB recovery criteria are met, then professional judgment may be used to qualify only those sample results in the affected concentration range.

E. EXAMPLES

Example #1: (Low ICSAB recovery)

The ICSAB recovery for antimony was 70.5%. All samples associated with the ICSAB contained levels of aluminum, calcium, iron, and magnesium which were less than 50% of their respective levels in the ICSAB, with the exception of sample MAGF02 which contained iron at a concentration greater than 50% of the level detected in the ICSAB solution. Antimony was not detected in sample MAFG02. The validator estimates (UJ) the antimony non-detect on the Data Summary Table to indicate the possibility of a false negative. The validator notes the problem in the Data Validation Memorandum.

Example #2: (Cadmium ICSA TV is 0; ICSA result > ICSA TV ± 2x the MDL)

The ICSA true value for cadmium is 0. The MDL for cadmium is 2 ug/L. Cadmium was reported in the ICSA at 6 ug/L which exceeds the criteria of the ICSA true value ± 2x the MDL (0 ± 4 ug/L). All samples associated with the ICSA contained levels of interferents that were less than 50% of their respective levels in the ICSA, with the exception of samples MACB01 and MACB02. Iron concentrations in these two samples were greater than 50% of that in the ICSA.

- Sample MACB01 contains cadmium at 10 ug/L. Since 100,000 ug/L of iron yielded 6 ug/L of cadmium in the ICSA, 70,000 ug/L of iron in sample MACB01 is expected to yield a potential estimated interference of 4 ug/L. The validator estimates (J) the positive cadmium result in sample MACB01 on the Data Summary Table to indicate potential high bias.

- Sample MACB02 contains cadmium at 50 ug/L. The iron concentration of 60,000 ug/L in sample MACB02 is expected to yield a potential estimated interference of 3.6 ug/L which is less than 10% of the reported cadmium concentration in the sample. The validator accepts the positive cadmium result in sample MACB02.

The validator discusses all sample qualifications in the Data Validation Memorandum.

E. EXAMPLES (continued)

Example #3: (High lead ICSA result)

The ICSA true value for lead is 15 ug/L. The MDL for lead is 4 ug/L. The validator uses the criteria of the ICSA true value ± 2x the MDL (7-23 ug/L) rather than 80-120% recovery (12-18 ug/L) in evaluating the ICSA data since ± 2x the MDL yields wider criteria. Lead was detected in the ICSA at 39 ug/L which exceeds the ICSA true value ± 2x the MDL criteria. All samples associated with the ICSA contained levels of aluminum, calcium, iron, and magnesium which were less than 50% of their respective levels in the ICSA, with the exception of three samples. Samples MAGF02, MAGF04, and MAGF07 contained concentrations of aluminum which were greater than 50% of that in the ICSA.

- Lead was not detected in sample MAGF02. The validator accepts the lead non-detect in sample MAGF02.

- Sample MAGF04 contains lead at 150 ug/L. Since 250,000 ug/L of aluminum yielded 24 ug/L above the true value of lead in the ICSA, 150,000 ug/L of aluminum in sample MAGF04 is expected to yield a potential estimated interference of approximately 14.4 ug/L. Since the estimated interference comprises less than 10% of the reported sample result of 150 ug/L, no action is taken and the validator accepts the positive lead result on the Data Summary Table.

- Sample MAGF07 contains lead at 20 ug/L. Since 250,000 ug/L of aluminum yielded 24 ug/L above the true value of lead in the ICSA, 200,000 ug/L of aluminum in sample MAGF07 is expected to yield a potential estimated interference of approximately 19.2 ug/L. Therefore, the validator rejects (R) the positive lead results in sample MAGF07 on the Data Summary Table since the result is suspected of being due entirely to interference.

The validator discusses all sample qualifications in the Data Validation Memorandum.

Example #4: (Antimony ICSA TV is 0; Negative antimony ICSA result)

The ICSA true value for antimony is 0. The antimony MDL is 14 ug/L. Antimony was reported in the ICSA at -41 ug/L, outside the criteria of the ICSA true value ± 2x the MDL (0 ± 28 ug/L). The levels of interferents aluminum, iron, calcium, and magnesium in the samples associated with the ICSA are below 50% of their respective levels in the ICSA solution with the exception of iron in samples MAGF02 and MAGF03, which was present in these samples at levels greater than 50% of that in the ICSA.

- Antimony was reported as a non-detect in sample MAGF02. The validator estimates (UJ) the antimony non-detect in sample MAGF02 on the Data Summary Table due to the possibility of a false negative.

- Sample MAGF03 contains antimony at 65 ug/L. Since 100,000 ug/L iron yielded -41 ug/L of antimony in the ICSA, 80,000 ug/L of iron in sample MAGF03 is expected to yield a potential estimated interference of -33 ug/L. Therefore, the validator estimates (J) the positive antimony result in sample MAGF03 on the Data Summary Table and notes the possibility of low bias.

The validator discusses all sample qualifications in the Data Validation Memorandum.

VI. ICP-MS INTERFERENCE CHECK SAMPLE (ICS)

A. OBJECTIVE

ICP-MS Interference Check Samples (ICSs) contain interfering elements that provide a test of the adequacy of the measurement system to correct for isobaric interferences. Interference Check Samples are analyzed to determine the existence and magnitude of interferences and to verify the instrument's ability to correct for those interferences.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. a. The ICS solutions must contain interferents and analytes specified in the method. Typically, the ICS consists of two solutions. The ICSA solution contains the interferents, and the ICSAB solution contains the analytes mixed with the interferents.

   b. The ICS solutions should be from a source providing certified solutions or prepared by the laboratory at interferent and analyte concentrations specified in the method.

   c. The ICS must be analyzed at the method-required frequency and, minimally, once within each analytical run after the initial calibration verification (ICV) but prior to sample analysis.

2. a. The results of all target analytes in the ICSAB solution must be within 80-120 percent of the ICSAB true value.

   b. The results of all target analytes in the ICSA solution must be within 80-120 percent of the ICSA true value or within the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from ICS anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Verify that the method-required ICSA and ICSAB solutions were analyzed at the proper frequency and, minimally, once within each analytical run.	1. a. If the ICS solutions were not analyzed at the method-required frequency, then the validator should use professional judgment to assess the impact of potential interferences on data quality. A discussion of the rationale for data qualification of the qualifiers used should be documented in the data validation memorandum.
* b. Verify that the ICS solutions were obtained from a source providing certified solutions. If the ICS solution was prepared by the laboratory, verify that the required concentrations of analytes and interferents were used. Note: The tabulated forms may not contain results for all ICS interferents. It may be necessary to review the raw data (instrument raw data or standards preparation logs) to confirm that the proper components were used to prepare the ICS solutions. Note that monitoring the interference source in ICP-MS analysis does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent.	b. If the ICS solution was not obtained from a source providing certified solutions or if the required analytes and interferents were not used to prepare the ICS solutions at the required concentrations, then the validator should use professional judgment to determine if sample qualification is required.

C. EVALUATION	D. ACTION
2. Evaluate the recoveries of all analytes in the ICSAB solution. Verify that all ICSAB recoveries are within 80-120% of the ICSAB true value.	2. a. If any ICSAB percent recovery is greater than 120% but less than or equal to 150%, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS. ii. Accept non-detects for the affected analyte in all samples associated with the ICS. b. If any ICSAB percent recovery is greater than 150%, then the validator should use professional judgment to estimate (J) or reject (R) positive detects and accept non-detects for the affected analyte in all samples associated with the ICS. c. If any ICSAB percent recovery is less than 80% but is greater than or equal to 50%, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS. ii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICS. d. If any ICSAB percent recovery is less than 50%, then the validator should reject (R) positive detects and non-detects for the affected analyte in all samples associated with the ICS.

C. EVALUATION	D. ACTION
Note: **The CLP SOW ILM05.4 ICS method QC acceptance criteria differ from the Region I Functional Guidelines ICS criteria. If data quality objectives allow for greater variability of data, then expanded ICS validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP. If greater ICS recoveries are allowed, then the validator should ensure that there is sufficient QC data to support the use of the expanded criteria.**	
3. a. Evaluate the results of all analytes in the ICSA solution. Verify that the ICSA result is within 80-120% of the ICSA true value or within the ICSA true value ± 2x the MDL, whichever range of control limits is greater.	3. a. For any ICSA analyte result, positive or negative, which is not within 80-120% of the ICSA true value or within the ICSA true value ± 2x the MDL, whichever range is greater, the following actions should be taken: i. If any ICSA result is greater than 120% of the ICSA true value or is greater than the ICSA true value plus 2x the MDL, whichever value is greater, then the validator should: • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential high bias. • Accept non-detects for the affected analyte in all samples associated with the ICSA.

C. EVALUATION	D. ACTION
3. a. Continued from above.	3. a. ii. If any ICSA result (positive or negative) is less than 80% of the ICSA true value or is less than the ICSA true value minus 2x the MDL, whichever value is lower, then the validator should: • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential low bias. • Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICSA.
b. Evaluate the extent of the deviation of the ICSA result from the ICSA true value for the analyte.	b. Depending upon the extent of the deviation of the ICSA result, the validator may use professional judgment to take further action, taking into consideration the project DQOs. It may be necessary to reject (R) positive detects and/or non-detects for the affected analyte in samples associated with that ICS.
c. For any ICSA analyte which does not meet criteria, evaluate whether or not the ICSAB criteria were met.	c. If any analyte does not meet acceptance criteria for the ICSA but meets ICSAB criteria, then the validator may use professional judgment to qualify only those sample results in the affected concentration range, taking into consideration the project DQOs. For example, if a false positive/high bias or false negative/low bias is observed in the ICSA, but the ICSAB result for the analyte is within recovery criteria, then the validator may use professional judgment to qualify only those sample results less than the ICSAB true value and accept the sample results greater than or equal to the ICSAB true value. A discussion of the rationale for data qualification or data acceptance should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. Continued from above.	3. d. Actions regarding the interpretation and/or the qualification of sample results due to ICS results can be complex. The validator should use professional judgment in determining the need to accept, qualify or reject the associated sample data based on the ICS results and project DQOs. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*4. a. Check raw data to verify that ICS results are accurately reported on the tabulated forms. Confirm that results equal to or below the negative MDL are also reported on the forms. b. Check and recalculate the ICS percent recovery for at least one analyte per each pair of ICSA/ICSAB. Verify that the calculated value agrees within ±10% of the reported value.	4. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.4

Table INORG-VI-1:

QUALIFICATION OF ICP-MS ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSAB RECOVERIES

Sample Results	ICSAB Recoveries				
	%R < 50%	50% ≤ %R < 80%	80% ≤ %R ≤ 120%	120% < %R ≤ 150%	%R > 150%
Detects	R	J	A	J	R*
Non-detects	R	UJ	A	A	A

* Professional judgment should be used to estimate (J) positive detects, taking into consideration project DQOs.

Table INORG-VI-2:

QUALIFICATION OF ICP-MS ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSA RESULTS

Sample Results	ICSA Concentration or % Recovery*		
	%R < 80% or Conc. < TV-(2xMDL)	%R = 80-120% or Conc. = TV±(2xMDL)	%R > 120% or Conc. > TV+(2xMDL)
Detects	J	A	J
Non-detects	UJ	A	A

TV = ICSA True Value

* ICSA criteria are based on either 80-120% of the ICSA true value or the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

Note: Professional judgment may be used to reject (R) positive detects and/or non-detects for the affected analytes.

E. EXAMPLES

Example #1: (One low ICSAB recovery)

The validator notes that the ICSAB recovery for cadmium is 70.5%. In the associated samples, cadmium was detected in samples MAFG02 through MAFG05 and was a non-detect in sample MAFG06. Therefore, the validator estimates (J) the positive detects for cadmium and estimates (UJ) the cadmium non-detect in the samples associated with the ICSAB. The validator reports the qualified results on the Data Summary Table and notes that the detected cadmium results are biased low and the cadmium non-detect contains a possible false negative in the Data Validation Memorandum.

Example #2: (One high ICSA result; ICSAB within acceptance limits)

The observed ICSA concentration for manganese is 9.0 ug/L. The ICSA true value for manganese is 7.0 ug/L. The MDL for manganese is 0.3 ug/L. The validator uses the 80-120% R criteria (5.6 to 8.4 ug/L) rather than the ICSA true value ± 2x the MDL criteria (6.4 to 7.6 ug/L) since the recovery criteria gives wider control limits. The ICSA for manganese recovered at 129%, outside the acceptance limit. In the associated samples, manganese was detected in samples MAGF02 through MAGF04 and was a non-detect in samples MAGF05 through MAGF07. The ICSAB result for manganese was within 80-120% R criteria. Therefore, the validator accepts the manganese non-detects and uses professional judgment to estimate (J) only those positive detects less than the ICSAB true value since manganese was within criteria for the ICSAB.

Example #3: (One negative ICSA result)

The ICSA true value for arsenic is 0 and the arsenic MDL is 0.5 ug/L. The validator notes that arsenic in the ICSA is reported as -2.0 ug/L, which is outside the ICSA true value ± 2x the MDL criteria (-1.0 to 1.0 ug/L). In the associated samples, arsenic was detected in samples MAFG01 and MAFG02, and was a non-detect in sample MAFG03. The ICSAB result for arsenic was within the 80-120% recovery criteria. Therefore, the validator estimates (UJ) the arsenic non-detect in sample MAFG03, and uses professional judgment to estimate (J) only those positive detects less than the ICSAB true value for samples MAFG01 and MAFG02 since arsenic was within criteria for the ICSAB. The validator reports the qualified data on the Data Summary Table and notes in the Data Validation Memorandum that the qualified positive results are biased low and the arsenic non-detect may be a possible false negative.

VII. ICP-MS INTERNAL STANDARDS

A. OBJECTIVE

Instrument performance and stability, physical/sample matrix interferences, and laboratory precision throughout an analytical sequence are monitored by the addition of internal standard analytes. Internal standards (ISs) are added to every field sample, QC sample, standard and blank at identical levels to determine the existence and magnitude of instrument drift and physical interferences. Evaluation of the behavior of internal standards is not necessarily straightforward. Interfering sample matrix effects are frequently outside of the laboratory's control and may adversely affect the analysis of internal standards.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. The internal standard analytes specified in the method must be added to all samples, QC samples, standards and blanks at the required concentrations. An appropriate internal standard analyte is required for each target analyte determined by ICP-MS. Generally, the atomic mass of each internal standard is within 50 amu of the mass of its associated target analyte.

2. The intensities of the internal standards must be monitored throughout the analytical run and compared to their respective intensities in the blank calibration standard (from the initial calibration). Internal standard intensities must be within the method QC acceptance criteria.

3. Samples must be reanalyzed at the method-required dilutions if internal standard method QC acceptance criteria are not met.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from internal standard anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct internal standard analytes were added to all samples, QC samples, standards and blanks at the method-specified concentrations. (Internal standards are not added to the tuning solution.) Verify that there is an internal standard associated with each target analyte.	1. If the laboratory did not add the required internal standard analytes to all samples, QC samples, standards and blanks at the correct concentrations, or if there is no internal standard associated with each target analyte, then the validator must use professional judgment to determine how the associated sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Verify that all IS percent relative intensities are within the method QC acceptance criteria.	2. a. If the percent relative intensity of an IS is not within the method QC acceptance criteria, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects for the analytes associated with that IS in the affected sample. b. If internal standard performance exhibits a major deviation from method QC criteria, indicating a severe loss of sensitivity or possible interferences, then the validator may use professional judgment to reject the associated sample data. Other relevant QC information should be taken into account. The rationale for data qualification and the qualifiers used should be discussed in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. a. Verify that if any internal standard relative intensity is outside the method QC acceptance criteria, then the method-required reanalysis was performed on the diluted sample with the appropriate amounts of internal standards and/or the method-required recalibration was performed. b. If there are two analyses for a particular sample and analyte, then the validator must determine which data to report. Considerations should include but are not limited to: - Magnitude of the IS intensity shift; - Comparison of the values of the target analytes reported in each analysis; - Other relevant QC; - Project DQOs.	3. a. If a laboratory fails to reanalyze a sample with an internal standard that is outside the method QC acceptance criteria or fails to recalibrate the instrument, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/ contractual deficiency in the Data Validation Memorandum. b. If a sample has been analyzed and reported more than once, then the validator should use professional judgment when considering which analysis or portion of an analysis to report. The validator must consider all relevant QC information in making a decision. Generally, the following actions should be taken: i. If the relative intensity of an internal standard in the diluted sample analysis does not meet the method QC acceptance criteria, then the data from the original undiluted sample analysis should be reported qualified according to the guidelines above. ii. If the relative intensity of an internal standard in the diluted sample analysis meets the method QC acceptance criteria, then the data from the diluted sample analysis should be reported without qualification. iii. The validator should review the project DQOs and determine which sample result better achieves project objectives. The validator should document and discuss all technical decisions made based on professional judgment in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. c. Verify that all IS percent relative intensities in the calibration verification solutions (ICV/CCV) and calibration blanks (ICB/CCB) are within 80-120%.	3. c. Evaluate the percent relative intensities of internal standards in the ICV/CCV and ICB/CCB QC samples. If the percent relative intensity of an IS in any of these QC samples is outside 80-120% of the percent relative intensity of the IS in the blank calibration standard, then this may indicate an instrumental problem. Professional judgment should be used to evaluate the impact of the QC sample IS results on the IS results in the field samples. A discussion of the rationale for all data qualifications should be documented in the Data Validation Memorandum.
*4. Check raw data to verify that IS relative intensities are accurately reported on the tabulated forms. Recalculate the internal standard percent relative intensities for each internal standard in at least one sample. Verify that the recalculated value agrees within ±10% of the reported value.	4. If errors greater than 10% are detected in the percent relative intensity calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.4

Table INORG-VII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON INTERNAL STANDARD RELATIVE INTENSITIES

Sample Results	Internal Standard Relative Intensities (RI)*		
	% RI < LL	LL ≤ % RI ≤ UL	% RI > UL
Detects	J	A	J
Non-detects	UJ	A	UJ

LL = Lower Limit of method QC acceptance criteria
UL = Upper Limit of method QC acceptance criteria
* Professional judgment may be used to reject data for severe loss of sensitivity.

E. EXAMPLES

Example #1: (Instrument drift; Sample IS % RI < LL of method QC acceptance criteria)

The validator reviews the IS % relative intensities for samples analyzed by CLP SOW ILM05.4 and notes that the yttrium response decreases over time and the response in sample MAAB04 is 40%, below the lower method QC acceptance limit of 60% of the response in the blank calibration standard. Upon review of the data, the validator determines that the laboratory did not perform the required reanalysis of the sample at a two-fold dilution. The validator ascertains from the data that arsenic (mass 75), selenium (mass 82), and zinc (mass 66) were associated with the yttrium IS (mass 89). Therefore, on the Data Summary Table, the validator estimates (J) positive detects and estimates (UJ) non-detects in sample MAAB04 for arsenic, selenium, and zinc. The validator discusses the instrument's loss in sensitivity, the sample qualifications, and the laboratory's deviation from the method in the Data Validation Memorandum.

Example #2: (Sample IS % RI > UL of method QC acceptance criteria; Acceptable IS % RI from two-fold dilution)

The validator reviews the IS % relative intensities for samples analyzed by CLP SOW ILM05.4 and notes that the rhodium and indium IS intensities increase over time. In addition, the validator notes that sample MA1GN8 was reported from a two-fold dilution. The QC data indicate that the IS % relative intensities for rhodium and indium for sample MA1GN8 in the original undiluted sample analysis are above the upper method QC acceptance limit of 125% of the IS response in the calibration blank. The laboratory performed the method-required reanalysis of the sample at a two-fold dilution with the rhodium and indium % relative intensities meeting the method QC acceptance criteria. The validator reports the sample results from the two-fold dilution without qualification and with the raised quantitation limits for non-detects on the Data Summary Table and discusses the reason for reporting sample results from the diluted sample analysis in the Data Validation Memorandum.

VIII. MATRIX SPIKES

A. OBJECTIVE

Data for matrix spikes are generated to determine method bias for specific sample matrices at the time of sample preparation and analysis. Matrix spike data can be used to determine long-term inter-laboratory bias of an analytical method for various matrices and are used in setting quality control acceptance criteria for spiking compounds.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. In accordance with the SAP, QAPP and/or method, a field sample of each matrix is spiked with known concentrations of specific target analytes to generate a matrix spike sample. Concurrently, the laboratory analyzes an unspiked aliquot and the matrix spike of the field sample.

2. a. Field samples (not equipment or bottle blanks and not PE samples) must be spiked to assess matrix effects.

   b. Field samples chosen for matrix spike analysis should not contain high levels of matrix spiking analytes prior to spiking. Preferably, field samples chosen for matrix spike analysis should contain low levels of the spiking analytes.

