Department of Energy
Carlsbad Area Office
P. O. Box 3090
Carlsbad, New Mexico 88221

March 20, 1997

MAR 1997
RECEIVED

Mr. Benito J. Garcia, Chief
Hazardous and Radioactive Materials Bureau
New Mexico Environment Department
2044 A Galisteo St.
Santa Fe, New Mexico 87505

Subject: Groundwater Monitoring Program Plan - Response to February 25, 1997 Request

Dear Mr. Garcia:

Please find enclosed a revised Appendix D18 entitled "Groundwater Monitoring Program Plan." This package is designed to replace the material currently found in the RCRA Part B permit application.

This submittal is being provided in response to your February 25, 1997, letter received by this office on March 6, 1997. Additionally, we are providing both a cross reference summary table which identifies the location where we previously described the monitoring efforts or added additional material to your request, along with responses to the specific requests outlined in your letter.

A hard and electronic copy is being provided to your technical contractor at the request of Mr. Steve Zappe. If we can be of any further assistance or provide any additional information, please contact Craig Snider at (505) 234-7452.

Sincerely,

For George E. Dials
Manager

Enclosure

cc:
C. Walker, AT Kearney

Printed on recycled paper

970310

cc: w/o encl.
C&C File
K. Day, WID
L. Kilgore, CAO
L. King, CAO

Response to Comments and Information Requests by the NMED on Appendix D18, the WIPP Groundwater Monitoring Program

The NMED submitted a letter to the DOE on February 25, 1997 outlining the additional information that they require in the new Groundwater Monitoring Program Plan (Appendix D18 in the RCRA Part B Permit Application). Old Appendix D18 has been completely rewritten, by extracting existing information from various sections of the application, adding new data, and rewriting some of the existing text. The new monitoring plan addresses all of the specific information requirements of a complete detailed groundwater monitoring plan. The new plan was also written to respond to each of the information requests contained in the above-mentioned letter. The following discussion summarizes each NMED comment (bullet list) and the area of the monitoring plan where the comment is addressed.

- **Specify explicitly the proposed monitoring network.**

  Response: Chapter D18-3d describes the monitor well locations and clearly discusses that the GMP will use Wells WQSP-1 through -6A for background and detection monitoring. Figure D18-9 shows the monitor well network location and the WIPP compliance point which is the WIPP site boundary.

- **Identify the point of compliance for groundwater monitoring.**

  Response: Chapter D18-3d also clearly describes the WIPP monitoring point of compliance. The point of compliance which is the WIPP site boundary, is shown on Figures D18-2 and D18-9.

- **Provide a complete discussion of the appropriateness of the monitor well locations and suitability of the monitored interval to detect releases of hazardous constituents. In particular, address what appears to be a detection gap between Wells WQSP-3 and WQSP-4.**

  Response: Chapter D18-3d addresses the technical information and the previous investigations at WIPP which led to the selection of the monitor well locations. The general rationale for monitoring in the Culebra is also discussed in this chapter. D18-3d also describes how pumping and other activities at WIPP have impacted potentiometric surfaces and groundwater flow directions over the short term, and that using nondensity corrected flow maps gives inaccurate and misleading interpretations and should not be used to judge the adequacy of well placement. Figure D18-9 shows the most recent Culebra freshwater head potentiometric surface and the proposed monitor well network.

  Furthermore, even if one assumes the highest transmissivities occur throughout the region, travel time to the compliance point are on the order of 50 to 100 years. Consequently, any downgradient monitoring well located midway between the release point and the compliance point would detect a release prior to it reaching the compliance point.

- **Provide geologic/hydrologic information (or reference other sections of the permit application) to support the well locations and monitoring intervals.**

  Response: New Appendix D18, Groundwater Monitoring Plan, has considerable added information summarizing the WIPP area geology and hydrology. Chapters D18-1, D18-3, and D18-6 all have discussion of the physical characteristics of the site. Numerous references to Chapters D and E and Appendices D6 and E1 of the existing application have been added to direct the reader to greater detail on hydrology and geology. In addition, reference to a recent Sandia report on model simulations of the groundwater basin have been added as a source of additional information.