3. Spike recoveries must be within the QC acceptance criteria specified in the method, SAP, or QAPP.

4. If a post-digestion spike analysis is required by the method, SAP, or QAPP for analytes whose matrix spike recoveries are not within method QC acceptance criteria, then an unspiked aliquot of the prepared field sample chosen for matrix spike analysis is spiked with known concentrations of specific target analytes. The concentration and acceptance criteria for the post-digestion spike analysis should be stated in the method, SAP, or QAPP.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from matrix spike sample analysis anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct analytes were added to the sample at the required concentrations, that matrix spike samples were analyzed at the proper frequency, and that matrix spike results are provided for each sample matrix. If more than one analytical method was used to report sample results for an analyte, then verify that matrix spike results for that analyte are provided by each method.	1. If the laboratory did not use the required analytes at the concentration and frequency specified in the method for each sample matrix and method, then the validator must use professional judgment to determine whether the associated sample data should be qualified.
2. a. Verify that a field sample was chosen for the matrix spike.	2. a. If an equipment blank, a bottle blank, or a PE sample was used for the matrix spike sample, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing method bias, sample matrix effects and, ultimately, data usability.
b. Determine if an inappropriate sample containing high levels of the spiking analytes was chosen for the matrix spike.	b. If the matrix spike analytes were present in the field sample at high concentrations (e.g., 4x the spike concentration) before spiking, then the validator must use professional judgment in assessing matrix spike recoveries. Generally, spike recovery limits do not apply when the sample concentration exceeds the spike concentration by a factor of 4 or more.
c. Ascertain if the matrix spike sample required dilutions.	c. If no matrix spike data can be reported because of sample dilution, then the validator should note this problem in the Data Validation Memorandum and discuss the impact on assessing data usability in the case where method bias information is absent.

C. EVALUATION	D. ACTION
	Note: Action applies to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method.
3. Verify that all spike recoveries are within the QC acceptance criteria specified in the method.	3. a. If any spike recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Accept non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. b. If any recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 30%, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. c. If any recovery is less than 30%, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Reject (R) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method.

C. EVALUATION	D. ACTION
3. Continued from above.	3. d. If the majority of spike analyte recoveries for a method are outside the method QC acceptance criteria, then the validator may use professional judgment to estimate or reject all positive detects and non-detects in all samples of the same matrix prepared and analyzed by the same method.
4. Verify that a post-digestion spike sample was analyzed at the proper frequency, that the correct analytes were added at the required concentrations, and that post-digestion spike results are provided in accordance with method requirements if matrix spike method QC acceptance criteria are not met.	4. Generally, no action is taken based solely on the post-digestion spike result. However, in some cases, post-digestion spike data may aid in evaluating matrix interferences. The data validator should use professional judgment to qualify sample data based on post-digestion spike results. A discussion of any possible impacts on the data should be included in the Data Validation Memorandum.
*5. Check and recalculate the analytical concentrations and percent recovery for at least one spiked analyte per method. Verify that the recalculated value agrees within ±10% of the reported value.	5. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should contact the laboratory to requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. Evaluate the appropriateness of not qualifying the entire data set based on method/matrix bias results.	6. Generally, action based on the matrix spike results is applied to the affected analyte in all samples of the same matrix prepared and analyzed by the same method in a sample delivery group. However, professional judgment may be used to apply the action only to a specific subset of samples of the same matrix or to the matrix spike sample if there is information to support such an action. All justifications for not qualifying data should be documented in the Data Validation Memorandum and the potential impact on data usability in meeting the project DQOs should be discussed.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.5

Table INORG-VIII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON MATRIX SPIKE RECOVERIES*

Sample Results	% Recovery			
	%R < 30%	30% ≤ %R < LL	LL ≤ %R ≤ UL	%R > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL = Lower Limit of method QC acceptance criteria
UL = Upper Limit of method QC acceptance criteria
* Qualification is applied to the affected analyte in all samples of the same matrix analyzed by the same method; however, the validator may use professional judgment to apply actions to all positive detects and non-detects if the majority of spike analyte recoveries are outside method QC acceptance criteria.

E. EXAMPLES

Example #1: (High matrix spike recovery for one analyte)

Aqueous QC sample MADG56MS, analyzed by ICP-AES under CLP SOW ILM05.4, has a high matrix spike recovery result for zinc.

Sample No.	Analyte	MS % Rec.	MS % Rec. Criteria	Post-digestion Spike % Rec.
MADG56	Zinc	137	75-125	132

The validator concludes that the sample matrix causes a positive bias for zinc in all aqueous samples associated with this sample delivery group. The validator estimates (J) positive detects for zinc in all aqueous samples on the Data Summary Table. The validator discusses the high matrix spike recovery in the Data Validation Memorandum and notes that the post-digestion spike recovery for zinc was also high, confirming a matrix interference, and that recoveries for the other matrix spike analytes were acceptable.

Example #2: (Extremely low matrix spike recovery for one analyte)

Soil QC sample MAFG77MS, analyzed by ICP-AES under CLP SOW ILM05.4, has an extremely low antimony matrix spike recovery result.

Sample No.	Analyte	MS % Rec.	MS % Rec. Criteria	Post-digestion Spike % Rec.
MAFG77	Antimony	18	75-125	43

The validator concludes that the sample matrix causes a negative bias for antimony in all soil samples associated with this sample delivery group. The validator estimates (J) positive detects and rejects (R) non-detects for antimony in all soil samples on the Data Summary Table. The validator discusses the low matrix spike recovery in the Data Validation Memorandum and notes that the post-digestion spike recovery for antimony was also low, confirming a matrix interference, and that recoveries for the other matrix spike analytes were acceptable.

E. EXAMPLES (Continued)

Example #3: (One analyte analyzed by two methods; Low matrix spike recovery for one method, acceptable matrix spike recovery for the other method)

Aqueous samples in an SDG were analyzed for lead under CLP SOW ILM05.4. Some lead sample results were reported from the ICP-AES analysis and other lead sample results were reported from the ICP-MS analysis. Aqueous QC sample MAAG23MS has a low matrix spike recovery for lead analyzed by ICP-MS and an acceptable recovery for lead analyzed by ICP-AES.

Sample No.	Analyte	Method	MS % Rec.	MS % Rec. Criteria	Post-digestion Spike % Rec.
MAAG23	Lead	ICP-MS	53	75-125	80
		ICP-AES	87		NA

NA = Not Applicable

The validator concludes that the sample matrix causes a negative bias for lead in all aqueous samples analyzed by ICP-MS in this sample delivery group. The validator estimates (J) positive detects and estimates (UJ) non-detects for lead in all aqueous samples reported from the ICP-MS analysis. The validator accepts all lead results in aqueous samples analyzed by ICP-AES. The validator discusses the low matrix spike recovery and the acceptable post-digestion spike recovery for lead analyzed by ICP-MS in the Data Validation Memorandum and notes that recoveries for lead in aqueous samples analyzed by ICP-AES and the other matrix spike analytes were acceptable.

IX. LABORATORY DUPLICATE SAMPLES

A. OBJECTIVE

Data for laboratory duplicate sample analyses are generated to determine laboratory precision for specific sample matrices at the time of sample preparation and analysis. Duplicate sample analysis data can be used to determine long-term interlaboratory precision of an analytical method for various matrices.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. In accordance with the SAP, QAPP, and/or method, a field sample is prepared and analyzed in duplicate for each matrix.

2. Field samples (not equipment or bottle blanks and not PE samples) must be used to assess laboratory precision.

3. Relative Percent Differences (RPDs) or absolute differences between the laboratory duplicate samples must be within the QC acceptance criteria specified in the method for the specific matrix.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from laboratory duplicate sample anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that laboratory duplicate samples were prepared and analyzed at the proper frequency and that duplicate sample results are provided for each sample matrix. If more than one analytical method was used to report sample results for an analyte, then verify that laboratory duplicate sample results are provided for that analyte by each method.	1. If the laboratory did not prepare and analyze laboratory duplicates at the frequency specified in the method for each sample matrix and method, then the validator must use professional judgment to determine whether the associated sample data should be qualified.
2. Verify that a field sample was chosen for the laboratory duplicate sample.	2. If an equipment blank, a bottle blank, or a PE sample was used for the duplicate sample analysis, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing laboratory precision, sample matrix effects and, ultimately, data usability.
3. Verify that all Relative Percent Differences or absolute differences between the sample and the laboratory duplicate are within the QC acceptance criteria specified in the method for the specific matrix.	Note: Action applies to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method. 3. If any RPD or absolute difference for a laboratory duplicate sample is outside the method QC acceptance criteria, then the validator should: a. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. b. Estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method.

C. EVALUATION	D. ACTION
3. Continued from above. **Note:** **The CLP SOW ILM05.4 laboratory duplicate sample method QC acceptance criteria do not differentiate between aqueous and soil matrices. Because laboratory variability arising from the sub-sampling of non-homogenous soil samples is common, the data validation criteria in Table INORG-IX-3 should be used for non-aqueous matrices. If data quality objectives allow for other criteria to be used, then the validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP.**	3. c. If any analyte is detected at concentrations less than the sample quantitation limit or are non-detects in both aqueous laboratory duplicate samples, then no action is taken. d. If the majority of the laboratory duplicate sample results are outside method QC acceptance criteria, then the validator may use professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects in all samples of the same matrix.
*4. Check and recalculate the analytical concentrations and RPD for at least one duplicate sample per analytical method. Verify that the recalculated value agrees within ±10% of the reported value.	4. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
5. Evaluate the appropriateness of qualifying only the laboratory duplicate sample results or only a subset of samples of the same matrix for the affected analyte. Field duplicate sample data should be evaluated to identify overall precision issues.	5. Generally, action based on the laboratory duplicate sample results is applied to the affected analyte in all samples of the same matrix prepared and analyzed by the same method in a sample delivery group (SDG). If there is information to indicate that the poor laboratory precision is limited to the duplicate sample or to a select group of samples in the SDG, then professional judgment may be used to apply the action only to the field sample used for the laboratory duplicate sample analysis or to a select group of samples in the SDG. All justifications for not qualifying the entire data set should be documented in the Data Validation Memorandum, and the potential impact on data usability in meeting the project DQOs should be discussed.
6. Evaluate laboratory duplicate precision data to confirm the laboratory's ability to generate precise data and field duplicate precision data to assess overall precision.	6. If precision data for the laboratory duplicate sample and field duplicate pair indicate a heterogeneous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify all field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting the project DQOs should be discussed. Refer to Section INORG-X (Field Duplicates) for additional guidance.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.4

Table INORG-IX-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES*

Sample Results	Laboratory Duplicate Sample Results	
	RPD or Abs. Diff. ≤ QC Limit	RPD or Abs. Diff. > QC Limit
Detects	A	J
Non-detects	A	UJ

* If QC acceptance criteria for the specific matrix are specified in the method, then use the method QC criteria in this table. If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in Table INORG-IX-2 or INORG-IX-3.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the method QC acceptance criteria.

Table INORG-IX-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES - AQUEOUS MATRICES*

Sample Results	Aqueous Laboratory Duplicate Sample Results			
	Both Sample and Duplicate ≥ 5xQL		One or Both Sample and Duplicate < 5xQL	
	RPD ≤ 20%	RPD > 20%	Abs. Diff. ≤ QL	Abs. Diff. > QL
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

* If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in this table (from Appendix I). If QC acceptance criteria for the specific matrix are specified in the method, then use the criteria in Table INORG-IX-1 above.

QL = Sample Quantitation Limit

- When applying the absolute difference criteria, the sample quantitation limit for the sample (vs. the duplicate sample) is used.
- No action is applied when both sample and duplicate values are detected at < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the QC acceptance criteria.

Table INORG-IX-3:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES - NON-AQUEOUS MATRICES*

Sample Results	Non-Aqueous Laboratory Duplicate Sample Results			
	Both Sample and Duplicate ≥ 5xQL		One or Both Sample and Duplicate < 5xQL	
	RPD ≤ 35%	RPD > 35%	Abs. Diff. ≤ 2xQL	Abs. Diff. > 2xQL
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

* If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in this table. If QC acceptance criteria for the specific matrix are specified in the method, then use the criteria in Table INORG-IX-1 above.

QL = Sample Quantitation Limit

- When applying the absolute difference criteria, the sample quantitation limit for the sample (vs. the duplicate sample) is used.
- No action is applied when both sample and duplicate values are detected at < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the QC acceptance criteria.

E. EXAMPLES

Example #1: (Both aqueous sample and laboratory duplicate sample concentrations ≥ 5xSQL; RPD > 20%; Poor laboratory precision by ICP-AES; Acceptable laboratory precision by ICP-MS)

Aqueous sample MACH79 and laboratory duplicate sample MACH79D, digested and analyzed by ICP-AES under CLP SOW ILM05.4, have a high RPD for lead. Sample MACH79 has a lead concentration of 60 ug/L and laboratory duplicate sample MACH79D has a lead concentration of 81 ug/L. Both results are greater than 5xSQL with an RPD of 30%.

Analyte (method)	MACH79		MACH79D		RPD	RPD Criteria
	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)		
Lead (ICP-AES)	60	10/ 50	81	10/ 50	30%	20%

The SDG also contained some samples which were analyzed for lead by ICP-MS. ICP-MS aqueous sample MACJ07 has a lead concentration of 9.3 ug/L and laboratory duplicate sample MACJ07D has a lead concentration of 10.5 ug/L. Both results are greater than 5xSQL with an RPD of 12%.

Analyte (method)	MACJ07		MACJ07D		RPD	RPD Criteria
	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)		
Lead (ICP-MS)	9.3	1/ 5	10.5	1/ 5	12%	20%

Laboratory precision for lead is acceptable for the ICP-MS analysis but did not meet method QC criteria by ICP-AES analysis. The validator evaluates the field duplicate pair analyzed by ICP-AES and determines that the RPDs and absolute differences are within the field duplicate QC criteria indicating acceptable overall precision for this sampling event. The validator then concludes that the poor laboratory precision for lead in this sample is specific to the ICP-AES analysis. The validator estimates (J) positive detects and estimates (UJ) non-detects for lead in all aqueous samples reported from the ICP-AES analysis on the Data Summary Table. The validator discusses the poor laboratory precision for lead analyzed by ICP-AES in the Data Validation Memorandum and notes that laboratory precision for the other analytes as well as for lead analyzed by ICP-MS was acceptable.

E. EXAMPLES (continued)

Example #2: (Both soil sample and laboratory duplicate sample concentrations < 5xSQL; Absolute difference > 2xSQL; Poor laboratory precision for manganese; Acceptable laboratory precision for thallium)

Soil sample MAAZ33 (1.02 g wet wt.) and laboratory duplicate MAAZ33D (1.01 g wet wt.), digested and analyzed under CLP SOW ILM05.4 by ICP-AES, have a percent solids of 92%. Sample MAAZ33 has a manganese concentration of 3.0 mg/kg (dry wt.) and a thallium concentration of 2.1J mg/kg (dry wt.). The duplicate sample MAAZ33D has a manganese concentration of 7.9 mg/kg (dry wt.) and a non-detect for thallium. The validator notes that all results are less than 5 times the sample quantitation limits (QLs).

Analyte	MAAZ33		MAAZ33D		Abs. Diff. (mg/kg)	Abs. Diff. Criteria (2xSQL) (mg/kg)
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Manganese	3.0	1.6/ 8.0	7.9	1.6/ 8.0	4.9	3.2
Thallium	2.1J	2.7/ 13.5	2.7U	2.7/ 13.5	NA	NA

The validator uses the laboratory duplicate QC criteria specified in Table INORG-IX-3 for non-aqueous matrices. Since the sample values are less than 5xSQL, the validator uses the 2xSQL criteria to evaluate the absolute difference between the sample duplicate concentrations rather than the RPD criteria. The duplicate precision for thallium by ICP-AES is not evaluated since both results are below the SQL or non-detects. For manganese the absolute difference between the duplicate samples is greater than the 2xSQL criteria. The validator evaluates the field duplicate pair and determines that the RPDs and absolute differences are within the QC criteria specified in the QAPP, indicating acceptable overall precision for this sampling event. The validator then concludes that the lack of laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) positive detects and estimates (UJ) non-detects for manganese in all soil samples on the Data Summary Table. The validator discusses the poor laboratory precision for manganese in the Data Validation Memorandum and notes that laboratory precision for the other analytes was acceptable.

E. EXAMPLES (continued)

Example #3: (One soil sample value ≥ 5xSQL and laboratory duplicate sample value < 5xSQL; Absolute difference > 2xSQL; Poor laboratory precision)

Soil sample MACC11 (1.02 g wet wt.) and laboratory duplicate sample MACC11D (1.04 g wet wt.), digested and analyzed under CLP SOW ILM05.4, have a percent solids of 88%. Sample MACC11 has a potassium concentration of 3900 mg/kg (dry wt.). The duplicate sample MACC11D has a potassium concentration of 2100 mg/kg (dry wt.). The validator notes that one result is less than 5xSQL and one result is greater than 5xSQL.

Analyte	MACC11		MACC11D		Abs. Diff. (mg/kg)	Abs. Diff. Criteria (2xSQL) (mg/kg)
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Potassium	3900	557/ 2790	2100	546/ 2730	1800	1114

The validator uses the laboratory duplicate QC criteria specified in Table INORG-IX-3 for non-aqueous matrices. Since one sample value is less than 5xSQL and the other sample value is greater than 5xSQL, the QC criteria of 2xSQL rather than the RPD must be used to evaluate the differences between the sample concentrations. The absolute difference between the duplicate samples is 1800 mg/kg which is greater than 2xSQL. The validator evaluates the field duplicate pair and determines that the RPDs and absolute differences are within the QC criteria specified in the QAPP, indicating overall precision for this sampling event was acceptable. The validator then concludes that the poor laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) positive detects and estimates (UJ) non-detects for potassium in all soil samples on the Data Summary Table. The validator discusses the poor laboratory precision for potassium in the Data Validation Memorandum and notes that laboratory precision for the other analytes was acceptable.

X. FIELD DUPLICATES

A. OBJECTIVE

Field duplicates measure the cumulative effects of both field and laboratory precision and hence provide an indication of overall precision. Therefore, field duplicates may have greater variability than laboratory duplicates which measure only laboratory precision. It is also expected that non-aqueous matrices will have a greater variance than aqueous matrices due to the heterogeneity of most non-aqueous samples (such as soil/sediment samples).

B. CRITERIA

1. The frequency of field duplicate analysis must support the site-specific Data Quality Objectives (DQOs) and must be documented in the EPA-approved QAPP or SAP.

2. a. Aqueous Field Duplicates

   i. For all analytes detected at concentrations greater than or equal to five times the sample quantitation limit in both field duplicate samples of aqueous matrices, the RPD must be less than or equal to 30 percent.

   ii. For all analytes detected at concentrations less than five times the sample quantitation limit in either field duplicate sample of aqueous matrices, the absolute difference must be less than or equal to twice the sample quantitation limit.

   b. Non-Aqueous Field Duplicates

   i. For all analytes detected at concentrations greater than or equal to five times the sample quantitation limit in both field duplicate samples of non-aqueous matrices, the RPD must be less than or equal to 50 percent.

   ii. For all analytes detected at concentrations less than five times the sample quantitation limit in either field duplicate sample of non-aqueous matrices, the absolute difference must be less than or equal to four times the sample quantitation limit.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from field duplicate anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Identify the samples which are field duplicates from the Chain-of-Custody Record and/or the Traffic Report.	1. a. If field duplicates are not listed on the Chain-of-Custody Record or the Traffic Report, then the validator should contact the sampler to ascertain if field duplicates were collected. If the forms were completed incorrectly or if field duplicates were not collected, then the validator should document this on the Data Validation Worksheet and in the Data Validation Memorandum.
b. Verify that the appropriate number of field duplicates per matrix sampled were collected and analyzed to support project DQOs.	b. If field duplicates were not collected at the required frequency to support project DQOs, then the validator should note the absence of field precision data in the Data Validation Memorandum and discuss how the lack of field precision data might potentially increase uncertainty surrounding site decisions.

C. EVALUATION	D. ACTION
	Note: Action applies to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method.
2. a. Aqueous Field Duplicates	2. a. Aqueous Field Duplicates
i. Calculate the RPD for all analytes detected at concentrations greater than or equal to 5x the sample quantitation limit in both aqueous field duplicates.	i. If any analyte is detected at concentrations greater than or equal to 5x the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should: - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method.
ii. Calculate the absolute difference for all analytes detected at concentrations less than 5x the sample quantitation limit in either one or both of the aqueous field duplicate samples (including the case where one duplicate sample result is a non-detect and the other result is a positive detect).	ii. If any analyte is detected at concentrations less than 5x the sample quantitation limit in either one or both of the aqueous field duplicate samples and the absolute difference is greater than 2x the sample quantitation limit, then the validator should: - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. - If any analyte is detected at concentrations less than the sample quantitation limit in both of the field duplicate samples, or if any analyte is a non-detect in both of the field duplicate samples, then no action is taken.

C. EVALUATION	D. ACTION
2. b. Non-Aqueous Field Duplicates i. Calculate the RPD for all analytes detected at concentrations greater than or equal to 5x the sample quantitation limit in both non-aqueous field duplicates. ii. Calculate the absolute difference for all analytes detected at concentrations less than 5x the sample quantitation limit in either one or both of the non-aqueous field duplicate samples (including the case where one duplicate sample result is a non-detect and the other result is a positive detect). Note: When applying the criteria of 4x the sample quantitation limit, the sample quantitation limit is calculated using the sample weight, volume, and percent solids for the sample versus the duplicate sample.	2. b. Non-Aqueous Field Duplicates i. If any analyte is detected at concentrations greater than or equal to 5x the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should: - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. If any analyte is detected at concentrations less than 5x the sample quantitation limit in either one or both of the non-aqueous field duplicate samples and the absolute difference is greater than 4x the sample quantitation limit, then the validator should: - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. - If any analyte is detected at concentrations less than the sample quantitation limit in both of the field duplicate samples, or if any analyte is a non-detect in both of the field duplicate samples, then no action is taken.
*3. Check and recalculate the analytical concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or soil sample) for each analytical method in each field duplicate sample.	3. If calculation and/or transcription errors are detected, then the validator should follow the procedures outlined in Section INORG-XIV (Analyte Quantitation and Reported Quantitation Limits), D.1-D.3.