- **Provide well construction documentation, including installation diagrams, installation methodologies, construction techniques, well development, core logs, soil sampling, and justification from any deviations from typical well construction standards as defined by the RCRA Groundwater Monitoring Technical Enforcement Guidance Document and the RCRA Groundwater Monitoring: Draft Technical Guidance.**

  Response: Chapter D18-3d(1) provides all details of the drilling, coring, and well installation for Wells WQSP-1 through -6A. These descriptions include depths drilled, casing and well screen configurations, core intervals, materials used, and sand, gravel, bentonite sealing, and well annulus cementing. Figures D18-10 through 16 illustrate "as builts" and construction details of each well proposed for the monitoring program.

- **Identify the proposed monitoring frequency for the detection monitoring program.**

  Response: Chapter D18-6b describes the proposed monitoring frequency and Table D18-1 summarizes both sampling and water-level-monitoring frequencies for the background and detection monitoring programs. These frequencies are water-level monitoring, conducted monthly, and sampling, conducted semiannually, for both background and detection monitoring, respectively.

- **Include detailed sampling procedures, types of containers used for sample collection, sample collection methodologies, sample preservation requirements, volumes required for analyses, chain of custody programs, etc.**

  Response: Sampling procedures are discussed in Section D18-6(d) and in Attachment A. Types of containers used are discussed in Section D18-6(d) and in Attachment B (specified in the contract laboratory's SOPs). Sample preservation requirements are found in Attachment B. Volumes required for analyses are found in Attachment B. Chain of custody protocols are discussed in Section D18-7, Sample Documentation and Custody.

- **Describe equipment decontamination, management of investigation-derived waste, and surveying procedures.**

  Response: Surveying procedures for the existing monitor wells are addressed in Section D18-6(a), pg. D18-20. Decontamination procedures are discussed in Section D18-6d(2),

pg. D18-23. Management of investigation-derived waste is discussed in Section D18-6d(2), pg. D18-23.

- **Include discussions of quality assurance/quality control for sample collection, such as frequency of duplicate, trip blank, and other sample collections.**

  Response: QA/QC protocols are discussed in Section D18-5. Sample collection frequency is discussed in Section D18-6(b). WIPP sample collection procedures are included as Attachment A and are described in Section D18-6(d).

- **Provide a complete proposed list of indicator parameters, waste constituents, and reaction products for monitoring, the rationale for their selection, and the analytical methods to be used.**

  Response: Laboratory and field analytical parameters are addressed in Section D18-6(c), and in Table D18-2. The DOE plans to analyze for all Appendix IX compounds for background monitoring. Detection monitoring will target TCLP metals and eight organic compounds shown to constitute the greatest hazard associated with the waste.

- **Identify the contract laboratory performing the sample analyses and include all associated SOPs.**

  Response: The current contract laboratory SOPs are included in Attachment B. The relationship between WIPP and the contract laboratory is described in Section D18-6d(3). This section describes how the contract laboratory is selected, and that the selected laboratory may change over time.

- **Include applicable and necessary quality assurance information within the plan, rather than reference governing documents.**

  Response: QA/QC is addressed in Section D18-5, pgs. D18-14 through D18-19. Specific QA/QC information is also addressed in Attachments A and B, WIPP Operating Procedures, and Contract Laboratory Operating Procedures.

- **Provide procedures to statistically evaluate data.**

  Response: A discussion of statistical analyses is included in Section D18-6e(1) through (4), pgs. D18-25 through D18-27.

- **Include procedures and frequencies for collecting water level measurements.**

  Response: Included in Section D18-8b, pgs. D18-32 through D18-35, Section D18-6b, pgs. D18-20 through D18-21, and in Table D18-1.

- **Provide a table summarizing groundwater quality data obtained to date.**

  Response: Relevant water quality data collected to date for Wells WQSP-1 through -6A are included as Attachment C of revised Appendix D18.

Cross Reference Summary Table

Section of New Appendix D18 Groundwater Monitoring	Where Located Previously in Permit Application; or New Text
D18-1	Chapter E
D18-1a	WIPP Environmental Monitoring Plan
D18-1b	New Text
D18-1c and 1c(1)	Chapter D and Appendix D6
D18-1c(2)	Chapter D and Appendix D6
D18-2	Appendix E1
D18-3	New Text
D18-3a, 3b, 3c, and 3d	New Text, Appendix D18, and Appendix D6
D18-4	New Text
D18-5a and 5b	New Text
D18-5c through 5k	Appendix D18 and New Text
D18-6	New Text (modified from various sources)
D18-6a	Appendix D18
D18-6b	New Text
D18-6c	New Text, Chapter C, and Appendix C6
D18-6d	Appendix D18
D18-6d(1) and (2)	Appendix D18
D18-6d(3)	Appendix D18 and Chapter D
D18-6e(1) and (2)	New Text and Chapter D
D18-6e(3)	Chapter D
D18-6e(4)	New Text
D18-7	New Text following QA Guidance from EPA
D18-8	New Text
D18-8a(1), (2), (3), and (4)	Appendix D18 and New Text
D18-8b(1), (2), (3), (4), (5), and (6)	Appendix D18
D18-8c, 8d, and 8e	Appendix D18
D18-9	Appendix D18 and New Text
D18-10	Appendix D18 and New Text

APPENDIX D18

WIPP GROUNDWATER SURVEILLANCE PROGRAM PLAN AND DETAILS ON THE WQSP WELLS

Effective Date: 3/12/96

WP 02-1
Revision 3

Groundwater Surveillance Program Plan

Cognizant Section: ENVIRONMENTAL MONITORING

Approved By: S. R. Krump

Cognizant Department: ENVORONMENT, SAFETY, & HEALTH

Approved By: Chuan-Fu Wu for KSD

WP 02-1 Rev. 3
GROUNDWATER SURVEILLANCE PROGRAM PLAN

TABLE OF CONTENTS

1.0 INTRODUCTION 1

2.0 REFERENCES 1

3.0 RESPONSIBILITIES 1

4.0 GSP QUALITY ASSURANCE PLAN 3

4.1 Introduction 3

4.1.1 Department of Energy (DOE) Order 5400.1 4
4.1.2 DOE/EH 0173T 4
4.1.3 Resource Conservation and Recovery Act (RCRA) 4
4.1.4 Final Environmental Impact Statement (FEIS) Commitments 4
4.1.5 Future Land Use Decisions 5

4.2 GSP Quality Assurance Requirements 5

4.2.2 Quality Assurance Program 5
4.2.3 Design Control 5
4.2.4 Procurement Document Control 5
4.2.5 Instructions, Procedures, and Drawings 5
4.2.6 Document Control 6
4.2.7 Control of Purchased Material, Equipment and Services 6
4.2.8 Identification and Control of Items 6
4.2.9 Control of Processes 7
4.2.10 Inspection/Surveillance 7
4.2.11 Test Control 7
4.2.12 Control of Monitoring and Data Collection Equipment 8
4.2.13 Handling, Storage, and Shipping 8
4.2.14 Inspection and Acceptance Testing 8
4.2.15 Control of Nonconforming Conditions 8
4.2.16 Corrective Action 9
4.2.17 Quality Assurance Records 9
4.2.18 Assessments 9

5.0 GSP WATER QUALITY SAMPLING PLAN 10

5.1 Scope 10

5.1.1 General 10

5.2 Surveillance Well Construction 12

5.3 Sampling Program Description 13

5.3.1 Serial Sampling 13
5.3.2 Final Samples 14

5.4 Groundwater Pumping and Sampling Systems 14

5.5 Pressure Monitoring Systems 16

5.6 Sample Analysis 17

5.6.1 Serial Samples 18
5.6.2 Final Samples 19

5.7 Sample Preservation, Tracking, Packaging and Transportation 19

5.8 Quality Assurance, Records Management and Document Control 20

5.9 Calibration Requirements 21

6.0 WATER LEVEL MONITORING PLAN 21

6.1 Scope 21
6.2 Introduction 22
6.3 Objective 22
6.4 Field Methods 24
6.5 Records and Document Control 24
6.6 Reporting 25
6.7 Calibration Requirements 26

GROUNDWATER SURVEILLANCE PROGRAM PLAN

1.0 INTRODUCTION

This is the controlling document for the Waste Isolation Pilot Plant (WIPP) Groundwater Surveillance Program (GSP). The GSP is administered as part of the WIPP Environmental Monitoring Program by the Environmental Monitoring (EM) Section of the Environment, Safety and Health (ES&H) Department.