C. EVALUATION	D. ACTION
4. a. Evaluate the appropriateness of qualifying only the field duplicate sample results or only a subset of samples of the same matrix for the affected analyte.	4. a. Generally, action based on field duplicate results is applied to the affected analyte across all samples of the same matrix prepared and analyzed by the same method. If there is information to indicate that the matrix heterogeneity and/or potential sampling error are limited to the field duplicate samples or to a specific subset of samples of the same matrix, then professional judgment may be used to apply the action only to the field duplicate samples or to a specific subset of samples of the same matrix. The validator should discuss in the Data Validation Memorandum the justification for not qualifying all samples of the same matrix and limiting the qualification to specific samples. The potential impact on the representativeness and usability of the data in meeting project DQQs should be discussed.
b. Evaluate field duplicate precision data to assess overall precision and to verify the field sampler's ability to collect representative duplicate samples. Laboratory duplicate sample data should be evaluated to verify the laboratory's ability to generate precise data. Matrix spike data can also be evaluated to identify overall matrix issues.	b. If field duplicate data indicate poor field precision and general sample heterogeneity and/or possible sampling error, then professional judgment may be used to qualify data for all analytes in all samples of the same matrix. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting project DQOs should be discussed. Refer to Section IX (Laboratory Duplicate Samples) for additional guidance.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.3

Table INORG-X-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON FIELD DUPLICATES - AQUEOUS MATRICES

Sample Results	Aqueous Field Duplicate Sample Results			
	Both Duplicates ≥ 5xQL		One or Both Duplicates < 5xQL*	
	RPD ≤ 30%	RPD > 30%	Abs. Diff. ≤ 2xQL	Abs. Diff. > 2xQL
Detects	A	J	A	J
Non-detects	A	UJ	A	J

QL = Sample Quantitation Limit

* No action is taken when both field duplicate results are positive detects < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the field duplicate results are outside QC acceptance criteria.

Table INORG-X-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON FIELD DUPLICATES - NON-AQUEOUS MATRICES

Sample Results	Non-Aqueous Field Duplicate Sample Results			
	Both Duplicates ≥ 5xQL		One or Both Duplicates < 5xQL*	
	RPD ≤ 50%	RPD > 50%	Abs. Diff. ≤ 4xQL	Abs. Diff. > 4xQL
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

QL = Sample Quantitation Limit

* No action is taken when both field duplicate results are positive detects < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the field duplicate results are outside QC acceptance criteria.

E. EXAMPLES

Example #1: (Both soil field duplicate sample concentrations ≥ 5xSQL; RPD > 50%; Acceptable laboratory precision)

Soil samples MADF61 and MADF62 are field duplicates analyzed under CLP SOW ILM05.4. Sample MADF61 has a detected manganese concentration of 120 mg/kg. Sample MADF62 has a detected manganese concentration of 275 mg/kg. The validator notes that both results are greater than 5x the sample quantitation limit (SQL). (The samples contain 82% and 91% solids, respectively, and 1.0 g wet weight was used for each.) Therefore, the validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 78%.

Analyte	Sample MADF61		Duplicate MADF62		RPD	RPD Criteria
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Manganese	120	1.8/ 9.0	275	1.6/ 8.0	78%	50%

The validator reviews the laboratory duplicate sample data and determines that laboratory precision was acceptable. The validator estimates (J) positive detects and estimates (UJ) non-detects for manganese in all soil samples on the Data Summary Table based on poor field precision. The validator notes the qualification and justification in the Data Validation Memorandum and also notes that poor field precision may be due to a heterogeneous matrix or may be a result of sampling error.

E. EXAMPLES (continued)

Example #2: (Both aqueous field duplicate sample concentrations < 5xSQL; Absolute difference > 2xSQL; Acceptable laboratory precision)

Aqueous samples MAEL21 and MAEL22 are field duplicates analyzed under CLP SOW ILM05.4. Sample MAEL21 has a detected cobalt concentration of 59 ug/L, and sample MAEL22 has a detected cobalt concentration of 187 ug/L. The validator notes that both field duplicate results are below 5x the sample quantitation limit. Since both field duplicate sample values are less than 5xSQL, the validation criteria of 2xSQL is used to evaluate the difference between the sample concentrations. The validator determines that the absolute difference between the field duplicates is 128 ug/L, which is greater than 2xSQL.

Analyte	Sample MAEL21		Duplicate MAEL22		Abs. Diff. (ug/L)	Abs. Diff. Criteria (2xSQL) (ug/L)
	Sample Conc. (ug/L)	SQL/ 5xSQL (ug/L)	Sample Conc. (ug/L)	SQL/ 5xSQL (ug/L)		
Cobalt	59	50/ 250	187	50/ 250	128	100

The validator reviews the laboratory duplicate sample data and determines that laboratory precision was acceptable. The validator estimates (J) positive detects and estimates (UJ) non-detects for cobalt in all aqueous samples on the Data Summary Table based on poor field precision. The validator notes the qualification and justification in the Data Validation Memorandum and also notes that poor field precision may be due to sampling error.

E. EXAMPLES (continued)

Example #3: (One soil field duplicate sample concentration < 5xSQL, other soil field duplicate sample concentration ≥ 5xSQL; Absolute difference > 4xSQL; Poor field and laboratory precision.)

Soil samples MADL22 and MADL23 are field duplicates analyzed under CLP SOW ILM05.4. Sample MADL22 has a detected zinc concentration of 18 mg/kg; sample MADL23 has a detected zinc concentration of 54 mg/kg. The validator notes that one field duplicate result is less than 5xSQL and the other result is greater than 5xSQL. Therefore, the validation criteria of 4xSQL rather than the RPD is used to evaluate the difference between the sample concentrations. The validator calculates the 4xSQL criteria using the sample quantitation limit of the sample. (The samples contain 89% and 95% solids, respectively, and 1.0 g wet weight was used for each.) The absolute difference between the field duplicates is 36 mg/kg which is greater than 4xSQL.

Analyte	Sample MADL22		Duplicate MADL23		Abs. Diff. (mg/kg)	Abs. Diff. Criteria (4xSQL) (mg/kg)
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Zinc	18	6.7/ 34	54	6.3/ 32	36	27

The validator reviews the laboratory duplicate sample data and determines that laboratory precision was unacceptable for zinc. The validator is unable to determine the source of the imprecision since both laboratory and field precision were poor. The validator estimates (J) positive detects and estimates (UJ) non-detects for zinc in all soil samples on the Data Summary Table. The validator notes the qualifications and justifications in the Data Validation Memorandum and also notes that the source of the imprecision cannot be determined.

XI. ICP SERIAL DILUTIONS

A. OBJECTIVE

Serial dilution sample results are generated to assess physical and chemical interferences caused by the sample matrix in ICP analysis. These interferences can cause suppression or enhancement of the analyte signal. If the analyte concentration in the sample is sufficiently high, an analysis of the diluted sample should agree within some acceptable QC criteria that have been established for that matrix and method. If not, a physical or chemical interference effect should be suspected.

B. CRITERIA

The Region-I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. In accordance with the SAP, QAPP and/or method, a field sample of each matrix is diluted, typically five-fold, to generate a serial dilution sample.

2. Field samples (not equipment or bottle blanks and not PE samples) must be diluted and analyzed to assess matrix interference effects.

3. The percent difference (% D) between the serial dilution sample result (after correction for the dilution) and the original determination (undiluted sample result) must be within the QC acceptance criteria specified in the method, SAP or QAPP. The analyte concentration must be sufficiently high, in accordance with method requirements, in order to apply the % D criteria.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from ICP serial dilution anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that a serial dilution sample was prepared and analyzed at the proper dilution and frequency, and that serial dilution sample results are provided for each sample matrix and for each ICP-AES and ICP-MS method used to report sample results. If the sample used for the serial dilution was diluted in order to bring the analyte's result within the initial calibration or linear range of the instrument, then the serial dilution must be performed on the diluted sample (from which the sample result was reported) for evaluating matrix interferences for that particular analyte.	1. If the laboratory did not prepare and analyze a serial dilution sample at the required dilution and frequency specified in the method for each sample matrix and ICP method, then the validator must use professional judgment to determine whether or not the associated sample data should be qualified.
2. Verify that a field sample was chosen for the serial dilution sample.	2. If an equipment blank, a bottle blank, or a PE sample was used for the serial dilution sample, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing sample matrix effects and, ultimately, data usability

C. EVALUATION	D. ACTION
	Note: Action applies to the affected analytes in <u>all</u> samples of the same matrix prepared and analyzed by the same method.
3. Verify that serial dilution percent differences are within the QC acceptance criteria specified in the method. If the analyte concentration in either the original (undiluted) sample or the diluted sample is sufficiently high, as specified in the method, then evaluate whether the serial dilution sample result (corrected for dilution) is greater than or less than the undiluted sample result to assess the potential for bias. - A serial dilution sample result that is greater than the undiluted sample result may indicate possible suppression of the analyte signal due to matrix interferences and potential low bias in sample results. - A serial dilution sample result that is less than the undiluted sample result may indicate possible enhancement of the analyte signal due to matrix interferences and potential high bias.	3. a. If any serial dilution percent difference is greater than the method QC acceptance criteria and the serial dilution sample result is greater than the undiluted sample result, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. b. If any serial dilution percent difference is greater than the method QC acceptance criteria and the serial dilution sample result is less than the undiluted sample result, then the validator should: i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Accept non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to estimate non-detects if the direction of the bias cannot be determined. c. If the majority of analyte percent differences for a method are outside the QC acceptance criteria, then the validator may use professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects in all samples of the same matrix analyzed by the same method.

C. EVALUATION	D. ACTION
4. Evaluate the appropriateness of qualifying only the results of the sample used for the serial dilution analysis or a subset of the samples of the same matrix for the affected analyte.	4. Generally, action based on the serial dilution sample results is applied to the affected analyte across all samples of the same matrix prepared and analyzed by the same method in a sample delivery group. However, professional judgment may be used to determine sample matrix similarity and to apply the action only to the field sample used for the serial dilution analysis, or to a select group of samples in the SDG, if there is information to support such an action. All justifications for not qualifying all samples of the same matrix and limiting the qualification to specific samples should be documented in the Data Validation Memorandum and the potential impact on data usability in meeting the project DQOs should be discussed.
*5. Check and recalculate the analytical concentrations and percent differences for at least one analyte per analytical method. Verify that the recalculated values and % differences agree within ±10% of the reported values. Confirm that the laboratory used the appropriate method criteria.	5. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsection is applicable only to a Tier III data validation:**

C.5

Table INORG-XI-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON SERIAL DILUTION PERCENT DIFFERENCES

Sample Results	% D ≤ QC Limit	% D > QC Limit	
		Serial Dilution Sample Result > Undiluted Sample Result	Serial Dilution Sample Result < Undiluted Sample Result
Detects	A	J	J
Non-detects	A	UJ	A*

Note: Qualification is applied to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. However, the validator may use professional judgment to qualify all positive detects and non-detects if the majority of the serial dilution analyte percent differences are outside the method QC acceptance criteria.

* Professional judgment may be used to estimate (UJ) non-detects if the direction of the bias cannot be determined.

E. EXAMPLES

Example #1: (Serial dilution % D > QC limit, Serial dilution sample result > undiluted sample result)

Aqueous sample MAEF47, analyzed by CLP SOW ILM05.4, has a high % D of 21% for lead in the five-fold serial dilution sample analysis by ICP-AES. The lead result of 175.1 ug/L in the original (undiluted) sample was greater than 50x the MDL of 3 ug/L, sufficiently high to apply the % D criteria. The lead result of 211.8 ug/L from the diluted sample analysis was greater than the original (undiluted) sample concentration of 175.1 ug/L.

Sample No.	Lead MDL (ug/L)	50x MDL (ug/L)	Undiluted Sample Result (ug/L)	Diluted Sample Result (corrected for dilution) (ug/L)	% D	% D QC Acceptance Criteria
MAEF47	3	150	175.1	211.8	21	10

As a result, the validator estimates (J) positive detects and estimates (UJ) non-detects for lead in all aqueous samples analyzed by ICP-AES on the Data Summary Table and notes in the Data Validation Memorandum the possibility of biased low results.

E. EXAMPLES (continued)

Example #2: (Serial Dilution % D > QC limit; Serial dilution sample result < undiluted sample result)

Soil sample MA7D14, analyzed by CLP SOW ILM05.4, has a high % D of 22% for chromium in the five-fold serial dilution sample analysis by ICP-AES. The serial dilution form, which reports the soil sample results as ug/L in the final digestate, shows a chromium result of 1331.26 ug/L in the original (undiluted) sample. This is greater than 50x the chromium MDL of 5 ug/L and sufficiently high to apply the % D criteria. The chromium result of 1037.42 ug/L from the five-fold diluted sample, reported on the serial dilution form, was less than the original (undiluted) sample concentration of 1331.26 ug/L.

Sample No.	Chromium MDL (ug/L)	50x MDL (ug/L)	Undiluted Sample Result (ug/L)	Diluted Sample Result (corrected for dilution) (ug/L)	% D	% D QC Acceptance Criteria
MA7D14	5	250	1331.26	1037.42	22	10

As a result, the validator estimates (J) positive detects and accepts non-detects for chromium in all soil samples analyzed by ICP-AES on the Data Summary Table and notes in the Data Validation Memorandum the possibility of biased high results.

XII. SENSITIVITY CHECK

A. OBJECTIVE

Many EPA methods including the CLP SOWs incorporate the analysis of sensitivity checks by requiring that a Method Detection Limit (MDL) study be performed prior to sample analysis and/or that a Laboratory Fortified Blank (LFB) be analyzed at the time of sample analysis. The MDL study generates statistically-based detection limits and can be used to assess method sensitivity, laboratory precision, and method bias for specific analytes within an analytical method on a specific instrument. An LFB, a type of Laboratory Control Sample, is a reagent blank which is spiked with the target analytes at their quantitation limits and is processed along with the samples. LFB data can be used to assess laboratory sensitivity and bias for specific analytes at the quantitation limit within an analytical method on a specific instrument at the time of sample preparation and analysis.

Region I routinely uses MDL studies as a pre-qualification check to verify the laboratory's ability to meet the technical specification/method requirements prior to contract award and field sample receipt. LFB analyses document the method sensitivity and bias associated with the day-to-day preparation and analysis of field samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. Method Detection Limit (MDL) Study

a. The method detection limit (MDL) for each analyte of interest must be established in accordance with the specified method and the Code of Federal Regulations (40 CFR Part 136, App. B). A minimum of seven replicates must be analyzed for each matrix of interest.

b. Samples must be analyzed on the same instrument, under the same conditions, as was used for the MDL study.

c. The MDL study must be performed within one year prior to the start of the preparation and/or analysis of the samples.

d. The MDL for each target analyte must be less than or equal to that analyte's method-required quantitation limit.

2. **Laboratory Fortified Blank (LFB)**

a. Verification of laboratory accuracy at the quantitation limit requires the routine analysis of an LFB spiked with target analytes at the quantitation limit.

b. An LFB containing all of the target analytes at their respective quantitation limits must be prepared and analyzed along with the samples at the method-required frequency and analyzed at least once immediately prior to sample analysis but after instrument calibration.

c. Method QC acceptance criteria must be met for target analytes and internal standards (if applicable).

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
Qualification of data should be based on a combined evaluation of both the MDL study and LFB results. To determine appropriate sample qualification, the MDL study should be evaluated first and then the LFB results.	All potential impacts on the sample data resulting from MDL and/or LFB study anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Method Detection Limit (MDL) Study	**1. Method Detection Limit (MDL) Study**
a. Verify that the samples were analyzed on the same instruments as those used for the MDL study.	a. If the samples were not analyzed on the same instruments as the MDL study, then the validator should contact the laboratory to obtain a correct MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality, taking into consideration the results of other low level standards analyzed.

C. EVALUATION	D. ACTION
1. b. Compare the date of the MDL study to the dates of all associated sample analyses to verify that the MDL study was performed within one year prior to the start of the first sample prepared and/or analyzed in the sample delivery group.	1. b. If the MDL study was not submitted or was not performed within one year of the start of preparation and/or analysis of the first sample in the SDG, then the validator should contact the laboratory to obtain a current MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should evaluate the instrument's response to the lowest standard of the initial calibration or other low level standard (e.g., Quantitation Limit Check Standard) and use professional judgment to assess the impact of analytical sensitivity on data quality.
c. Verify that all MDLs are less than or equal to the method-required quantitation limits.	c. If the MDL study reveals that a target analyte has a detection limit greater than the method-required quantitation limit, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should: i. Elevate the quantitation limit for that target analyte in all samples associated with that MDL study to the lowest concentration calibration standard, other low level standard (e.g., Quantitation Limit Check Standard) analyzed, or to the laboratory-reported MDL, whichever is higher. ii. Estimate (J) positive detects which were below the elevated quantitation limit for that target analyte in all samples associated with that MDL study. iii. The validator should evaluate the elevated quantitation limits in relation to the method-required quantitation limits and project DQOs. The validator should discuss any impact of the elevated quantitation limits on project objectives and data usability in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*1. d. Verify that the MDL study was generated in accordance with the method and 40 CFR Part 136, App. B. Verify that a minimum of seven replicates for each matrix of interest was prepared and analyzed.	1. d. If the required MDL study was not performed at all or was not performed according to the method or the CFR criteria, then the validator should evaluate the LFB data, if available, to determine the action to be taken. See Table INORG-XII-1. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality. The results of other low level standards (e.g., Quantitation Limit Check Standard) should be evaluated and appropriate action taken. (See Section III, Calibrations.)
* e. For applicable methods (e.g., ICP-MS), verify that internal standard responses meet QC acceptance criteria.	e. If the MDL study reveals that a target analyte has a detection limit greater than the method-required quantitation limit, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should:
* f. Check and recalculate the MDL value for at least one analyte per MDL study per method. Verify that the recalculated values agree within ±10% of the reported results. (Note: The MDL study raw data may not be provided with the data packages and may not be readily available to allow for verification or recalculation, as in the case of the CLP SOWs.)	f. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
2. Laboratory Fortified Blank (LFB)	**2. Laboratory Fortified Blank (LFB)**
a. Verify that an LFB was prepared (digested/distilled) and analyzed at the proper frequency and that it was spiked with the correct analytes at their quantitation limits.	a. If an LFB analysis was not performed or if the LFB was not analyzed for the correct analytes at the proper frequency and concentration, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality. Professional judgment should be used to qualify sample quantitation limits.
b. Verify that the reported recoveries for all LFB spike analytes are within the method QC acceptance criteria. If the LFB criteria are not met, then laboratory performance related to method bias and method/instrument sensitivity is questionable.	b. If any of the LFB analyte recoveries are outside the method QC acceptance criteria, then the LFB results should be used to qualify sample data for the affected analytes that are included in the LFB solution. The validator should use professional judgment to qualify sample data for non-LFB analytes, taking into account information that may exist in the Sample Delivery Group for other low level standards. i. If an LFB analyte recovery is greater than the upper method QC limit, then the validator should: - Estimate (J) positive detects less than 2x the LFB true value for the affected analyte in all samples associated with that LFB to indicate potential high bias. The validator may use professional judgment to reject data based on the project DQOs. - Accept the non-detects for the affected analyte in all samples associated with that LFB.

C. EVALUATION	D. ACTION
2. b. Continued from above.	2. b. ii. If an LFB analyte recovery is less than the lower method QC limit but greater than or equal to 40%, then the validator should: - Estimate (J) positive detects less than 2x the LFB true value for the affected analyte in all samples associated with that LFB to indicate potential low bias. - Estimate (UJ) the non-detects at the quantitation limits for the affected analyte in all samples associated with that LFB to indicate potential low bias. iii. If an LFB analyte recovery is less than 40%, then the validator should: - Estimate (J) positive detects less than 2x the LFB true value for the affected analyte in all samples associated with that LFB. Professional judgment should be used to reject data taking into account project DQOs. - Reject (R) the non-detects at the quantitation limits for the affected analyte in all samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives. iv. For multi-analyte analysis, if more than half of the LFB analyte recoveries are outside the method QC acceptance criteria, then the validator may use professional judgment to apply validation actions to all positive detects and all non-detects at the quantitation limits associated with that LFB. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
2. b. Continued from above.	2. b. v. The validator should evaluate the LFB recoveries to determine whether project DQOs were achieved. Professional judgment should be used to determine whether the associated sample data should be qualified or rejected, taking into consideration the extent of deviation, the potential bias, and project DQOs. In some cases, it may be necessary to reject data. The Data Validation Memorandum should include a discussion of the rationale for data qualification and/or rejection. vi. The validator may use professional judgment to further apply data validation qualifiers to positive detects greater than 2x the LFB true value for the affected analyte, taking into account the extent of deviation from the LFB method QC acceptance criteria and the project DQOs. The validator should evaluate all relevant QC data which may provide information regarding the bias at the time of sample preparation and analysis and the laboratory's ability to accurately quantitate target analytes at concentration ranges greater than 2x the LFB true value. Qualification of results greater than 2x the LFB true value but less than the value of the next highest concentration QC sample, such as the laboratory control sample or PE sample, may be warranted. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
2. b. Continued from above.	2. b. vii. If data quality objectives allow for greater variability of data at levels near the quantitation limit, then expanded LFB validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP.
* c. Check and recalculate the % recovery for at least one target analyte per method and LFB. Verify that the recalculated value agrees within ±10% of the reported result.	c. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.d, C.1.e, C.1.f, C.2.c

Table INORG-XII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LFB RECOVERIES

Sample Results	% Recovery			
	% R < 40%	40% ≤ % R < LL	LL ≤ % R ≤ UL	% R > UL
Detects*	J (< 2x TV)**	J (< 2x TV)	A	J (< 2x TV)**
Non-detects	R	UJ	A	A

LFB = Laboratory fortified blank spiked with target analytes at their quantitation limits.
LL = Lower Limit of method QC acceptance criteria.
UL = Upper Limit of method QC acceptance criteria.
* Action is applied to positive detects less than 2x the LFB true value.
** Professional judgment may be used to reject positive results less than 2x the true value taking into account project DQOs. Professional judgment may be used to estimate positive detects greater than or equal to 2x the LFB true value but less than the value of the next highest concentration QC sample.