2.0 REFERENCES

DOE Order 5400.1, General Environmental Protection Program

DOE/EH 0173T, Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance

Groundwater Protection Management Program Plan

WP 02-3, Environmental Procedures Manual

WP 10-AD, WIPP Maintenance Administrative Procedures Manual

WP 12-1, Waste Isolation Pilot Plant Safety Manual

WP 12-107, Hazard Communication Program

WP 13-1, WID Quality Assurance Program Description

WP 15-6, Purchasing Policies and Procedures Manual

WP 15-PR, Records Management Plan

3.0 RESPONSIBILITIES

The overall organizational structure of the Westinghouse WID is described in Part I, Section 1 of the Quality Assurance Program Description (QAPD). The GSP is the responsibility of the ES&H Department. The GSP is conducted by the EM Section of this department.

The EM manager assumes responsibility for the overall design and implementation of the GSP including the following areas:

- Development and approval of specific procedures for the conduct of all GSP activities.

- Establishment of minimum qualification criteria and training requirements for all program personnel.
- Review and approval of programmatic reports.
- Oversight of appropriate levels of cooperation and consultation between the EM Section and the state of New Mexico regarding environmental monitoring.
- Preparation of the QA section of the GSP Plan.

The EM manager and staff are responsible for achieving and maintaining quality in the GSP. Job descriptions will be maintained for the EM manager, professional, technical, and administrative staff positions. All GSP data shall be reviewed and approved by the EM manager, or designee, prior to release.

The EM manager appoints a GSP Team Leader (TL), assigning the following responsibilities to the TL:

- Direct GSP per written approved procedures.
- Initiate review of programmatic plans and procedures.
- Review and evaluate sample data.
- Prepare and review programmatic reports.
- Assure that appropriate samples are collected and analyzed.
- Assure that adequate technical support is provided to the Quality and Regulatory Assurance (Q&RA) Department, when required during audits of vendor facilities.

The EM manager designates one or more scientists, engineers, or technicians who will be responsible for the following items:

- Collection and subsequent distribution of samples.
- Preparation and maintenance of appropriate data sheets and sample tracking documentation.
- Monitoring of equipment operability status.
- Reporting of equipment malfunctions.
- Reporting of nonconformance to the TL or EM manager.

- Overseeing of quality control checks of data.
- Conducting field activities in accordance with written procedures.

The Q&RA manager provides independent oversight of the GSP, via the assigned cognizant Q&RA engineer, to verify that quality objectives are defined and achieved. The Q&RA manager ensures objective, independent assessments of GSP quality performance. The Q&RA manager has been delegated authority and given organizational freedom by the WID General Manager to access work areas, identify quality problems, initiate or recommend corrective actions, verify implementation of corrective actions, and ensure that work is controlled or stopped until adequate disposition of an unsatisfactory condition has been implemented.

The EM manager assures that basic qualifications for GSP personnel are carried out in accordance with Section 2 of the QAPD.

The EM manager assures that position descriptions for assigned GSP personnel are adequately prepared. Each position description will include position purpose, principal responsibilities, nature of work, and scope.

The EM manager and/or TL assures that training is performed on an individual basis to maintain an acceptable level of proficiency by all new or temporary GSP staff and by all permanent GSP staff.

New GSP employees are required to review pertinent program documentation, become familiar with applicable procedures, and complete appropriate qualifications prior to undertaking any unsupervised GSP task. To become qualified to perform a specific task or series of tasks, an employee must demonstrate subject knowledge and practical skills and become certified in performing the task(s) by a board-certified subject matter expert (SME). Employees who have not completed the appropriate qualification card will not be allowed to conduct unsupervised GSP activities.

The EM manager, TL, or task SME may determine the need for retraining of GSP personnel. Retraining may be noted by Q&RA during any surveillance or audit or during a periodic review initiated by the EM manager, TL, or SME.

The EM manager assures that documents detailing all staff training are current and properly filed. Copies of training records shall be on file in the WID Technical Training Section.

4.0 GSP QUALITY ASSURANCE PLAN

4.1 Introduction

This section is the quality assurance (QA) plan for the WIPP GSP. The objective of this QA plan is to establish the specific QA requirements associated with the GSP. The GSP currently consists of two activities: the Water Quality Sampling Program (WQSP) and the Water Level Monitoring Program (WLMP). Technical implementation of each specific activity is controlled by an individual program plan and unique operating procedures. The GSP provides a mechanism for addressing the following:

4.1.1 Department of Energy (DOE) Order 5400.1

Chapter 3 of the DOE Order 5400.1, General Environmental Protection Program, states that "... all Department of Energy (DOE) sites will conduct a groundwater protection management program." The order requires each DOE site to provide for the design and implementation of a groundwater monitoring effort that supports resource management and complies with applicable environmental laws and regulations.

4.1.2 DOE/EH 0173T

DOE/EH 0173T, Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance, states that:

> It is the policy of DOE to conduct effluent monitoring and environmental surveillance programs that are adequate to determine whether the public and the environment are adequately protected during DOE operations and whether operations are in compliance with DOE and other applicable Federal, State, and local radiation standards and requirements. It is also DOE policy that Departmental monitoring and surveillance programs be capable of detecting and quantifying unplanned releases and meet high standards of quality and credibility. It is DOE's objective that all DOE operations properly and accurately measure radionuclides in their effluent and in ambient environmental media.

4.1.3 Resource Conservation and Recovery Act (RCRA)

By virtue of a Groundwater Monitoring Waiver, prepared under 40 CFR 265, the WIPP Project is not required to monitor groundwater to comply with the U.S. Environmental Protection Agency (EPA) RCRA. The WIPP GSP provides a basis for future compliance to the RCRA, as well as any other groundwater protection-related regulations, should the need arise.

4.1.4 Final Environmental Impact Statement (FEIS) Commitments

Section J.2.2 of the FEIS states that "...long-term groundwater sampling and water level monitoring will be conducted as part of the WIPP Environmental Monitoring Program."

4.1.5 Future Land Use Decisions

Data collected from the program will aid in making future groundwater-land use decisions (i.e., designing long term and passive institutional controls for the site).

This QA plan is driven by, and is supplemental to, both the WID QAPD, WP 13-1, and implementing WIPP Q&RA procedures.

4.2 GSP Quality Assurance Requirements

The following specific Q&RA requirements are unique to the GSP.

4.2.2 Quality Assurance Program

This plan is governed by the following documents: WP 13-1, WID Quality Assurance Program Description; and WP 02-3, Environmental Procedures Manual. Steps to ensure quality are incorporated, as needed, in the technical procedures used for groundwater surveillance activities. The EM manager or assigned designee is responsible for developing and maintaining this QA plan and groundwater surveillance procedures.

In accordance with the WID QAPD, Part I, Section 1, groundwater surveillance data activities are classified as Quality Code II.

4.2.3 Design Control

The design control requirements used by Westinghouse at the WID are described in Part II, Section 6 of the QAPD. The GSP will adhere to all applicable portions of these requirements when performing design activities.

4.2.4 Procurement Document Control

Procurement is carried out in accordance with WID procurement policies and procedures, as outlined in Part II, Section 7 of the QAPD, and WP 15-6, Purchasing Policies and Procedures Manual. Both documents require specification of a quality code and design class and concurrence by the Q&RA Department with procurement documents. Technical requirements for procured items and services are developed and specified in procurement documents. If deemed necessary to ensure attainment of the required characteristics, procurement documents may require suppliers to have an adequate QA program.

4.2.5 Instructions, Procedures, and Drawings

Provisions and responsibilities for the preparation and use of instructions and procedures at the WIPP are outlined in Part II, Section 4 of the QAPD. Quality-

affecting activities performed by or on behalf of groundwater surveillance are required to be performed in accordance with documented and approved procedures.

Technical procedures have been developed for each quality-affecting function performed for groundwater surveillance. The technical procedures unique to the GSP are contained in the procedures section of this manual. The procedures are as detailed as required and include, when applicable, quantitative or qualitative acceptance criteria to determine that activities have been satisfactorily accomplished.

Procedure requirements are in accordance with Section 4 of WP 13-1. Procedures will be prepared in accordance with applicable technical writer's guides.

4.2.6 Document Control

Requirements for the control of documents are outlined in Part II, Section 4 of the WID QAPD. Controls ensure that the latest approved versions of procedures are used in performing groundwater surveillance functions and that obsolete materials are removed from work areas.

4.2.7 Control of Purchased Material, Equipment and Services

WIPP policy requirements and associated responsibilities for the control of purchased material, equipment, and services are outlined in Part II, Section 7 of the QAPD. In accordance with current WIPP procurement policies and procedures, measures will be taken to ensure that procured items and services conform to specified requirements. These measures will include one or more of the following:

- An evaluation of the supplier's capability to provide items or services in accordance with the requirements, including the history of providing similar products or services satisfactorily.