E. EXAMPLES

Example #1: (Low LFB recoveries for several analytes)

Water samples were analyzed by ICP-AES. Only the MDL values were reported with the data; the MDL study raw data were not available. The validator compares all MDL values to the method-required quantitation limits and determines that all MDLs were less than the required QLs. The LFB analytes chromium, nickel, and zinc recovered at 65%, 60%, and 53%, respectively, below the method QC acceptance criteria. The validator estimates (J) positive detects less than 2x the LFB true value for chromium, nickel, and zinc in all the samples associated with that LFB to indicate potential low bias and estimates (UJ) the non-detects at the quantitation limit for the chromium, nickel, and zinc non-detects in all samples associated with that LFB to indicate a decrease in sensitivity and the possibility of false negatives. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #2: (High LFB recoveries)

Water samples were analyzed by ICP-MS. Only the MDL values were reported with the data; the MDL study raw data were not available. The validator compares all MDL values to the method-required quantitation limits and determines that all MDLs were less than the required QLs. The LFB for selenium and thallium recovered above the method QC acceptance criteria. The validator estimates (J) positive detects less than 2x the LFB true value for selenium and thallium and accepts all non-detects. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #3: (Low LFB recovery < 40%)

Soil samples were analyzed by ICP-AES. Only the MDL values were reported with the data; the MDL study raw data were not available. The validator compares all MDL values to the method-required quantitation limits and determines that all MDLs were less than the required QLs. The cadmium LFB recovered below the method QC acceptance criteria and below 40%. Therefore, the validator rejects (R) all non-detects for cadmium and estimates (J) all positive cadmium detects less than 2x the true value of the LFB. Since the laboratory control sample and PE sample results at higher spike concentrations showed acceptable recoveries, and the QL Check Standard was within the method QC criteria, the validator uses professional judgment to take no further action on sample results greater than 2x the LFB true value. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

XIII. PERFORMANCE EVALUATION SAMPLES/ACCURACY CHECK

A. OBJECTIVE

Data for Performance Evaluation Samples (PESs) are generated to provide information on the overall accuracy and bias of the analytical method and on laboratory performance. PESs are evaluated for false negatives, false positives, and inaccurate target analyte quantitation. In general, the most serious problem a PES can expose is the failure of the laboratory to properly detect and identify a PES analyte. This failure is known as a false negative. False negatives significantly increase the "uncertainty" surrounding any site decisions made concerning the "cleanliness" or contamination present at a site. A second problem revealed by PES analysis is the laboratory's erroneous detection of target and non-target analytes that were not spiked into the PES, otherwise known as false positives. False positives should always be evaluated in conjunction with blank data to ascertain the probable source(s) of contamination.

Finally, the PES provides information on the magnitude and direction of quantitative bias for the entire laboratory method, including sample preparation (digestion/distillation) and analysis (calibration). Sample data that are biased high or low can potentially impact site decisions, especially when sample data have target analyte concentrations at or near project action levels.

Ideally, a PES is comprised of the same matrix as the field samples being evaluated. However, for some matrices PESs may not be available. In these situations, a PES of another matrix may be analyzed with the field samples to assess laboratory performance on the "analysis" portion, even though laboratory performance on the "sample preparation" portion cannot be assessed. The validator should use professional judgment when evaluating samples of one matrix using PES data from another matrix.

B. CRITERIA

1. Zero Blind Performance Evaluation Samples

A Zero Blind PES is a quality control sample that is of a composition and concentration known to the laboratory.

A Laboratory Control Sample (LCS) is a Zero Blind PES which is often used by the laboratory as an internal quality control check of analytical accuracy and method bias, including the efficiency of the digestion/distillation procedure.

An LCS containing several or all of the target analytes spiked at concentrations at or near their quantitation limits is called a Laboratory Fortified Blank (LFB). Refer to Section XII (Sensitivity Check) for additional LFB guidance.

a. An LCS is required by some EPA methods and CLP SOWs. The frequency, concentration, acceptance criteria and corrective actions for LCS analysis should be stated in the method, Sampling and Analysis Plan (SAP) or the Quality Assurance Project Plan (QAPP) and should support the DQOs of the project. The LCS should be prepared in the proper matrix for each parameter at the concentration level and frequency required in the EPA-approved project SAP, QAPP, and/or method. The LCS must contain the target analytes and must be prepared and analyzed concurrently with field samples contained in the sample delivery group.

B. CRITERIA (continued)

1. b. The percent recoveries for LCS analytes must be within the method QC acceptance criteria.

   c. Internal standards, if applicable, for the LCS must meet validation criteria as per Section VII (ICP-MS Internal Standards) of this document.

2. Single Blind Performance Evaluation Samples

A Single Blind PES is a quality control sample that is of a composition and concentration not known to the laboratory, but the sample is identified to the laboratory as a PES.

A Single Blind PES may be submitted with a sample delivery group to assess method bias, laboratory performance, and to evaluate data quality. A Single Blind PES may also be submitted for analysis prior to sample shipment to pre-qualify a laboratory for a specific matrix and/or parameter.

a. The latest revision of the EPA Region I Performance Evaluation Program Guidance requires that a Single Blind or Double Blind PES be sent with each sample delivery group (20 samples or less) that is sent to a laboratory. A PES is required for each matrix, parameter, and concentration level unless an EPA or non-EPA PES does not currently exist for that particular matrix, parameter, or concentration level.

   The PE Program applies to the Superfund program including EPA Fund-lead and PRP/Federal Facility Oversight Projects. In addition, the PE Program applies to Fund-lead projects performed by States under Cooperative Agreements and other Federal Agencies under Interagency Agreements. The PE Program also applies to Non-Fund-lead Superfund projects undertaken by potentially responsible parties. The PE Program also applies to Non-Superfund Programs.

   EPA-provided PE samples are available for certain categories of Superfund work as specified in the latest revision of the EPA Region I Performance Evaluation Program Guidance. The EPA Performance Evaluation Chemist provides the current list of EPA-provided PE samples upon request. For those categories of Superfund work that do not have access to EPA-provided PE samples and for all Non-Superfund program work, scientifically defensible PE samples should be obtained from commercial vendors.

b. Acceptance criteria for EPA PESs are statistically-derived by the Analytical Services Branch under the QATS contract. Tabulated report forms for EPA PESs must be submitted to the Region I OEME-QA Unit for scoring at the time of data validation, in accordance with the latest revision of the EPA Region I Performance Evaluation Program Guidance.

c. True values and QC acceptance criteria for all non-EPA PESs should be provided by the manufacturer and these acceptance criteria must be fully documented and must be scientifically defensible.

d. Internal standards, if applicable, for the PES must meet validation criteria as per Section VII (ICP-MS Internal Standards) of this document.

B. CRITERIA (continued)

3. Double Blind Performance Evaluation Samples

A Double Blind PES is a quality control sample that is of a composition and concentration not known to the laboratory and the sample is **not** identifiable as a PES nor is it identified to the laboratory as a PES.

A Double Blind PES may be submitted with a sample delivery group, in lieu of a Single Blind PES, to assess method bias, laboratory performance, and to evaluate data quality.

a. The use of Double Blind PESs is dictated by the project DQOs and should be documented in the EPA-approved SAP and/or QAPP.

b. True values and acceptance criteria for Double Blind PESs must be fully documented and must be scientifically defensible.

c. Internal standards, if applicable, for the PES must meet validation criteria as per Section VII (ICP-MS Internal Standards) of this document.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from performance evaluation sample anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Zero Blind PES - LCS a. Verify that an appropriate LCS sample (correct parameter, concentration level, target analytes and matrix) was prepared and analyzed at the required frequency for each sample delivery group and for each batch of samples prepared (digested/distilled) in accordance with the EPA approved project SAP, QAPP and/or method. Note: In the CLP SOW ILM05.4, the digested ICV serves as the aqueous LCS for mercury, and the distilled ICV serves as the aqueous LCS for cyanide. See Section III, Calibrations.	**1. Zero Blind PES - LCS** a. If an appropriate LCS was not analyzed at the required frequency for the correct parameters, concentration levels, target analytes or matrices, then the validator should use professional judgment to determine if the sample data should be qualified or rejected.

C. EVALUATION	D. ACTION
1. b. Verify that the required LCS results are provided for each sample delivery group and for each batch of samples prepared (digested/distilled).	1. b. If the required LCS results were not submitted for each sample delivery group and for each batch of samples prepared, then the validator should contact the laboratory to obtain raw data and tabulated results.
c. Verify that the reported recoveries for all LCS spike analytes are within the method QC acceptance criteria.	c. Sample data should be qualified based on the analytes that recover outside the method QC acceptance criteria and on the degree that analyte recoveries exceed the criteria.
Note: The CLP SOW ILM05.4 aqueous LCS method acceptance criteria of 80-120% recovery does not apply to antimony and silver, which have no control limits. For data validation purposes, the 80-120% recovery method QC acceptance criteria shall be applied to both antimony and silver. If data quality objectives allow for greater variability of data, then an expanded LCS validation criterion should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP.	i. If any of the LCS analyte recoveries are outside the method QC acceptance criteria, then the LCS results should be used to qualify sample data for the specific analytes that are included in the LCS solution. Professional judgment should be used to qualify sample data for non-LCS analytes, taking into account any analytical problems historically associated with the analyte or that were encountered by the laboratory.
Note: Non-aqueous LCSs obtained from EPA as well as certified materials obtained from other sources typically provide upper and lower concentration control limits specific to that LCS. In this case, the validator should use the criteria established for that specific material to evaluate the non-aqueous LCS results. Confirm that LCS acceptance criteria are fully documented and scientifically defensible.	ii. If an aqueous LCS analyte recovery is greater than the upper limit of the method QC acceptance criteria, but less than or equal to 150%, or if the non-aqueous LCS analyte result is greater than the upper control limit, then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that LCS to indicate potential high bias. - Accept the non-detects for the affected analyte in all samples associated with that LCS.

C. EVALUATION	D. ACTION
1. c. Continued from above.	1. c. iii. If an analyte recovery for an aqueous LCS is greater than 150%, then the validator should: - Reject (R) positive detects for the affected analyte in all samples associated with that LCS. - Accept non-detects for the affected analyte in all samples associated with that LCS. iv. If an aqueous LCS analyte recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 50%, or if a non-aqueous LCS analyte result is less than the lower control limit, then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that LCS to indicate potential low bias. - Estimate (UJ) non-detects for the affected analyte in all samples associated with that LCS to indicate potential low bias. v. If an analyte recovery for an aqueous LCS is less than 50%, then the validator should: - Reject (R) positive detects and non-detects for the affected analyte in all samples associated with that LCS.

C. EVALUATION	D. ACTION
1. c. Continued from above.	1. c. vi. If more than half of the LCS analyte recoveries for one LCS analyzed by a particular method are greater than the upper method QC acceptance criteria, or if more than half of the LCS analyte recoveries for a particular method are less than the lower method QC criteria, then the validator should use professional judgment to apply the above validation actions to all positive detects and non-detects in all samples associated with that LCS. vii. If more than half of the LCS analyte recoveries for a particular method are outside the method QC acceptance limits in one LCS, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, or all of the analytes, for samples associated with that LCS. viii. Based upon the number of analytes misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire method is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
*1. d. Check and recalculate the percent recovery for at least one analyte per method per LCS. Verify that the recalculated value agrees within ±10% of the reported result.	1. d. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Single Blind <u>and</u> Double Blind PESs a. Verify that an appropriate Single Blind or Double Blind PES (correct parameter, concentration level, target analytes and matrix) was analyzed at the required frequency for each sample delivery group in accordance with Region I PE policy and/or the EPA-approved SAP and/or QAPP. b. Verify that Single Blind PES results are provided for each sample delivery group in accordance with Region I PE policy.	**2. Single Blind <u>and</u> Double Blind PESs** a. If a required Single Blind or Double Blind PES was not analyzed at the required frequency for the correct parameters, concentration levels, target analytes or matrices, then the validator should use professional judgment to determine if the sample data should be qualified or rejected. b. If the PES results were not submitted for each sample delivery group, then the validator should contact the laboratory to obtain raw data and/or tabulated results. If a PES was not submitted to the laboratory by the sampler, then the validator should contact the sampler to confirm the omission of a PES and document that fact on the worksheet and in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
2. c. **Region I EPA PESs** If the PES was supplied and scored by Region I OEME-QA, then the Region I PES Score Report must be evaluated to determine how many of the analytes met or exceeded PES acceptance criteria. i. Evaluate each PES "Analyte Missed" to assess the potential for low bias and false negative sample results.	2. c. **Region I EPA PESs** Note: PES results should not be qualified based on QC sample data and should not be reported on the Data Summary Table. Rather, PES results should be discussed in the Data Validation Memorandum or Tier I Validation Cover Letter, and PES Score Reports should be attached as supporting documentation. i. Sample data should be qualified based on the PES "Analytes Missed" identified on the Region I PES Score Report. If a PES analyte is not identified in the PES, then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that PES to indicate potential low bias. - Reject (R) non-detects for the affected analyte in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives. - Based upon the number of analytes that were not identified and a review of the project DQOs, the validator should use professional judgment to determine if all data generated by a particular method are unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
2. c. ii. Evaluate each PES "Contaminant" in conjunction with blank data to assess the potential for high bias and false positive sample results.	2. c. ii. Sample data should not be qualified based on the number of PES "Contaminants" identified on the Region I PES Score Report alone. - If a PES "Contaminant" is detected in the PES and is also found in a blank, then the validator should evaluate and qualify sample data based upon blank contamination in accordance with Section III (Blanks). - If a PES "Contaminant" is detected in the PES but is not present in any blank, then that interference is specific to the PES and does not impact sample data.
iii. Evaluate the PES analytes that were misquantified ("Action High"/ "Action Low") to assess the potential for high and/or low bias in sample data.	iii. Sample data should be qualified based on the number and type of misquantified PES analytes ("Action High"/ "Action Low") identified on the Region I PES Score Report. **Sample data should not be qualified based on "Warning Low"/ "Warning High" scores for PES analytes.** • If any of the PES analytes do not meet PES acceptance criteria, then the PES results should be used to qualify sample data for the specific analytes that are included in the PES. Professional judgment should be used to qualify sample data for non-PES analytes taking into account the analytical problems historically associated with the analyte or that were encountered by the laboratory.

C. EVALUATION	D. ACTION
2. c. iii. Continued from above.	2. c. iii. Continued from above. • If a PES analyte is scored in the "Action High" category, then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that PES to indicate potential high bias. - Accept the quantitation limits of the affected analyte in all samples associated with that PES. • If a PES analyte is scored in the "Action Low" category, then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that PES to indicate potential low bias. - Reject (R) the quantitation limits of the affected analyte in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives. • If more than half of the PES analytes for one PES analyzed by a particular method are scored in the "Action High" category, or if more than half of the PES analytes for a particular method are scored in the "Action Low" category, then the validator should use professional judgment to apply the above validation actions to all positive detects and non-detects in all samples associated with that PES.

C. EVALUATION	D. ACTION
2. c. iii. Continued from above.	2. c. iii. Continued from above. • If more than half of the PES analytes for a particular method are scored in the "Action" levels in one PES, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte or all analytes for samples associated with that PES. • Based upon the number of analytes misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire method is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.
iv. Evaluate ICP-MS internal standards for the EPA PES.	iv. Action on non-compliant internal standard relative intensities should follow the guidance provided in Section VII (ICP-MS Internal Standards). Professional judgment should be used to evaluate the impact that non-compliant EPA PES internal standard relative intensities have on the sample data.

C. EVALUATION	D. ACTION
2. d. **Non-EPA PESs** If the PES was obtained from a source other than Region I OEME-QA, then the validator should use the vendor's criteria to evaluate the PES results. Confirm that PES acceptance criteria are fully documented and scientifically defensible. i. Evaluate the PES analytes missed to assess the potential for low bias and false negative sample results. ii. Evaluate the PES contaminants in conjunction with blank data to assess the potential for high bias and false positive sample results.	2. d. **Non-EPA PESs** If the non-EPA PES acceptance criteria are not fully documented and/or scientifically defensible, then the validator should use professional judgment to qualify or reject the sample data. i. Sample data should be qualified based on the PES analytes missed identified from the vendor's acceptance criteria. If a PES analyte is not identified in the PES, then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that PES to indicate potential low bias. - Reject (R) the non-detects for the affected analyte in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives. - Based upon the number of analytes that were not identified for a particular method and PES, and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire method is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied. ii. Sample data should not be qualified based on the number of PES contaminants identified from the vendor's acceptance criteria <u>alone</u>.

C. EVALUATION	D. ACTION
2. d. ii. Continued from above.	2. d. ii. Continued from above. - If a PES contaminant is detected in the PES and is also found in a blank, then the validator should evaluate and qualify sample data based upon blank contamination in accordance with Section III (Blanks). - If a PES contaminant is detected in the PES but is not present in any blank, then that interference is specific to the PES and does not impact sample data.
iii. Evaluate the PES analytes reported that were misquantified to assess the potential for high and/or low bias in sample results.	iii. Sample data should be qualified based on the misquantified PES analytes identified from the vendor's acceptance criteria. • If any of the PES analytes do not meet acceptance criteria, then the validator should use the PES results to qualify sample data for the specific analytes that are included in the PES sample. Professional judgment should be used to qualify sample data for non-PES analytes, taking into account the analytical problems associated with the analyte either historically or that were encountered by the laboratory. • If a PES analyte recovery is outside the upper limit of the vendor's documented acceptance limits (note: the validator should confirm that the vendor's acceptance limits are calculated as plus and minus three standard deviations from the mean, similar to EPA-PES "Action Limits"), then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that PES to indicate potential high bias.

C. EVALUATION	D. ACTION
2. d. iii. Continued from above.	2. d. iii. Continued from above. - Accept non-detects for the affected analyte in all samples associated with that PES. • If a PES analyte recovery is outside the lower limit of the vendor's documented acceptance limits (see note above, Section 2.d.iii), then the validator should: - Estimate (J) positive detects for the affected analyte in all samples associated with that PES to indicate potential low bias. - Reject (R) non-detects for the affected analyte in all samples associated with that PES to indicate that the data are unusable due to the possibility of false negatives. • If more than half of the PES analyte recoveries for one PES analyzed by a particular method are outside the upper limit of the vendor's documented acceptance limits, or if more than half of the PES analyte recoveries for a particular method are outside the lower limit of the vendor's documented acceptance limits, then the validator should use professional judgment to apply the above validation actions to all positive detects and non-detects in all samples associated with that PES.

C. EVALUATION	D. ACTION
2. d. iii. Continued from above.	2. d. iii. Continued from above. • If more than half of the PES analyte recoveries for a particular method are outside the vendor's documented acceptance limits in one PES (see note above, Section 2.d.iii), where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte or all analytes for samples associated with that PES. • Based upon the number of analytes misquantified and a review of the project DQOs, the validator should use professional judgment to determine if the data set for an entire fraction or parameter is unusable and, therefore, should be rejected. Rejected data should be returned to the laboratory and payment denied.
iv. Evaluate ICP-MS internal standards for the non-EPA PES.	iv. Action on non-compliant internal standard relative intensities should follow the guidance provided in Section VII (ICP-MS Internal Standards). Professional judgment should be used to evaluate the impact that non-compliant non-EPA PES internal standard relative intensities have on the sample data.
* e. Determine what percentage of PES analytes were below or above PES acceptance criteria.	e. If more than half of the PES analytes for a particular method are high or low, then the validator should check the raw data and/or contact the laboratory to verify that the PE sample was prepared according to the PE instructions (if applicable). Check also that the appropriate PE instructions (for that PE concentration level) were sent to the laboratory.

C. EVALUATION	D. ACTION
*2. f. Check and recalculate the analytical concentrations for at least one analyte per method per PES. Verify that the recalculated value agrees within ±10% of the reported result.	2. f. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum. i. If corrected data reports affect the original results reported on the initial EPA PES score report, then the validator should resubmit the corrected PES results to Region I OEME-QA for a PES rescore. Sample data should be reevaluated and requalified based on the corrected PES data. ii. If corrected data reports affect the original results reported for the initial non-EPA PES, then the validator should reevaluate and requalify sample data based on the corrected PES data.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1.d, C.2.e, C.2.f

Table INORG-XIII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LCS RESULTS*

Sample Results	Aqueous LCS % Recovery				
	%R < 50%	50% ≤ %R < LL	LL ≤ %R ≤ UL	UL < %R ≤ 150%	%R > 150%
Detects	R	J	A	J	R
Non-detects	R	UJ	A	A	A

Sample Results	Non-aqueous LCS Result		
	Result < LL	LL ≤ Result ≤ UL	Result > UL
Detects	J	A	J
Non-detects	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

* If more than half of the LCS analyte recoveries for a particular method fall within one of the above categories, then professional judgment may be used to apply the action to all analytes in all samples associated with that LCS. Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table INORG-XIII-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON PES RESULTS*

Sample Results	• Single Blind • Double Blind PES < Lower Limit "Action Low" or "Analyte Missed"	• Single Blind • Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	• Single Blind • Double Blind PES > Upper Limit "Action High"
Detects	J	A	J
Non-Detects	R	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

* If more than half of the PES analytes fall within one of the above categories, then professional judgment may be used to apply the action to all analytes in all samples associated with that PES. Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

E. EXAMPLES

Example #1: (One LCS analyte < lower LCS limit; One LCS analyte > upper LCS limit)

An aqueous Laboratory Control Sample (LCS) containing 22 analytes is found to have silver with a percent recovery of 60% and zinc with a percent recovery of 130%. The method QC acceptance criteria for aqueous LCS recoveries are 80-120% (exception: there are no method QC acceptance criteria for silver). The validator estimates (J) positive detects for zinc and silver in all field samples associated with that LCS. The validator accepts the zinc non-detects and estimates (UJ) the silver non-detects in all field samples associated with that LCS. The validator reports qualified data on the Data Summary Table and notes in the Data Validation Memorandum that the zinc positive detects are biased high, the silver positive detects are biased low, and the silver non-detects contain possible false negatives.

Example #2: (One Single Blind PES analyte < lower PES acceptance limit)

A Single Blind Performance Evaluation Sample (PES) is found to have a selenium positive result that scored below the lower PES acceptance limit. Therefore, the validator estimates (J) positive selenium detects and rejects (R) the non-detects for selenium in all field samples associated with that PES. The validator reports the qualified data on the Data Summary Table and notes in the Data Validation Memorandum that the positive selenium detects are biased low and selenium non-detects are rejected due to the possibility of false negatives.

Example #3: (More than one-half of PES analytes analyzed by ICP-AES > upper PES acceptance limits)

A Single Blind PES containing 22 analytes analyzed by ICP-AES is found to have more than one-half of the spiked PES analytes analyzed with % recoveries above the upper PES acceptance limits. The validator uses professional judgment to estimate (J) all positive detects identified by ICP-AES in all field samples associated with that PES and accept all ICP-AES non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and notes that the positive ICP-AES results are biased high in the Data Validation Memorandum.

Example #4: (More than one-half of PES analytes "Action High" or "Action Low")

A Single Blind PES is found to have more than one-half of the 22 spiked PES analytes analyzed by ICP-AES with results that do not meet PES acceptance criteria. Some of the PES analytes are flagged "Action Low" and some are flagged "Action High". The validator determines that analytical error yields uncertainty in quantitative accuracy which may adversely affect site decisions. Therefore, the validator uses professional judgment to estimate (J) all positive detects in all field samples associated with that PES and reject (R) all non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and discusses the limited use of the data in the Data Validation Memorandum.

E. EXAMPLES (continued)

Example #5: (One PES "Analyte Missed")

A Single Blind PES is found to have one PES "Analyte Missed" for antimony which is a contaminant of concern at the site. The validator estimates (J) all positive antimony detects and rejects (R) all antimony non-detects in all field samples associated with that PES. The validator reports qualified data on the Data Summary Table and discusses this in the Data Validation Memorandum.

Example #6: (One PES "Contaminant", also in blank)

A Single Blind PES is found to have one PES "Contaminant", sodium, at 450 ppb. The preparation blank contained 80 ppb of sodium, resulting in a Blank Action Level of 400 ppb. The validator uses the sodium Blank Action Level to evaluate the sample data and reports qualified data on the Data Summary Table. The validator suspects that the sodium false positive PES analyte is a result of laboratory contamination and discusses this in the Data Validation Memorandum. PES results are not reported on the Data Summary Table.

Example #7: (One PES "Contaminant", not in blank)

A Single Blind PES is found to have one PES "Contaminant", barium, which is not detected in any of the blanks but is detected in two samples. The validator determines that the barium is an interference specific to the PES because it was not detected in any of the preparation or instrument blanks. The validator uses professional judgment to accept the positive barium detects in the field samples. The validator reports the data unqualified on the Data Summary Table and discusses this in the Data Validation Memorandum.

XIV. ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS

A. OBJECTIVE

The objective for the evaluation of analyte quantitation and reported quantitation limits is to ensure that reported quantitative results and quantitation limits are accurate. To this end, laboratory calculations from raw data to the final reported concentrations are checked for accuracy.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. Reported quantitation limits must meet project-required DQOs.

2. a. Reported concentrations for positive detects and analyte quantitation limits for non-detects and adjustments of those concentrations/analyte quantitation limits must be calculated according to the appropriate method requirements.

   b. Reported concentrations for positive detects and analyte quantitation limits for non-detects must be adjusted for percent solids, dilutions, concentrations, and sample preparation procedures that are not accounted for in the method.

3. Target analyte quantitation must be based on the masses and internal standards (ISs) specified in the method, if applicable.

4. Target analyte quantitation must be within the initial calibration range or within the established linear range of the ICP, if applicable.

5. All soil/sediment sample results must be adjusted for percent solids and must have percent solids greater than 30 percent.[1]

   Sediment samples are collected at CERCLA sites to establish whether or not the presence of hazardous chemicals has impacted the resident organisms and their natural environment. The data quality objectives for ecological risk assessment generally require that the analytical method used for sediment analysis achieve, at a minimum, the dry weight CLP SOW quantitation limits.

[1]U.S. EPA Office of Water Regulations and Standards Industrial Technology Division - Method 1620, p. 29, Section 14.16, Draft September 1989.

5. (Continued)

Most analytical methods that deal with soil-type matrices are applicable to both soils and sediments with no difference in how those two matrices are prepared and analyzed. Since a definition for soil and sediment matrices is not provided in the analytical methodology, Region I has adopted the definition for soil samples used by the Office of Water Regulations and Standards Industrial Technology Division (ITD). This definition states that soil samples are "soils, sediments, and sludge samples containing more than 30% solids".

High moisture sediments may or may not not be successfully analyzed by routine CLP analytical methods. Additional sampling and analytical preparation steps may need to be employed. For example, standing water may first be decanted, and then the sample may be centrifuged or filtered to remove excess water. To achieve the dry weight quantitation limits, the laboratory must perform a percent solids analysis prior to preparation and the initial volume of sample digested/distilled must be increased accordingly. This presumes that the samplers have collected sufficient volume, above and beyond normal volume requirements, so that additional sample can be digested/distilled. As a last resort, the laboratory can decrease the final prepared volume.

Sampling and analytical methodologies must be determined during project scoping processes and must be based on the project data quality objectives. For more information, see Attachment A of the Data Validation Manual.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from analyte quantitation anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the reported quantitation limits meet project-required DQOs.	1. If reported quantitation limits do not meet the project-required DQOs, then the validator must investigate and document the cause of the deficiency and use professional judgment to assess sample data.

C. EVALUATION	D. ACTION
*2. a. Recalculate from the raw data the concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or a soil sample) for each method, in every field sample to verify that laboratory-reported sample results were accurately calculated according to the method.	2. a. If incorrect values, equations or factors have been used to calculate sample results and/or sample quantitation limits, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
* b. Verify that the concentrations for positive detects and sample quantitation limits have been adjusted to reflect sample dilutions, concentrations, sample preparation factors, and dry weight factors that are not accounted for in the method.	b. If the concentrations for positive detects and/or sample quantitation limits were not correctly adjusted for sample dilutions, concentrations, preparation methods, or dry weight factors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. Verify that the concentrations for positive detects are within the initial calibration range of the instrument or within the established linear range of the ICP, if applicable.	3. a. If the concentrations for positive detects exceed the upper limit of the initial calibration range or exceed the linear range of the ICP, if applicable, and no dilutions were reported, then the validator should estimate (J) those positive detects that exceed the initial calibration range or linear range. b. If the concentrations for positive detects fall below the lower limit of the initial calibration range (or below the lowest concentration standard), then the validator should estimate (J) those positive detects.
4. Ascertain if any soil/sediment/solid sample has less than or equal to 30 percent solids. Determine if appropriate sampling and/or analytical preparation steps were employed for high moisture content samples.	4. a. If a soil/sediment/solid sample has greater than 30 percent solids, then the validator should accept all sample data. b. If a soil/sediment/solid sample has percent solids of less than or equal to 30% but greater than or equal to 10%, then the validator should: • Estimate (J) positive detects. • Reject (R) non-detects. c. If a soil/sediment/solid sample has less than 10 percent solids, then the validator should reject (R) positive and non-detect sample results as unusable.

C. EVALUATION	D. ACTION
4. Continued from above.	4. d. The validator should include a discussion of the sample matrices having low percent solids in the Data Validation Memorandum. The validator may need to contact the field sampler to determine whether sampling techniques were appropriate for the sample matrix. e. If any sampling and/or analytical preparation steps were employed to address high moisture soil/sediment/solid samples, such as removing the aqueous medium or increasing the sample size to account for low percent solids, then the validator should use professional judgment to determine whether the associated sample data should be qualified or accepted and whether project DQOs were achieved. The validator must take into consideration the dry weight quantitation limits, whether the sampling and analytical methods were appropriate for the sample matrix, and the project DQOs. The Data Validation Memorandum should include a discussion of the rationale for data qualification and/or data acceptance.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.2.a, C.2.b

Table INORG-XIV-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON SAMPLE PERCENT SOLIDS*

Sample Result	% Solids > 30%	10% ≤ % Solids ≤ 30%	% Solids < 10%
Detects	A	J	R
Non-detects	A	R	R

* Professional judgment should be used to accept, qualify or reject the associated sample data when sampling and/or analytical preparation steps were employed to address high moisture soil/sediment samples.

E. EXAMPLES

Example #1: (10% ≤ % Solids ≤ 30%)

DQOs for the Maple Street site specify that soil samples be analyzed for low level metals to assess human health risk posed by the site contamination. Metals soil sample MAAH72 had 20% solids. Due to the low percent solids, all positive results are estimated (J) and all non-detects are rejected (R) as unusable because the elevated sample quantitation limits do not meet project DQOs. The validator reports the qualified data on the Data Summary Table and notes this problem in the Data Validation Memorandum.

Example #2: (% Solids < 10%)

Sediment sample MAGH11 had 9% solids. As a result of the extremely low percent solids (< 10%), the validator rejects (R) as unusable all positive detects and non-detects for this sample. The validator contacts the field sampler to determine if sampling techniques were inappropriate for the sample matrix resulting in high moisture content. The validator reports the qualified data on the Data Summary Table and discusses the high moisture content of the sample and the inappropriateness of the sampling and/or analytical methods in the Data Validation Memorandum.

Example #3: (10% ≤ % Solids ≤ 30%; Sample weight increased)

Soil sample MACH50 had 20% solids. The validator reviews the SDG narrative and preparation log which indicate that a higher sample weight was employed for sample digestion to address the sample's high moisture content. The validator determines that the dry weight CRQLs and project DQOs were achieved by the increased sample weight. Therefore, the validator uses professional judgment to accept the sample results. The validator reports the data unqualified on the Data Summary Table and discusses the high moisture content, the preparatory method, and the rationale used to accept the data in the Data Validation Memorandum.

XV. SYSTEM PERFORMANCE

A. OBJECTIVE

The objective of assessing overall system performance is to determine if any method preparatory and/or analytical procedures result in qualitative and/or quantitative system error or bias. All sample, QC sample, and blank results are reviewed for accuracy, precision, sensitivity, and contamination to ascertain if there are any general trends in data quality.

B. CRITERIA

Since there are no specific criteria for system performance, professional judgment should be used to assess the overall performance.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
*1. The results of Zero (LCS), Single and Double Blind PESs, MDL studies, LFBs, QL Check Standards, calibration standards, calibration verifications, ICP interference check samples, and matrix spike analyses may be used to assess the overall system accuracy including sample digestion/distillation efficiency and instrument response. * a. Evaluate all PES and other relevant QC data to determine if any analytical trends exist over the sample analysis period. * b. The validator should ascertain from the PES and other relevant QC data if there is a high or low quantitative bias for a particular analyte or group of analytes. * c. The validator should also ascertain from the PES and other relevant QC data if there is a potential for false negatives and/or false positives to be reported. * d. The validator should ascertain from the matrix spike analyses if the sample matrix effects impact analyte recovery, thus indicating a method bias outside the control of the laboratory.	1. The validator should refer to the previous sections for specific guidance in evaluating accuracy using PES/LCS, MDL study, LFB, QL Check Standard, calibration standard, calibration verification, ICP interference check sample, and matrix spike data. If the validator determines that analytical trends indicate a qualitative and/or quantitative systematic bias, then the validator should use professional judgment to determine whether or not to qualify or reject the sample data based on the extent of the impact. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should differentiate between sample matrix-related preparatory and analysis problems that are outside the laboratory's control and those preparatory and analysis problems that are within the laboratory's control.

C. EVALUATION	D. ACTION
*2. The results of the calibration verification, internal standard (ICP-MS), analytical replicate (e.g., replicate integrations), laboratory duplicate, and field duplicate analyses may be used to assess overall system precision. * a. Compare the calibration verification results to ascertain if the instrument generated consistent responses over the sample analysis period. * b. Review the responses of the ICP-MS internal standards for each sample to ascertain if there is a change in instrument response. * c. Review the results of the analytical replicates to evaluate the precision of the individual determinations and to ascertain if there are any trends in precision over the entire analytical run. * d. The validator should evaluate the laboratory duplicate results in conjunction with the field duplicate results to identify any analytical trends, ascertain if sample matrices were homogeneous or heterogeneous, and determine if sampling error may have contributed to field imprecision.	2. The validator should refer to the previous sections for specific guidance on evaluating laboratory and field precision and analytical replicate and internal standard analyses. If the validator determines that an instrument produces erratic responses, then they should use professional judgment to qualify or reject sample data. If laboratory duplicate results indicate laboratory imprecision, then the validator should suspect laboratory technique and take into consideration the field duplicate results when using professional judgment to qualify sample data. If field duplicate results indicate field imprecision resulting from heterogeneous sample matrices or field sampling error, then the validator should use professional judgment to qualify sample data based on the extent of impact. The validator should differentiate between lack of precision due to instrument performance problems and that caused by matrix effects or sampling error.

C. EVALUATION	D. ACTION
*3. The results of the LFB, QL Check Standard, PES, calibration and internal standard (ICP-MS) analyses may be used to assess the overall system sensitivity. * a. Review all low level calibration standards, LFBs, QL Check Standards, and PES data to evaluate sensitivity for each instrument to verify that no instrument has lost its ability to accurately quantitate and identify analytes at the quantitation limit over the sample analysis period, which could potentially result in false negatives and low biased results. * b. Check the responses of the individual sample, QC sample, calibration verification, and blank internal standards (ICP-MS) as well as calibration verification results to monitor instrument sensitivity changes. * c. For instrument printouts which produce a baseline, review the raw data for abrupt shifts in the baseline which may indicate a change in the instrument's sensitivity or zero setting. Evaluate negative values which could indicate a decrease in the instrument's sensitivity caused by instrument or baseline drift, possibly resulting in target analytes at or near the detection limit to miss detection (false negatives). Similarly, the validator should also check for any abrupt shift which may cause a false positive to be reported. A decrease or increase in the baseline may result in incorrect integration and subsequent misquantitation. * d. The validator may determine that instrument sensitivity is adequate but sample matrix effects may preclude obtaining the quantitation limits required by the project DQOs using the analytical method employed.	3. The validator should refer to the previous sections for specific guidance on evaluating sensitivity, accuracy, and analyte identification and quantitation. If the validator determines that instrument sensitivity is unacceptable, then the validator should use professional judgment to qualify or reject the affected sample data. The validator should discuss and justify all technical decisions in the Data Validation Memorandum. The validator should also note if sample matrix interferences did not allow quantitation limits to be achieved and should recommend additional and/or alternate preparatory or analytical methods for future site work.

C. EVALUATION	D. ACTION
*4. The results of the PES and preparation blank, calibration blank, equipment/rinsate blank, and bottle blank analyses may be used to assess overall system contamination. * a. Review all blank and sample results to evaluate the possibility of sample contamination introduced via either cross-contamination from a previously run sample or from general lab contamination. * b. Compare blank analyses to determine if the contamination is instrument related or if the interferents are present in the blank from sample processing activities. * c. Assess whether problematic blank results are reproducible when replicate aliquots are analyzed or are sporadic interferences. Sporadic interferences may indicate that the interferent is introduced from the laboratory environment. The validator should review the raw data for suspected outlier interferents.	4. The validator should refer to the previous sections for specific guidance on evaluating blank contamination. If the validator determines that there is a systematic blank error introduced during sample collection or processing (digestion/distillation or analysis), then the data should be qualified according to Section IV (Blanks). However, if the validator suspects intermittent or sporadic introduction of interferents during analysis, then the validator should use professional judgment to qualify or reject sample data and document and justify all technical decisions in the Data Validation Memorandum.

* **Note: This section is only applicable to a Tier III data validation. If a validator suspects system performance has degraded to the degree that data are affected and a Tier II validation has been requested, then the validator should contact the Site Manager to approve the necessary Tier III validation.**

E. EXAMPLE

Example #1: (Abrupt decrease in baseline)

The validator examines the instrument printout for the mercury analysis and observes a significant abrupt decrease in the baseline during the analysis of the last two samples, MAXN25 and MAXN26, as well as an erratic baseline. The instrument printout shows a steady baseline prior to the analysis of these two samples. The PE samples associated with these samples were acceptable; however, the PE samples were analyzed at the beginning of the run when the baseline was steady. The validator uses professional judgment to estimate (J) the positive detect and reject (R) the non-detect for mercury associated with these two samples. The validator reports the qualified data on the Data Summary Table. The validator notes the sensitivity loss of the mercury analyzer and justifies the decision to qualify sample data in the Data Validation Memorandum.

XVI. OVERALL EVALUATION OF DATA

A. OBJECTIVE

The objective of the final evaluation of a data package is to identify the "analytical error" and any "sampling error" associated with the data. The sum of the "analytical error" and the "sampling error" equals the "measurement error." "Measurement error" will then be used by the end user in conjunction with sampling variability (spatial variations in pollutant concentrations) to determine "total error" (total uncertainty) associated with the data. Ultimately, the end data user will assess data usability in the context of the predetermined Data Quality Objectives (DQOs) and resultant "total error" of the data.

B. CRITERIA

The Sampling and Analysis Plan (SAP) or Quality Assurance Project Plan (QAPP) and DQO Summary Form should specify the site-specific DQOs and acceptable levels of uncertainty or "total error."

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
1. Obtain the SAP, QAPP or DQO Summary Form to review the DQOs of the sampling event.	1. Synopsize in the first section of the Data Validation Memorandum, Overall Evaluation of Data, in bullet format, the appropriate project DQOs for the data package.
2. Evaluate the appropriateness of the analytical method chosen. For example, was the method capable of achieving quantitation limits sufficiently low to meet DQOs for risk assessment? Was the method capable of successfully analyzing each particular matrix sampled?	2. If an inappropriate method was chosen for sample analysis, then the validator should discuss the method deficiencies and identify more appropriate methods or modifications for use in subsequent sampling rounds. The validator should include this discussion in the Overall Evaluation of Data section of the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. Evaluate any analytical problems that were identified.	3. Estimate and describe the "analytical error" that contributes to the "measurement error" associated with the data package in the Overall Evaluation of Data section of the Data Validation Memorandum. a. If "analytical error" causes the data to be unusable, then the validator should reject the data and return it to the laboratory and deny payment. b. If "analytical error" causes the data to be of reduced worth to the Region, then the validator should recommend that the laboratory's payment be reduced.
4. Evaluate any sampling issues that were identified. Note: The validator is only responsible for evaluating those "sampling errors" that are identified during the routine data validation process. Other "sampling errors" may have occurred and they should be assessed by the end user prior to data use.	4. Estimate and describe the "sampling error" that contributes to the "measurement error" associated with the data package in the Overall Evaluation of Data section of the Data Validation Memorandum. Examples of "sampling error" for which the validator would have information include highly contaminated equipment blanks as well as delayed sample shipment that caused holding time violations. a. If "sampling error" severely impacts potential data usability, then the validator should note this in the Data Validation Memorandum. b. The end user should review the results of the sampler's field notes/trip report to determine additional "sampling error" issues with which to fully assess "measurement error."
5. Evaluate data quality in terms of "measurement error" as a combination of "analytical error" and "sampling error."	5. Discuss data quality in terms of "measurement error" as the sum of "analytical error" and "sampling error." All discussions should be included in the Overall Evaluation of Data section of the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. Identify potential usability issues raised by an unacceptable degree of "measurement error."	6. If data usability is potentially compromised by a high degree of "measurement error," then the validator should note this in the Overall Evaluation of Data section of the Data Validation Memorandum. If data quality impacts the use of those data by the end user, then the validator should detail in the Overall Evaluation of Data section of the Data Validation Memorandum how data use will be limited and for which end user, i.e., risk assessor, hydrogeologist, etc.
7. Sampling variability is not assessed during data validation and, therefore, should be assessed by the end user prior to data use.	7. The end user should review the results of the Data Validation Memorandum in conjunction with the sampler's field notes/trip report to assess the impact of sampling variability issues on data usability.

Appendix I

CLP SOW ILM05.4/ Inorganic Analysis
Method QC Criteria, Equations, and Definitions

APPENDIX I

The following method QC criteria, equations, and definitions apply to data generated according to the **USEPA CLP Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration, ILM05.4.**

SECTION I: PRESERVATION AND TECHNICAL HOLDING TIME CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-I-B, for preservation and technical holding time data validation criteria.

SECTION II: ICP-MS TUNE CRITERIA

Refer to the following ICP-MS tune method QC criteria for data validation:

The tuning solution for instrument tuning and mass calibration consists of beryllium, magnesium, cobalt, indium, and lead at 100 ug/L in 1% (v/v) nitric acid. The tuning solution must be performed prior to instrument calibration and may be analyzed as five separate analyses or as a single analysis with at least five integrations.

Mass calibration must be within 0.1 amu over the range of 6 to 210 amu and peak width must be within the manufacturer's specifications. The percent relative standard deviation (%RSD) of the absolute signals for all analytes in the tuning solution must not exceed 5%.

SECTION III: CALIBRATION CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-III-B, for calibration data validation criteria and the following method QC criteria:

Instruments must be calibrated daily or once every 24 hours and each time the instrument is set up.

Initial Calibration:

ICP-AES - A blank and at least one calibration standard must be used to calibrate the instrument. At least two replicate integrations are required for data acquisition.

ICP-MS - A blank and at least one calibration standard must be used to calibrate the instrument. At least three replicate integrations are required for data acquisition.

Cold Vapor Mercury AA - A blank and at least four calibration standards in graduated amounts in the appropriate range must be used to calibrate the instrument. One of the standards must be at the CRQL. The correlation coefficient for the initial calibration curve must be ≥ 0.995.

Cyanide - A blank and at least three calibration standards in graduated amounts in the appropriate range must be used to calibrate the instrument. One of the standards must be equal to the CRQL. The correlation coefficient for the initial calibration curve must be ≥ 0.995.

Initial and Continuing Calibration Verification (ICV/CCV):

An ICV must be analyzed immediately after instrument calibration at each wavelength used for analysis or at each mass used to report final results. If a certified solution for an analyte is not available, then a standard from a different source than that used for instrument calibration must be prepared at a concentration other than that used for calibration. For cyanide, the ICV must be distilled with each batch of samples distilled, and the distilled samples must be analyzed with that particular ICV.

A CCV must be analyzed for each wavelength used for analysis or mass used to report final results at the beginning of the run, at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent, and after the last analytical sample of the run. The analyte concentrations in the CCV standard must be different from the concentrations in the ICV and must be near the mid-range of the initial calibration range.

Table App.I.III-1 - INITIAL AND CONTINUING CALIBRATION VERIFICATION RECOVERY LIMITS

Analytical Method	Parameter	Method QC Criteria % Recovery
ICP-AES/ICP-MS	Metals	90 - 110
Cold Vapor AA	Mercury	80 - 120
Spectrophotometric	Cyanide	85 - 115

The ICV/CCV % recovery is calculated using the following equation:

$$\%\ Recovery = \frac{Found\,(ICV\,/\,CCV)}{True\,(ICV\,/\,CCV)}\ x\ 100$$

Where,
Found (ICV/CCV) = Concentration of the analyte determined in the ICV or CCV.
True (ICV/CCV) = True concentration of the ICV or CCV.

Contract Required Quantitation Limit (CRQL) Check Standard (CRI):

A standard at the CRQL (CRI) must be analyzed for every wavelength used for analysis or mass used to report final results at the beginning of the run, immediately following the ICV/ICB, at a frequency of not less than once per 20 analytical samples per analysis run, and at the end of each sample analysis run. The subsequent CRI analysis must be immediately followed by the CCV/CCB analysis. For ICP-AES, a CRI is not required for aluminum, barium, calcium, iron, magnesium, sodium, and potassium.

The CRI % recovery is calculated using the following equation:

$$\%\ Recovery = \frac{CRI\ Found}{CRI\ True}\ x\ 100$$

Where,
CRI Found = CRI concentration determined for the analyte.
CRI True = True CRI concentration for the analyte.

Table App.I.III-2 - CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) RECOVERY LIMITS

Analytical Method	Parameter	Method QC Criteria % Recovery
ICP-AES	Metals (except Sb, Pb, Tl)	70 - 130
	Sb, Pb, Tl	50 - 150
ICP-MS	Metals (except Co, Mn, Zn)	70 - 130
	Co, Mn, Zn	50 - 150
Cold Vapor AA	Mercury	70 - 130
Spectrophotometric	Cyanide	70 - 130

SECTION IV: BLANK CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-IV-B, for blank validation criteria and the following method QC criteria:

Method-Required Blanks

Preparation Blank - Contains all of the reagents, in the same volumes which were used to process samples. At least one preparation blank, processed through each sample preparation and analysis procedure, must be prepared and analyzed with each SDG or with each batch of samples prepared, whichever is more frequent.

Calibration Blank - Contains reagents in reagent water. A calibration blank must be analyzed at each wavelength used for analysis or mass used to report final results immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent, and at the beginning of the run and after the last analytical sample. (For mercury, the calibration blank is carried through the entire preparation and analysis procedure.)

SECTION V: ICP-AES INTERFERENCE CHECK SAMPLE (ICS) CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-V-B, for ICS validation criteria and the following method QC criteria:

An ICS analysis consists of analyzing Solution A (ICSA) and Solution AB (ICSAB) consecutively (starting with the ICSA) for all target analytes and interferents (target and non-target), at the beginning and end of each analytical run (but not before the ICV) and at a frequency of not less than once per 20 analytical samples per analysis run.

Table App.I.V-1 - INTERFERENT AND ANALYTE CONCENTRATIONS IN THE ICP-AES INTERFERENCE CHECK SAMPLE (ICS)

Analytes	Conc. (mg/L)	Interferents	Conc. (mg/L)
Silver	0.2	Aluminum	250
Arsenic	0.1	Calcium	250
Barium	0.5	Iron	100
Beryllium	0.5	Magnesium	250
Cadmium	1.0		
Cobalt	0.5		
Chromium	0.5		
Copper	0.5		
Manganese	0.5		
Nickel	1.0		
Lead	0.05		
Antimony	0.6		
Selenium	0.05		
Thallium	0.1		
Vanadium	0.5		
Zinc	1.0		

Note: ICS Solution A (ICSA) contains the interferents at the indicated concentrations; ICS Solution AB (ICSAB) contains all of the analytes plus all of the interferents in Solution A at the indicated concentrations.

Note: The CLP SOW ILM05.4 ICS method acceptance criteria differ from the Region I Functional Guidelines ICS criteria. If data quality objectives allow for greater variability of data, then expanded ICS validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP.

The ICS % recovery is calculated using the following equation:

$$ICS\ \%\ Recovery = \frac{Found\ \ Solution\ \ A/AB}{True\ \ Solution\ \ A/AB}\ x\ 100$$

Where,
Found Solution A/AB = Concentration (positive, negative, or zero) of the analyte measured in the ICSA or ICSAB.
True Solution A/AB = True concentration of the analyte in the ICSA or ICSAB.

SECTION VI: ICP-MS INTERFERENCE CHECK SAMPLE (ICS) CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-VI-B, for ICS validation criteria and the following method QC criteria:

An ICS analysis consists of analyzing Solution A (ICSA) and Solution AB (ICSAB) consecutively (starting with ICSA) for all target analytes and monitoring for all interferents at the beginning of each analytical sequence (but not before the ICV).

Table App.I.VI-1 - INTERFERENT AND ANALYTE CONCENTRATIONS IN THE ICP-MS INTERFERENCE CHECK SAMPLE (ICS)

Analytes	Conc. (μg/L)	Interferents	Conc. (mg/L)
Antimony	20	Aluminum	100
Arsenic	20	Calcium	100
Barium	20	Iron	100
Beryllium	20	Magnesium	100
Cadmium	20	Potassium	100
Chromium	20	Sodium	100
Cobalt	20	Phosphorus (as ortho-phosphate)	100
Copper	20	Sulfur (as sulfate)	100
Lead	20	Carbon	200
Manganese	20	Chloride	1000
Nickel	20	Molybdenum	2
Selenium	20	Titanium	2
Silver	20		
Thallium	20		
Vanadium	20		
Zinc	20		

Note: ICS Solution A (ICSA) contains the interferents at the indicated concentrations; ICS Solution AB (ICSAB) contains all of the analytes plus all of the interferents in Solution A at the indicated concentrations.

Note: The CLP SOW ILM05.4 ICS method acceptance criteria differ from the Region I Functional Guidelines ICS criteria. If data quality objectives allow for greater variability of data, then expanded ICS validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the

QAPP.

The ICS % recovery is calculated using the following equation:

$$ICS\ \%\ Recovery = \frac{Found\ Solution\ A/AB}{True\ Solution\ A/AB} x\ 100$$

Where,
Found Solution A/AB = Concentration (positive, negative, or zero) of the analyte measured in the ICSA or ICSAB.
True Solution A/AB = True concentration of the analyte in the ICSA or ICSAB.

SECTION VII: ICP-MS INTERNAL STANDARD CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-VII-B, for ICP-MS internal standard data validation criteria and the following method QC criteria:

For full range mass scans, a minimum of five internal standards must be used and the masses of the internal standards must bracket the masses of the analytes. Internal standards must be added to all samples, standards, and blanks at identical levels and in a similar manner. The concentrations of the internal standards should be sufficiently high for good precision and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. A concentration range of 20 ug/L to 200 ug/L for each internal standard is recommended.

Acceptable internal standards are listed below:

Table App.I.VII-1 - ICP-MS INTERNAL STANDARDS

Internal Standard	Mass
Lithium	6
Scandium	45
Yttrium	89
Rhodium	103
Indium	115
Terbium	159
Holmium	165
Lutetium	175
Bismuth	209

The absolute response of any internal standard must not deviate by more than 60-125% of the original response in the blank calibration standard.

If the internal standard does not meet the acceptance criteria, then the sample must be diluted by a factor of two, internal standards added, and the sample reanalyzed. If the internal standard responses for the diluted sample analysis are within criteria, then the results from the diluted sample analysis are reported. If the internal standard responses for the diluted sample analysis are not within criteria, then the results of the original undiluted sample

analysis are reported.

The internal standard percent relative intensity (%RI) is calculated using the following equation:

$$\%RI = \frac{I_n}{I_o} x\ 100$$

Where,
I_n = IS intensity in the sample.
I_o = IS intensity in the blank calibration standard.

SECTION VIII: MATRIX SPIKE CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-VIII-B, for matrix spike data validation criteria and the following method QC criteria:

A matrix spike sample must be analyzed for each group of samples of a similar matrix or for each SDG. The matrix spike analytes are spiked at the following concentrations:

ICP-AES: Water - aluminum and barium at 2000 µg/L; iron at 1000 µg/L; cobalt, manganese, nickel, vanadium, and zinc at 500 µg/L; copper at 250 µg/L; chromium at 200 µg/L; antimony at 100 µg/L; beryllium, cadmium, selenium, silver, and thallium at 50 µg/L; arsenic at 40 µg/L; and lead at 20 µg/L. Soil - spike concentrations are a factor of 5 less than the water spike concentrations in mg/kg (e.g., cobalt is at 100 mg/kg); no spike is required for aluminum and iron.

ICP-MS: Water - Barium at 2000 µg/L; cobalt, manganese, nickel, vanadium, and zinc at 500 µg/L; copper at 250 µg/L; chromium at 200 µg/L; antimony at 100 µg/L; beryllium, cadmium, silver, and thallium at 50 µg/L; arsenic at 40 µg/L; lead at 20 µg/L; and selenium at 10 µg/L.

Hg: 1 µg/L (water); 0.5 mg/kg (soil).

Cyanide: 100 µg/L in the final sample solution for analysis, e.g., 25 µg cyanide for a 250 mL final distillate volume (non-midi-distillation); 5 µg cyanide for a 50 mL final distillate volume (mini-distillation).

The method QC criteria for matrix spike recovery is 75-125%. When the sample concentration exceeds the spike added concentration by a factor of 4 or more, the method QC criteria for recovery do not apply.

If the matrix spike recovery is outside the method QC criteria and the sample result does not exceed 4 times the spike added concentration, a post-digestion/distillation spike must be performed using the same sample that was used for the matrix spike analysis for ICP-AES, ICP-MS, and cyanide (not mercury). The post-digestion spike concentration must be 2x the indigenous level or 2x the CRQL, whichever is greater.

The matrix spike sample % recovery is calculated using the following equation:

$$Matrix\ Spike\ \%\ Recovery = \frac{SSR - SR}{SA} x\ 100$$

Where,
SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

SECTION IX: LABORATORY DUPLICATE SAMPLE CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-IX-B, for laboratory duplicate sample data validation criteria and the following duplicate sample method QC criteria:

A laboratory duplicate sample must be analyzed for each group of samples of a similar matrix or for each SDG. Duplicate sample analysis is required for percent solids determination.

The method QC criteria for laboratory duplicate samples are as follows: For original and duplicate sample values ≥5xCRQL, the relative percent difference (RPD) must be ≤20%. If either the sample or the duplicate sample value is <5xCRQL, then the absolute difference between the two must be ≤CRQL. If both the sample and duplicate sample values are < CRQL or <MDL, then no control limit applies.

The duplicate sample relative percent difference (RPD) is calculated using the following equation:

$$RPD = \frac{|S - D|}{(S + D)/2} x\ 100$$

Where,
S = Sample result (original)
D = Duplicate sample result

SECTION X: FIELD DUPLICATE CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-X-B, for field duplicate data validation criteria.

SECTION XI: ICP SERIAL DILUTION CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-XI-B, for ICP serial dilution data validation criteria and the following method QC criteria:

The ICP serial dilution analysis must be performed for each group of samples of a similar matrix or for each SDG, whichever is more frequent.

The serial dilution (a five-fold dilution) must be within 10% of the original sample determination after correction for dilution, if the analyte concentration is minimally a factor of 50 above the MDL in the original sample.

The serial dilution percent difference (%D) is calculated using the following equation:

$$\%D = \frac{|I - S|}{I} x\ 100$$

Where,
I = Initial sample result (instrument reading)
S = Serial dilution result (instrument reading x 5)

SECTION XII: SENSITIVITY CHECK CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-XII-B, for sensitivity check data validation criteria.

SECTION XIII: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-XIII-B, for accuracy check data validation criteria and the following method QC criteria for laboratory control samples (Zero Blind PES):

An aqueous or solid laboratory control sample (LCS) must be analyzed for each analyte and for each matrix, SDG, or each batch of samples prepared, whichever is more frequent. An aqueous LCS is not required for mercury and cyanide (the ICV serves as the aqueous LCS).

The % recovery method QC criteria for the aqueous LCS are 80 -120% for all elements (except Ag and Sb by ICP-AES).

For the solid LCS, use the control limits documented and provided with the certified solid LCS.

The LCS % recovery is calculated using the following equation:

$$LCS\ \%\ Recovery = \frac{LCS\ found}{LCS\ true} x\ 100$$

Where,
LCS found = Concentration of the analyte measured in the LCS.
LCS true = True concentration of the analyte in the LCS.

SECTION XIV: ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-XIV-B, for analyte quantitation and reported quantitation limit data validation criteria and the following method QC criteria:

Target analytes must be quantitated using the established initial calibration. Internal standardization must also be used for ICP-MS. The sample target analytes must be reported to the CRQLs listed below.

Table App.I.XIV-1 - INORGANIC TARGET ANALYTE LIST (TAL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) for ILM05.4 SOW

Analyte	Quantitation Limits[1,2]		
	ICP-AES Water[3] (µg/L)	ICP-AES Soil[3,4] (mg/kg)	ICP-MS Water (µg/L)
Aluminum	200	20	--
Antimony	60	6	2
Arsenic	10	1	1
Barium	200	20	10
Beryllium	5	0.5	1
Cadmium	5	0.5	1
Calcium	5000	500	--
Chromium	10	1	2
Cobalt	50	5	1
Copper	25	2.5	2
Iron	100	10	--
Lead	10	1	1
Magnesium	5000	500	--
Manganese	15	1.5	1
Mercury[3]	0.2	0.1	--
Nickel	40	4	1
Potassium	5000	500	--
Selenium	35	3.5	5
Silver	10	1	1
Sodium	5000	500	--
Thallium	25	2.5	1
Vanadium	50	5	5
Zinc	60	6	2
Cyanide[3]	10	2.5	--

[1]The CRQLs are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).
[2]Method Detection Limits (MDLs) must be less than one-half of the CRQLs.
[3]Mercury is analyzed by cold vapor atomic absorption. Cyanide is analyzed by colorimetry/spectrophotometry.
[4]The CRQLs for soil are based on 100% solids. Samples quantitation limits must be adjusted for % solids, preparation factors, and dilutions.

SAMPLE CONCENTRATION FOR ICP-AES - The amount of analyte present in the sample is calculated using the following equations:

Sample concentration for water:

$$\mu g / L = \frac{(C)(V_f)(DF)}{(V_i)}$$

Where,
C = Instrument value in µg/L
V_f = Final digestion volume (mL)
V_i = Initial digestion volume (mL)
DF = Dilution Factor

Sample concentration for soil/sediment:

$$mg / kg\,(dry\,wt.) = \frac{(C)(V)(DF)}{(W)(S)}$$

Where,
C = Concentration (mg/L)
V = Final sample volume in Liters (L)
W = Wet sample weight (kg)
S = % Solids/100
DF = Dilution Factor

MDL/CRQL CALCULATIONS FOR ICP-AES

Adjusted MDL/CRQL for water:

The adjusted MDL or adjusted CRQL for water samples is calculated by substituting the value of the MDL (µg/L) or CRQL (µg/L) into the "C" term in the water equation above.

Adjusted MDL/CRQL for soil/sediment:

$$Adjusted\ MDL / CRQL = (C)\,x\,\frac{(W_M)(V_R)(DF)}{(W_R)(V_M)(S)}$$

Where,
C = MDL or CRQL concentration (mg/kg)
W_M = Minimum method-required wet sample weight (g)
W_R = Reported wet sample weight (g)
V_M = Method-required final sample volume (mL)
V_R = Reported final sample volume (mL)
S = % Solids/100
DF = Sample Dilution Factor

SAMPLE CONCENTRATION FOR ICP-MS - The amount of analyte present in the sample is calculated using the following equations:

Sample concentration for water prepared by method HW2:

$$\mu g / L = \frac{(C)(V_f)(V_f)(DF)}{(V_i)(20)}$$

Where,
C = Instrument value in µg/L
V_f = Final digestion volume (50 mL)

V_i = Initial digestion volume (100 mL)
DF = Dilution Factor

Sample concentration for water prepared by method HW3:

$$\mu g / L = \frac{(C)(V_f)(DF)}{(V_i)}$$

Where,
C = Instrument value in µg/L
V_f = Final digestion volume (mL)
V_i = Initial digestion volume (mL)
DF = Dilution Factor

MDL/CRQL CALCULATIONS FOR ICP-MS

Adjusted MDL/CRQL for water:

The adjusted MDL or adjusted CRQL for water samples is calculated by multiplying the value of the MDL (µg/L) or CRQL (µg/L) by the sample dilution factor.

SAMPLE CONCENTRATION FOR MERCURY - The amount of analyte present in the sample is calculated using the following equations:

Sample concentration for water by the Automated Technique:

A linear regression equation is used to determine the sample concentration. If samples were diluted for analysis, multiply the results from the linear regression by the dilution factor.

Sample concentration for water by the Manual Technique:

$$\mu g / L = \frac{(\mu g\ Hg, curve)(1000\, mL)}{(aliquot\ volume, mL)(1L)}$$

Sample concentration for soil/sediment:

$$mg / kg\,(dry\ wt.) = (\mu g / g) = \frac{(C)(0.1\,L)}{(W)(S)}$$

Where,
C = Concentration from curve (µg/L)
W = Wet sample weight (g)
S = % Solids/100

MDL/CRQL CALCULATIONS FOR MERCURY

Adjusted MDL/CRQL for water:

The adjusted MDL or adjusted CRQL for water samples is calculated by multiplying the value of the MDL (µg/L) or CRQL (µg/L) by the sample dilution factor.

Adjusted MDL/CRQL for soil/sediment:

$$Adjusted\ MDL/CRQL = (C)\,x\,\frac{(W_M)(DF)}{(W_R)(S)}$$

Where,
C = MDL or CRQL concentration (mg/kg)
W_M = Method-required wet sample weight (g)
W_R = Reported wet sample weight (g)
S = % Solids/100
DF = Sample Dilution Factor

SAMPLE CONCENTRATION FOR CYANIDE - The amount of analyte present in the sample is calculated using the following equations:

Non-Midi-Distillation

Sample concentration for water by semi-automated colorimetric determination (non-midi-distillation):

A linear regression equation is used to determine the sample concentration. Multiply the results by one-half since the original volume was 500 mL and the distillate volume was 250 mL. If samples were diluted for analysis, multiply the results by the dilution factor.

Sample concentration for water by manual colorimetric determination (non-midi-distillation):

$$\mu g/L = \frac{(A)(1000\,mL/L)(50\,mL)}{(B)(C)}$$

Where,
A = µg CN read from standard curve (per 250 mL)
B = mL of original sample for distillation
C = mL taken for colorimetric analysis
50 mL = Volume of original sample aliquot
1000 mL/L = Conversion mL to L
The minimum value that can be substituted for A is the Method Detection Limit (MDL) value adjusted for volume.

Sample concentration for soil/sediment by semi-automated colorimetric determination (non-midi-distillation):

$$mg/kg\,(dry\,wt.) = \frac{(A)(0.25)}{(C)(S)}$$

Where,
A = µg/L determined from standard curve
C = Wet weight of original sample (g)
0.25 = Conversion factor for distillate final volume
S = % Solids/100
The minimum value that can be substituted for A is the MDL value.

Sample concentration for soil/sediment by manual colorimetric determination (non-midi-distillation):

$$mg/kg\,(dry\,wt.) = \frac{(A)(50\,mL)}{(C)(B)(S)}$$

Where,
A = µg CN read from standard curve (per 250 mL)
B = mL of distillate taken for colorimetric determination
C = Wet weight of original sample (g)
50 mL = Standard volume taken for colorimetric determination
S = % Solids/100

Midi-Distillation

Sample concentration for water by semi-automated colorimetric determination (midi-distillation):

$$\mu g/L = \frac{(A)(D)(F)}{(B)}$$

Where,
A = µg/L CN of sample from regression analysis
B = Volume of original sample for distillation (0.050 L)
D = Any dilution factor necessary to bracket sample value within standard values
F = Sample receiving solution volume (0.050 L)
The minimum value that can be substituted for A is the MDL value.

Sample concentration for soil/sediment by semi-automated colorimetric determination (midi-distillation):

$$mg/kg\,(dry\,wt.) = \frac{(A)(D)(F)}{(B)(E)}$$

Where,
A = µg/L CN of sample from regression analysis curve
B = Wet weight of original sample
D = Any dilution factor necessary to bracket sample value within standard values
E = % Solids/100
F = Sample receiving solution volume (0.050 L)
The minimum value that can be substituted for A is the MDL value.

MDL/CRQL CALCULATIONS FOR CYANIDE

Adjusted MDL/CRQL for water (non-midi-distillation, semi-automated colorimetric determination):

Multiply the MDL or CRQL for water samples by one-half (1/2) since the original volume was 500 mL and the distillate volume was 250 mL. If samples were diluted for analysis, multiply only the MDL (µg/L) or CRQL (µg/L) by the dilution factor.

Adjusted MDL/CRQL for water (non-midi-distillation, manual colorimetric determination):

$$Adjusted\ MDL/CRQL = \frac{(A)(1000\,mL/L)(50\,mL)}{(B)(C)}$$

Where,
A = MDL or CRQL concentration (µg/L) X 0.25
B = mL of original sample for distillation

C = mL taken for colorimetric analysis
50 mL = Volume of original sample aliquot
1000 mL/L = Conversion mL to L

Adjusted MDL/CRQL for water (midi-distillation, semi-automated colorimetric determination):

$$Adjusted\ MDL/CRQL = (C)\,x\,\frac{(D)(F)}{(B)}$$

Where,
C = MDL or CRQL concentration (µg/L)
B = Volume of original sample for distillation (0.050 L)
D = Any dilution factor necessary to bracket sample value within standard values
F = Sample receiving solution volume (0.050 L)

Adjusted MDL/CRQL for soil/sediment (all methods):

$$Adjusted\ \ MDL\ /CRQL\ = (C)\,x\,\frac{(W_M)(DF)}{(W_R)(S)}$$

Where,
C = MDL or CRQL concentration (mg/kg)
W_M = Minimum method-required wet sample weight (g)
W_R = Reported wet sample weight (g)
S = % Solids/100
DF = Dilution Factor

SECTION XV: SYSTEM PERFORMANCE CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-XV-B, for system performance data validation criteria.

SECTION XVI: OVERALL ASSESSMENT CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, Section INORG-XVI-B, for overall assessment data validation criteria.

Appendix J

Inorganic Functional Guidelines Action Tables

Note: This appendix is a compilation of the data validation actions that appear in tabular format in Part IV - INORGANIC Data Validation Guidelines. Other actions that are not presented in tabular format are not contained in this appendix and the validator must refer to Part IV to obtain the complete set of actions.

APPENDIX J

Table INORG-I-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON PRESERVATION

Sample Results	Aq. Metals: HNO_3 to pH < 2? Aq. Cyanide[1]: NaOH to pH > 12?		Temperature Criteria Met?	
	Y	N	Y	N
Detects	A	J	A	Professional Judgment
Non-detects	A	UJ or R*	A	Professional Judgment

* Professional judgment may be used to estimate (UJ) or reject (R) non-detects based on the analyte's stability and magnitude of exceedance.

[1] Estimate (J) positive detects and use professional judgment to estimate (UJ) or reject (R) non-detects when cyanide samples are not tested nor treated, if needed, for oxidants or sulfides.

Table INORG-I-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON TECHNICAL HOLDING TIMES

Sample Results	Technical Holding Time (Aqueous/Soil/Sediment)	
	Metals: HT ≤ 6 Months	Metals: HT > 6 Months
	Mercury: HT ≤ 28 Days	Mercury: HT > 28 Days
	Cyanide: HT ≤ 14 Days	Cyanide: HT > 14 Days
Detects	A	J
Non-detects	A	UJ or R*

* Professional judgment may be used to estimate or reject non-detects based on the analyte's stability, magnitude of exceedance, and the effects of additional storage on the sample results.

- For other matrices, estimate (J) positive detects and use professional judgment to estimate (UJ) or reject (R) non-detects when Region I technical holding time criteria are not met.

Table INORG-II-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON THE ICP-MS TUNE

Sample Results	Mass Calibration (amu) > QC Limit	Mass Resolution/ Peak Width (amu) > QC Limit	%RSD > QC Limit
Detects	J	J	J
Non-detects	UJ	UJ	UJ

Table INORG-III-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON ICV AND CCV PERCENT RECOVERIES

Sample Results	% Recovery				
	ICP: %R < 75% Hg/CN: %R < 70%	ICP: 75% ≤ %R < LL Hg/CN: 70% ≤ %R < LL	LL ≤ %R ≤ UL	ICP: UL < %R ≤ 125% Hg/CN: UL < %R ≤ 130%	ICP: %R > 125% Hg/CN: %R > 130%
Detects	R	J	A	J	R
Non-detects	R	UJ	A	A	A

LL = Lower limit of method QC acceptance criteria
UL = Upper limit of method QC acceptance criteria

Table INORG-III-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON QUANTITATION LIMIT CHECK STANDARD RECOVERIES

Sample Results	% Recovery				
	%R < 50%	50% ≤ %R < LL	LL ≤ %R ≤ UL	UL < %R ≤ 180%	%R > 180%
Detects*	J/R (< 2x TV) ** Prof. Judg. (≥ 2x TV)**	J (< 2x TV)	A	J (< 2x TV)	J/R (< 2x TV)** Prof. Judg. (≥ 2x TV)**
Non-detects	R	UJ	A	A	A

LL = Lower limit of method QC acceptance criteria
UL = Upper limit of method QC acceptance criteria
* Action is applied to positive detects less than 2x the true value of the QL Check Standard.
** Professional judgment may be used to estimate or reject positive detects less than 2x the true value taking into account project DQOs. Professional judgment should be used to accept or estimate positive detects greater than or equal to 2x the true value of the Check Standard but less than the next highest concentration QC sample or standard.

Table INORG-IV-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON POSITIVE BLANK RESULTS

Positive Blank Result	Sample Result		Action
≥ MDL	> Blank Action Level and	≥ QL	A
		< QL	No further action (report estimated sample result)
	≤ Blank Action Level and	≥ QL	U - Raise the QL to the sample result and report as a non-detect
		< QL	U - Report the QL
	Non-detect (U)		A

Blank Action Level = 5x the highest blank concentration associated with the sample
QL = Sample Quantitation Limit

Note: Aqueous equipment (rinsate) and bottle blank results are not used to determine blank action levels for non-aqueous samples. Analytes present in both the non-aqueous sample and the associated aqueous equipment or bottle blanks should be flagged EB or BB, respectively.

Table INORG-IV-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON NEGATIVE BLANK RESULTS

Negative Blank Result	Sample Result		Action
≤ (-MDL)	> Negative Blank Action Level and	≥ QL	A
		< QL	No further action (Report estimated sample result)
	≤ Negative Blank Action Level and	≥ QL	J - Estimate the sample result
		< QL	No further action (report estimated sample result)
	Non-detect (U)		UJ - Estimate the QL

Negative Blank Action Level = absolute value of 5x the lowest negative blank value associated with the sample
QL = Sample Quantitation Limit

Table INORG-V-1:

QUALIFICATION OF ICP-AES ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSAB RECOVERIES

Sample Results	ICSAB Recoveries				
	%R < 50%	50% ≤ %R < 80%	80% ≤ %R ≤ 120%	120% < R ≤ 150%	%R > 150%
Detects	R	J	A	J	R*
Non-detects	R	UJ	A	A	A

* Professional judgment may be used to estimate (J) positive detects, taking into consideration project DQOs.
Note: Generally, action is applied when interferents are present in samples at greater than 50% of their respective levels in the ICS.

Table INORG-V-2:

QUALIFICATION OF ICP-AES ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSA RESULTS

Sample Results	ICSA Concentration or % Recovery*		
	%R < 80% or Conc. < TV-(2xMDL)	%R = 80-120% or Conc. = TV±(2xMDL)	%R > 120% or Conc. > TV+(2xMDL)
Detects	J	A	J
Non-detects	UJ	A	A

TV = ICSA True Value

* ICSA criteria are based on either 80-120% of the ICSA true value or the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

Note: Generally, action is applied when interferents are present in samples at greater than 50% of their respective levels in the ICS. Generally, no action is taken when the estimated interference is less than 10% of the sample result of the affected analyte. Professional judgment may be used to reject the data if the estimated interference comprises greater than 90% of the sample result. If ICSAB recovery criteria are met, then professional judgment may be used to qualify only those sample results in the affected concentration range.

Table INORG-VI-1:

QUALIFICATION OF ICP-MS ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSAB RECOVERIES

Sample Results	ICSAB Recoveries				
	%R < 50%	50% ≤ %R < 80%	80% ≤ %R ≤ 120%	120% < %R ≤ 150%	%R > 150%
Detects	R	J	A	J	R*
Non-detects	R	UJ	A	A	A

* Professional judgment should be used to estimate (J) positive detects, taking into consideration project DQOs.

Table INORG-VI-2:

QUALIFICATION OF ICP-MS ANALYTES BASED ON INTERFERENCE CHECK SAMPLE ICSA RESULTS

Sample Results	ICSA Concentration or % Recovery*		
	%R < 80% or Conc. < TV-(2xMDL)	%R = 80-120% or Conc. = TV±(2xMDL)	%R > 120% or Conc. > TV+(2xMDL)
Detects	J	A	J
Non-detects	UJ	A	A

TV = ICSA True Value

* ICSA criteria are based on either 80-120% of the ICSA true value or the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

Note: Professional judgment may be used to reject (R) positive detects and/or non-detects for the affected analytes.

Table INORG-VII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON INTERNAL STANDARD RELATIVE INTENSITIES

Sample Results	Internal Standard Relative Intensities (RI)*		
	% RI < LL	LL ≤ % RI ≤ UL	% RI > UL
Detects	J	A	J
Non-detects	UJ	A	UJ

LL = Lower Limit of method QC acceptance criteria

UL = Upper Limit of method QC acceptance criteria

* Professional judgment may be used to reject data for severe loss of sensitivity.

Table INORG-VIII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON MATRIX SPIKE RECOVERIES*

Sample Results	% Recovery			
	%R < 30%	30% ≤ %R < LL	LL ≤ %R ≤ UL	%R > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL = Lower Limit of method QC acceptance criteria
UL = Upper Limit of method QC acceptance criteria
* Qualification is applied to the affected analyte in all samples of the same matrix analyzed by the same method; however, the validator may use professional judgment to apply actions to all positive detects and non-detects if the majority of spike analyte recoveries are outside method QC acceptance criteria.

Table INORG-IX-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES*

Sample Results	Laboratory Duplicate Sample Results	
	RPD or Abs. Diff. ≤ QC Limit	RPD or Abs. Diff. > QC Limit
Detects	A	J
Non-detects	A	UJ

* If QC acceptance criteria for the specific matrix are specified in the method, then use the method QC criteria in this table. If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in Table INORG-IX-2 or INORG-IX-3.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the method QC acceptance criteria.

Table INORG-IX-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES - AQUEOUS MATRICES*

Sample Results	Aqueous Laboratory Duplicate Sample Results			
	Both Sample and Duplicate ≥ 5xQL		One or Both Sample and Duplicate < 5xQL	
	RPD ≤ 20%	RPD > 20%	Abs. Diff. ≤ QL	Abs. Diff. > QL
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

* If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in this table (from Appendix I). If QC acceptance criteria for the specific matrix are specified in the method, then use the criteria in Table INORG-IX-1 above.

QL = Sample Quantitation Limit

- When applying the absolute difference criteria, the sample quantitation limit for the sample (vs. the duplicate sample) is used.
- No action is applied when both sample and duplicate values are detected at < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the QC acceptance criteria.

Table INORG-IX-3:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES - NON-AQUEOUS MATRICES*

Sample Results	Non-Aqueous Laboratory Duplicate Sample Results			
	Both Sample and Duplicate ≥ 5xQL		One or Both Sample and Duplicate < 5xQL	
	RPD ≤ 35%	RPD > 35%	Abs. Diff. ≤ 2xQL	Abs. Diff. > 2xQL
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

* If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in this table. If QC acceptance criteria for the specific matrix are specified in the method, then use the criteria in Table INORG-IX-1 above.

QL = Sample Quantitation Limit

- When applying the absolute difference criteria, the sample quantitation limit for the sample (vs. the duplicate sample) is used.
- No action is applied when both sample and duplicate values are detected at < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the QC acceptance criteria.

Table INORG-X-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON FIELD DUPLICATES - AQUEOUS MATRICES

Sample Results	Aqueous Field Duplicate Sample Results			
	Both Duplicates ≥ 5xQL		One or Both Duplicates < 5xQL*	
	RPD ≤ 30%	RPD > 30%	Abs. Diff. ≤ 2xQL	Abs. Diff. > 2xQL
Detects	A	J	A	J
Non-detects	A	UJ	A	J

QL = Sample Quantitation Limit

* No action is taken when both field duplicate results are positive detects < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the field duplicate results are outside QC acceptance criteria.

Table INORG-X-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON FIELD DUPLICATES - NON-AQUEOUS MATRICES

Sample Results	Non-Aqueous Field Duplicate Sample Results			
	Both Duplicates ≥ 5xQL		One or Both Duplicates < 5xQL*	
	RPD ≤ 50%	RPD > 50%	Abs. Diff. ≤ 4xQL	Abs. Diff. > 4xQL
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

QL = Sample Quantitation Limit

* No action is taken when both field duplicate results are positive detects < QL or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the field duplicate results are outside QC acceptance criteria.

Table INORG-XI-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON SERIAL DILUTION PERCENT DIFFERENCES

Sample Results	% D ≤ QC Limit	% D > QC Limit	
		Serial Dilution Sample Result > Undiluted Sample Result	Serial Dilution Sample Result < Undiluted Sample Result
Detects	A	J	J
Non-detects	A	UJ	A*

Note: Qualification is applied to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. However, the validator may use professional judgment to qualify all positive detects and non-detects if the majority of the serial dilution analyte percent differences are outside the method QC acceptance criteria.

* Professional judgment may be used to estimate (UJ) non-detects if the direction of the bias cannot be determined.

Table INORG-XII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LFB RECOVERIES

Sample Results	% Recovery			
	% R < 40%	40% ≤ % R < LL	LL ≤ % R ≤ UL	% R > UL
Detects*	J (< 2x TV)**	J (< 2x TV)	A	J (< 2x TV)**
Non-detects	R	UJ	A	A

LFB = Laboratory fortified blank spiked with target analytes at their quantitation limits.
LL = Lower Limit of method QC acceptance criteria.
UL = Upper Limit of method QC acceptance criteria.
* Action is applied to positive detects less than 2x the LFB true value.
** Professional judgment may be used to reject positive results less than 2x the true value taking into account project DQOs. Professional judgment may be used to estimate positive detects greater than or equal to 2x the LFB true value but less than the value of the next highest concentration QC sample.

Table INORG-XIII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LCS RESULTS*

Sample Results	Aqueous LCS % Recovery				
	%R < 50%	50% ≤ %R < LL	LL ≤ %R ≤ UL	UL < %R ≤ 150%	%R > 150%
Detects	R	J	A	J	R
Non-detects	R	UJ	A	A	A

Sample Results	Non-aqueous LCS Result		
	Result < LL	LL ≤ Result ≤ UL	Result > UL
Detects	J	A	J
Non-detects	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

* If more than half of the LCS analyte recoveries for a particular method fall within one of the above categories, then professional judgment may be used to apply the action to all analytes in all samples associated with that LCS. Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table INORG-XIII-2:

QUALIFICATION OF INORGANIC ANALYTES BASED ON PES RESULTS*

Sample Results	• Single Blind • Double Blind PES < Lower Limit "Action Low" or "Analyte Missed"	• Single Blind • Double Blind PES "Within Warning Limits" "Warning High/Warning Low"	• Single Blind • Double Blind PES > Upper Limit "Action High"
Detects	J	A	J
Non-Detects	R	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

* If more than half of the PES analytes fall within one of the above categories, then professional judgment may be used to apply the action to all analytes in all samples associated with that PES. Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

Table INORG-XIV-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON SAMPLE PERCENT SOLIDS*

Sample Result	% Solids > 30%	10% ≤ % Solids ≤ 30%	% Solids < 10%
Detects	A	J	R
Non-detects	A	R	R

* Professional judgment should be used to accept, qualify or reject the associated sample data when sampling and/or analytical preparation steps were employed to address high moisture soil/sediment samples.

Appendix K

Inorganic Data Validation Worksheets

Note: Refer to Attachment J of Part I, Data Validation Manual: The Data Quality System, for the generic forms for the Data Validation Report (DQO Summary Form, Communication Form, Chain-of-Custody Form/Traffic Report).

REGION I, EPA-NE INORGANIC REGIONAL DATA ASSESSMENT (IRDA)*

CASE #:_____ SITE NAME:_____

LAB NAME:_____ # OF SAMPLES/MATRIX:_____

SDG #:_____ VALIDATION CONTRACTOR:_____

SOW #/CONTRACT #:_____ VALIDATOR'S NAME:_____

EPA-NE DV TIER LEVEL:_____ DATE DP REC'D BY EPA-NE:_____

PO: **ACTION_____ FYI_____ DV COMPLETION DATE:_____

ANALYTICAL DATA QUALITY SUMMARY

		ICP-AES	ICP- MS	HG	CN
I.	Preservation and Technical Holding Times				
II.	ICP-MS Tune				
III.	Calibrations				
IV.	Blanks				
V.	ICP-AES Interference Check Sample (ICS)				
VI.	ICP-MS Interference Check Sample (ICS)				
VII.	ICP-MS Internal Standards				
VIII.	Matrix Spikes				
IX.	Laboratory Duplicate Samples				
X.	Field Duplicates				
XI.	ICP Serial Dilutions				
XII.	Sensitivity Check				
XIII.	Performance Evaluation Samples/Accuracy Check				
XIV.	Analyte Quantitation and Reported Quantitation Limits				
XV.	System Performance				
XVI.	Overall Evaluation of Data				

o = Data had no problems or were qualified due to minor contractual problems.
m = Data were qualified due to major contractual problems.
z = Data were rejected as unusable due to major contractual problems.

ACTION ITEMS: (z items) _____

AREAS OF CONCERN: (m items) _____

COMMENTS: _____

* This form assesses the analytical data quality in terms of contractual compliance only. It does not assess sampling errors and/or non-contractual analytical issues that affect data quality.

** Check "ACTION" only if contractual defects resulted in reduced payment/data rejection recommendations.

Validator:_____ Date:_____

INSTRUCTIONS ON REVERSE SIDE

DRAFT 11/08

GUIDANCE FOR COMPLETING THE IRDA

The IRDA form provides the laboratory's CLP PO and other contract management personnel with an overview of the contractual analysis and reporting deficiencies found in an analytical data package and identifies those contractual deficiencies that resulted in reduced payment/data rejection recommendations/actions. The IRDA form is used to summarize analytical data quality only in terms of contractual compliance. Sampling errors and non-contractual analytical errors that affect data quality are not summarized on this form, but rather are documented in the Tier I Validation Cover Letter and Tier II/III Data Validation Reports. For instance, if the sampler did not ship the samples until after the holding time had expired, a notation would not be made on the IRDA form since the laboratory is not responsible for the sampler's actions.

The IRDA form should be completed as follows:

1. Fill in all the header information (with the exception of the PO Action/FYI field): Case Number, Site Name, Laboratory Name, number and matrix of samples in the data package, SDG number, Validation Contractor, SOW#/Contract#, Data Validator's Name, EPA-NE Data Validation Tier Level (i.e., I, II, III or partial II/III), Date the Data Package was received by EPA-NE, and the Data Validation Completion Date.

2. Summarize the contractual problems discovered during data validation by fraction and by evaluation criteria in the "Analytical Data Summary" table, and in the "Action Items" and "Areas of Concern" sections as described in items 3 through 6 below. Use the Data Validation Memoranda as a guide when completing the IRDA form.

3. The following qualifiers must be utilized to document contractual problems on the IRDA forms.

o	=	Data had no problems or were qualified due to minor contractual problems
m	=	Data were qualified due to major contractual problems
z	=	Data were rejected as unusable due to major contractual problems

4. If the data were acceptable, or were qualified due to minor contractual problems, enter the qualifier "o" into the appropriate column (fraction) and row (evaluation criteria). No further documentation is necessary on the IRDA form. An example of a minor problem would be an inorganic analyte that slightly exceeded the SOW-specified %R continuing calibration criterion.

5. If the data were qualified due to major contractual problems, enter the qualifier "m" into the appropriate column (fraction) and row (evaluation criteria). Use different superscripts (m^1, m^2, etc.) for each major contractual problem identified and provide a brief description of each major problem in the "Areas of Concern" section. An example of a major contractual problem resulting in data qualification would be an ICP-MS internal standard that had an extremely low % relative intensity (below the lower limit of the SOW-specified acceptance criteria) and reanalysis was not performed.

6. If the data were rejected as unusable due to major contractual problems, enter the qualifier "z" in the appropriate column (method/parameter) and row (evaluation criteria). Use a different superscript (z^1, z^2, etc.) for each major contractual problem identified and provide a brief description of each major problem in the "Action Items" section. An example of a major contractual problem resulting in data rejection would be contractual holding time criteria that were exceeded for cyanide.

7. Complete the PO Action/FYI field using the information contained in the "Action Items" and "Areas of Concern" sections. PO Action should be indicated with a check mark (√) in the space following "Action" only if the contractual defects resulted in reduced payment or data rejection. If no PO Actions are indicated, then a check mark (√) should be placed in the space following "FYI".

8. The validator who completed the IRDA form must sign his/her name in the "Validator" field and enter the IRDA completion date in the "Date" field.

Site Name _____
Reference No. _____

REGION I INORGANIC DATA VALIDATION

The following data package has been validated:

Lab Name _____
Case/Project No. _____
SDG No. _____
No. of Samples/Matrix _____

SOW/Method No. _____
Sampling Date(s) _____
Shipping Date(s) _____
Date Rec'd by lab _____

Traffic Report Sample Nos. _____
_____
_____
_____
Equipment Blank No. _____
Bottle Blank No. _____
Field Duplicate Nos. _____
PES Nos. _____

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, revision _____ was used to evaluate the data and/or approved modifications to the EPA-NE Functional Guidelines were used to evaluate the data and are attached to this cover page: (attach modified criteria from EPA approved QAPP or amendment to QAPP).

A Tier II or Tier III evaluation was used to validate the data (circle one). If a Tier II validation with a partial Tier III was used, then identify samples, parameters, etc., that received partial Tier III validation.
_____
_____

The data were evaluated based upon the following parameters:

- Overall Evaluation of Data
- Data Completeness (CSF Audit - Tier I)
- Preservation and Technical Holding Times
- ICP-MS Tune
- Calibrations
- Blanks
- ICP-AES Interference Check Sample (ICS)
- ICP-MS Interference Check Sample (ICS)
- ICP-MS Internal Standards
- Matrix Spikes
- Laboratory Duplicate Samples
- Field Duplicates
- ICP Serial Dilutions
- Sensitivity Check
- Performance Evaluation Samples/Accuracy Check
- Analyte Quantitation and Reported Quantitation Limits
- System Performance

Region I Definitions and Qualifiers:

A - Acceptable Data
J - Numerical value associated with analyte is an estimated quantity.
R - The data are rejected as unusable. The R replaces the numerical value or sample quantitation limit.
U - Analyte not detected at that numerical sample quantitation limit.
UJ - The sample quantitation limit is an estimated quantity.
BB, EB - Analyte detected in aqueous bottle blank or aqueous equipment blank associated with soil/sediment samples.

Validator's Name _____ Company Name _____ Phone Number _____

Date Validation Started _____ Date Validation Completed _____

Check if all criteria are met and no hard copy worksheet is provided. Indicate NA if worksheet is not applicable to the analytical method. Note: there is no standard worksheet for System Performance; however, the validator must document all system performance issues in the Data Validation Memorandum.

INORG Worksheets:

INORG	COMPLETE SDG FILE (CSF) AUDIT	_____
INORG-I	PRESERVATION AND TECHNICAL HOLDING TIMES	_____
INORG-II	ICP-MS TUNE	_____
INORG-III-A/B	CALIBRATIONS	_____
INORG-IV-A/B	BLANKS	_____
INORG-IV-C.1	BLANKS	_____
INORG-IV-C.2	BLANKS	_____
INORG-V-A	ICP-AES INTERFERENCE CHECK SAMPLE - ICSAB	_____
INORG-V-B.1	ICP-AES INTERFERENCE CHECK SAMPLE - ICSA	_____
INORG-V-B.2	ICP-AES INTERFERENCE CHECK SAMPLE - ICSA	_____
INORG-VI-A	ICP-MS INTERFERENCE CHECK SAMPLE - ICSAB	_____
INORG-VI-B	ICP-MS INTERFERENCE CHECK SAMPLE - ICSA	_____
INORG-VII	ICP-MS INTERNAL STANDARDS	_____
INORG-VIII	MATRIX SPIKES	_____
INORG-IX	LABORATORY DUPLICATE SAMPLES	_____
INORG-X	FIELD DUPLICATES	_____
INORG-XI	ICP SERIAL DILUTIONS	_____
INORG-XII-A/B	SENSITIVITY CHECK	_____
INORG-XIII-A	PE SAMPLES/ACCURACY CHECK- LCS	_____
INORG-XIII-B	PE SAMPLES/ACCURACY CHECK- PE RESULTS	_____
INORG-XIV	ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS	_____
TABLE II-WORKSHEET	OVERALL EVALUATION OF DATA	_____

I certify that all criteria were met for the worksheets checked above.

Signature: _____ Name: _____

Date: _____

The data validator generates a Data Validation Report, applicable to Data Validation Tiers II and III, that consists of the following components in the order specified below: (Refer to Part I - Data Validation Manual, Section 11 for a description of each of the Data Validation Report components).

1. Organic Regional Data Assessment/Inorganic Regional Data Assessment (ORDA/IRDA) Form
2. Data Validation Memorandum
   a. Narrative
   b. Table I-Qualifier Recommendation Summary Table
   c. Table II-Overall Evaluation of Data
   d. Data Summary Tables
3. Standard Data Validation Worksheets
   a. Manual
   b. Automated Data Review Reports
4. Support Documentation
   a. Copy of non-CLP analytical method, e.g., DAS methods, modified EPA methods
   b. Copies of PES Score Reports/Vendor PES QC Acceptance Limits
   c. Copies of Telephone Logs/Communication Forms for:
      - RSCC communications
      - Requests for laboratory data resubmissions/clarifications
      - Communications with samplers resolving sampling problems
      - Communications with PO/DV Chemist/Lead Chemist to report contractually-deficient data for rejection/reduced payment
      - Communications with EPA Site Manager concerning possible data rejection
      - EPA Site Manager authorization for alternate DV tier
   d. Copies of data supporting recommendations for reduced payment resulting from CSF Audit and/or PE sample result evaluation
   e. Original data to support recommendations for data rejection/non-payment identified from Tier II or Tier III data validation
   f. Copies of field sampling notes and/or field report supplied by field sampler
   g. Copies of EPA-approved amendments to QAPP and/or SAP describing modified criteria to be used for validating site data
5. CSF Completeness Evidence Audit
6. DQO Summary Form

The data validator is responsible for implementing all corrective actions required by the contractor Lead Chemist in response to EPA-NE data validation oversight findings.

EPA-NE - Data Validation Worksheet

Overall Evaluation of Data - Data Validation Memorandum - Table II

Site:_____ Case:_____ SDG:_____

INORGANICS					
DQO (List all DQOs)	Sampling and/or Analytical Method Appropriate? (Yes or No)	Measurement Error		Sampling Variability**	Potential Usability Issues
		Analytical Error	Sampling Error*		

* The evaluation of "sampling error" cannot be completely assessed in data validation.

** Sampling variability is not assessed in data validation.

Validator:_____ Date:_____

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EPA-NE - Data Validation Worksheet
INORG

COMPLETE SDG FILE (CSF) AUDIT

Inorganic Parameters:_____

Missing Information	Date Lab Contacted	Date Received

Validator: _____ Date: _____

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EPA-NE - Data Validation Worksheet
INORG-I

I. PRESERVATION AND HOLDING TIMES

Circle sample numbers with exceeded technical holding times or omitted preservation.
Circle all exceeded technical holding times.

Sample No.	Matrix	Pres. Code		Date Sampled	Metals		Hg		CN		Action
		Metals	CN		Date Analyzed	# of Days from Samp. to Analysis	Date Analyzed	# of Days from Samp. to Analysis	Date Analyzed	# of Days from Samp. to Analysis	

Preservation Code:
1. Cool (≤ 6°C)
2. pH < 2 with HNO_3
3. pH > 12 with NaOH
4. Room Temperature
5. Freeze
6. Reducing agent (for oxidants)
7. Treated for sulfides
8. Other - _____

Action Code:
J - Estimate (J) Detected Values
UJ - Estimate (UJ) Non-Detected Values
R - Reject (R) Non-Detected Values

Sampler:_____ Company:_____ Contacted: Y N Date:_____

Validator:_____ Date:_____

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II. ICP-MS TUNE

List all Tunes that are outside method QC acceptance criteria.

Method:_____

Method QC acceptance criteria for mass calibration:_____

Method QC acceptance criteria for resolution check/peak width:_____ at_____% peak height.

Method QC acceptance criteria for % RSD:_____

Analysis Date and Time	Analyte	Mass Calibration			Mass Resolution		% RSD of Absolute Signals	Analyte/Samples Affected	Action
		True Mass (amu)	Measured Mass (amu)	Absolute Difference of Masses (amu)	Peak Width (amu)	% Peak Height			
Comments:									

Validator:_____ Date:_____

III. CALIBRATIONS

A. Instrument Calibration - List all calibration correlation coefficients that are outside the method QC acceptance criteria.
Calibration correlation QC acceptance criteria: _____ Calibration Type: _____

Date/Time	Instr.	Analyte	Corr. Coef.	Samples Affected	Action

B. Initial and Continuing Calibration Verifications - List all ICV and CCV analyte recoveries that are outside the method QC acceptance criteria.
ICV method QC acceptance criteria: _____ CCV method QC acceptance criteria: _____

Date	Instr.	Analyte	ICV/CCV #	% R	Samples Affected	Action

C. Quantitation Limit Check Standard - List all QL Check Standard analytes that are outside method QC acceptance criteria.
QL Check Standard method QC acceptance criteria: _____

Date	Instr.	Analyte	QL Check Std. #	% R	Affected Range	Samples Affected	Action

Comments: _____

Validator: _____ Date: _____

EPA-NE - Data Validation Worksheet
INORG-IV-A/B

IV. BLANKS - List the blank contamination and negative blank results below.

Sampler: _____ Company: _____ Contacted: Y N Date: _____

A. Laboratory: Preparation (Method) and Calibration (Instrument) Blanks

Date Prepared	Date Analyzed	Blank Type (ICB/CCB#/Prep Blank)	Matrix	Instrument	Analyte	Conc. (units)

B. Field: Equipment (Rinsate) and Bottle Blanks

Date Sampled	Date Analyzed	Sample No. (Blank Type)	Matrix	Instrument	Analyte	Conc. (units)

Were the proper number of blanks analyzed at the proper frequency? Y N

For ICP MS - Are internal standard responses in all blanks within method QC acceptance criteria? Y N

Comments: _____

_____

Validator: _____ Date: _____

IV. BLANKS

C.1 Blank Contamination Worksheet

Circle the highest concentration of each contaminant.

Analyte	Date Analyzed	ICB	CCB							PBW	PBS	EB	BB	Max. Conc.	Action Level
			1	2	3	4	5	6	7						
Aluminum															
Antimony															
Arsenic															
Barium															
Beryllium															
Cadmium															
Calcium															
Chromium															
Cobalt															
Copper															
Iron															
Lead															
Magnesium															
Manganese															
Mercury															
Nickel															
Potassium															
Selenium															
Silver															
Sodium															
Thallium															
Vanadium															
Zinc															
Cyanide															

Validator: _____

Date: _____

IV. BLANKS

C.2 Blank Actions - List the maximum concentrations of each analyte among all blanks associated with each sample.

Analyte	Type of Blank	Date Blank Originated	Max. Conc. (units)	Blank Action Level (units)	Sample QL	Samples Affected	Action

Comments:

Validator: _____

Date: _____

V. A. ICP-AES INTERFERENCE CHECK SAMPLE - ICSAB

1. List all analytes in the ICSAB that are outside ICSAB percent recovery criteria.

Date	Analyte	%R	Conc. of Interferents Observed in ICSAB (ug/L)				Samples Affected	Action
			Al	Ca	Fe	Mg		

Comments:

Validator:_____ Date:_____

V. B.1 ICP-AES INTERFERENCE CHECK SAMPLE - ICSA

1. List all analytes in the ICSA that are outside ICSA criteria.

Date	Analyte	MDL (ug/L)	ICSA Observed Conc. (ug/L)	ICSA True Value (ug/L)	Criteria: 80-120% R or TV±2xMDL	Conc. of Interferents Observed in ICSA (ug/L)				Associated Samples
						Al	Ca	Fe	Mg	

Comments:

Validator:_____ Date:_____

V. B.2 ICP-AES INTERFERENCE CHECK SAMPLE - ICSA

2. List all affected sample results.

Affected Sample	Interferent	Conc. in Sample (ug/L)	Analyte	Conc. in Sample (ug/L)	Estimated Interference in Sample (ug/L)	Action
Comments:						

Validator:_____ Date:_____

VI. A. ICP-MS INTERFERENCE CHECK SAMPLE - ICSAB

List all analytes that are outside ICSAB criteria of 80-120% R.

Date	Analyte	%R	Samples Affected	Action

Comments:

Validator:_____ Date:_____

VI. B. ICP-MS INTERFERENCE CHECK SAMPLE - ICSA

List all analytes that are outside ICSA criteria.

Date	Analyte	MDL (ug/L)	ICSA Observed Conc. (ug/L)	ICSA True Value (ug/L)	Criteria: 80-120% R or TV ± 2xMDL	Samples Affected	Action

Comments:

Validator:_____

Date:_____

VII. ICP-MS INTERNAL STANDARDS

List all internal standards that are outside method QC acceptance criteria.

IS method QC acceptance criteria: _____

Sample Number	Date and Time Analyzed	IS, amu	% RI	Analytes Affected (amu)	Action
Comments:					

Validator:_____ Date:_____

EPA-NE - Data Validation Worksheet
INORG-VIII

VIII. MATRIX SPIKES - List all matrix spike analytes that are outside method QC acceptance criteria.

Use a separate worksheet for each matrix spike sample.

Sample No.: _____ Matrix: _____

Method	Analyte	Spiked Sample Result	Sample Result	Amount of Spike Added	MS % Recovery	Method QC Limits % Recovery	Post-Digest Spike % Recovery	Action

Comments:

Validator:_____ Date:_____

IX. LABORATORY DUPLICATE SAMPLES - List all analytes that are outside method QC acceptance criteria for the specific matrix.

Use a separate worksheet for each laboratory duplicate sample.

Sample No.:_____ Duplicate Sample No.:_____ Matrix:_____

Laboratory duplicate sample method QC acceptance criteria:_____

Method	Analyte	Sample Conc.	Sample		Duplicate Conc.	Duplicate		RPD or Abs. Diff.	QC Acceptance Criteria (RPD or Abs. Diff.)	Action
			SQL	5xSQL		SQL	5xSQL			

Do the field duplicate sample data indicate acceptable field precision? Y N

Comments:_____

Validator:_____ Date:_____

X. FIELD DUPLICATES - List all field duplicate analytes that are outside criteria.

Use a separate worksheet for each field duplicate pair.

Sample No.:_____ Duplicate Sample No.:_____ Matrix:_____

Method	Analyte	Sample Conc.	Sample		Duplicate Conc.	Duplicate		RPD or Abs. Diff.	QC Acceptance Criteria (RPD or Abs. Diff.)	Action
			SQL	5xSQL		SQL	5xSQL			

Do the laboratory duplicate sample data indicate acceptable laboratory precision? Y N

Comments: _____

Sampler Name: _____ Contractor Name: _____ Date Contacted: _____

Reason for contact and resolution obtained: _____

Validator: _____ Date: _____

XI. ICP SERIAL DILUTIONS

Use a separate worksheet for each serial dilution sample.

Sample No.:_____ Matrix:_____ Method:_____

List all serial dilution analytes that are outside method QC acceptance criteria.

% Difference method QC acceptance criteria: _____
Minimum concentration required to apply the % D criteria (e.g., 50x MDL): _____

Analyte	MDL	Min. Conc. Required	Sample Result	Serial Dilution Sample Result (corrected for dilution)	% D	Action

Comments:

Validator:_____ Date:_____

XII. SENSITIVITY CHECK

A. Method Detection Limit Study - List all analytes that are outside the MDL criteria.

- Has an appropriate MDL study been submitted with seven replicates for each analyte and matrix of interest? **Y N**
- Were samples analyzed within one year of the MDL study and on the same instrument? **Y N**

Matrix	Analyte	MDL	MDL > QL	Samples Affected	Action

If an MDL study has not been submitted, use only the LFB results to evaluate data.

B. Laboratory Fortified Blank - List all LFB analytes that are outside the LFB criteria.

- Has an appropriate and complete LFB been submitted at the proper frequency? **Y N**
- Does the LFB contain all target analytes at their QLs? **Y N**

Matrix	Method	Analyte	% R	Method QC Limits % R	Samples Affected	Action

Comments:_____

Validator:_____ Date:_____

EPA-NE - Data Validation Worksheet
INORG-XIII-A

XIII. PERFORMANCE EVALUATION SAMPLES/ACCURACY CHECK

A. Laboratory Control Samples - List all analytes that are outside criteria.

SDG No.:_____ Case:_____

Are more than one-half of the LCS analytes within criteria for each parameter and method? **Y** **N**

Date Prepared	Date Analyzed	Parameter/ Method	Matrix	Analyte	% Recovery (or Observed Conc.)	Method QC Acceptance Criteria	Samples Affected	Action

Comments:

Validator:_____ Date:_____

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XIII. PERFORMANCE EVALUATION SAMPLES/ACCURACY CHECK

B. Performance Evaluation Results - List all analytes that are outside criteria.

SDG No.:_____ Case:_____

Indicate the source of the PES: Region 1 EPA PES Non-EPA PES

Are more than one-half of the PES analytes within criteria for each parameter and method? **Y** **N**

PE Sample Number	Ampule Number	Parameter/ Method	Type of PES	Matrix	Analyte	Conc.	PES Score*	Samples Affected	Action

* For Region I PESs indicate the Region I PES Score Report Result: Action High, Action Low, Analyte Missed, Contaminant.
For Non-EPA PESs indicate the Non-EPA PES Score: PES Analyte Missed; PES Analyte Contaminant; PES Analyte Hit (% Recovery Limits).

Comments:_____

Validator:_____ Date:_____

XIV. ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS

Recalculate, from the raw data, the concentrations for one positive detect and one reported sample quantitation limit for a non-detect in a diluted sample or soil sample per analytical method. (Note: Although Section XIV, C.2.a, requires that one calculation for each method in each sample be performed, the validator is only required to reproduce an example, for each method, of one positive detect and one sample quantitation limit calculation on this worksheet.)

Do all soil/sediment samples have % solids greater than 30%? **Y** **N**

- If no, were any steps employed to address the high moisture content? _____
- Indicate the action and list the affected sample nos.: _____

Method		Calculation
ICP-AES		
Sample No.:		
Reported Analyte:		
Reported Value:		
Non-Detected Analyte:		
Reported Quantitation Limit:		
ICP-MS		
Sample No.:		
Reported Analyte:		
Reported Value:		
Non-Detected Analyte:		
Reported Quantitation Limit:		
Mercury		
Sample No.:		
Reported Value:		
Sample No.:		
Reported Quantitation Limit:		
Cyanide		
Sample No.:		
Reported Value:		
Sample No.:		
Reported Quantitation Limit:		

Validator: _____ Date: _____