- An evaluation of objective evidence of conformance, such as supplier submittal (i.e., QA plan).

- An examination and testing of items or services upon delivery.

If it is determined that additional measures are required to ensure quality in a specific procurement, additional steps may be provided in procurement documents and implemented by groundwater surveillance staff and/or the Q&RA Department. These additional assurances may include source inspection and audits or surveillance at the supplier's facilities.

4.2.8 Identification and Control of Items

Measures to ensure that only correct and accepted items are used at the WIPP are outlined in Part II, Section 8 of the QAPD. All items that potentially affect the quality of the GSP are uniquely identified and controlled to ensure that only accepted items are used.

Equipment is administered in accordance with WP 10-AD, WIPP Maintenance Administrative Procedures Manual. Calibration reports test data are maintained by the EM Department. Any "out-of-tolerance" condition is evaluated for potential impact on the validity of data. Impact evaluation and corrective actions are initiated per specific GSP instructions.

4.2.9 Control of Processes

All process control requirements of the QAPD are met by the GSP.

4.2.10 Inspection/Surveillance

Inspection and surveillance activities are conducted as outlined in Part II, Section 10 of the QAPD. The Q&RA Department is responsible for performing the applicable inspections and surveillance on the scope of work. Performance checks are performed by groundwater surveillance personnel as specified by the appropriate procedures, and by WID metrology laboratory personnel. Performance checks for the GSP are designed to determine the acceptability of purchased items and to assess degradation that occurs during use.

4.2.11 Test Control

Part II, Section 8 of the WID QAPD outlines the requirements and responsibilities of the WID for the control of tests. Tests to be performed for the GSP fall into two general categories: tests of items upon receipt and in service, and operability checks of equipment.

All tests are performed in accordance with documented and approved plans and/or procedures. Testing or experimental/monitoring plans or procedures contain the following provisions as applicable:

- Scope and/or definition or scope.
- Prerequisites such as calibrated instrumentation and supporting data; adequate test equipment and instrumentation, including accuracy requirements; completeness of item to be tested; suitable and controlled environmental conditions; and provisions for data collection and storage.
- Instructions for performing the test.

- Mandatory inspection and/or hold points to be witnessed by the WID or other designated representatives.
- Acceptance and rejection criteria.
- Methods of documenting or recording test data.
- Requirements for qualified personnel.
- Evaluation of test results by authorized personnel.

4.2.12 Control of Monitoring and Data Collection Equipment

Monitoring and Data Collection (M&DC) equipment is controlled and calibrated according WP 10-AD, WIPP Maintenance Administrative Procedures Manual, to ensure continued accuracy of groundwater surveillance data. Results of calibrations, maintenance, and repair are documented. Calibration records identify the reference standard and the relationship to national standards or nationally accepted measurement systems. Records are maintained to track uses of M&DC equipment. If M&DC equipment is found to be out of tolerance, the equipment is tagged and its use ceased until corrections are made. An evaluation shall be approved by the EM manager and corrective measures will be taken, as needed.

4.2.13 Handling, Storage, and Shipping

Handling, storage, packaging, and shipping of groundwater samples are controlled in accordance with WP 10-AD, WIPP Maintenance Administrative Procedures Manual. Proper documentation is prepared and maintained for each sample to minimize damage, loss, deterioration, and extraneous exposures.

4.2.14 Inspection and Acceptance Testing

Measures used by the WID to ensure that required inspections and tests performed are outlined in Part II, Section 8 of the WID QAPD. Controls are implemented in accordance with documented procedures to ensure that items are not used prior to passing required inspections and tests. The status is identified on the items or on documents traceable to the items. Items that have not been accepted are identified as such and stored separately from accepted items. The operating status of equipment is identified on the equipment or on the equipment list. Faulty equipment is tagged and, if practicable, physically segregated from the work area.

4.2.15 Control of Nonconforming Conditions

Part II, Section 8 of the WID QAPD describes the system used at the WIPP for ensuring that appropriate measures are established to control nonconforming

conditions. Nonconforming conditions connected to the GSP are identified in and controlled by documented procedures. Equipment that does not conform to specified requirements is controlled to prevent use. The disposition of defective items is documented on records traceable to the affected items. Prior to final disposition, faulty items are tagged and segregated. Repaired equipment is subject to the original acceptance inspections and tests prior to use.

4.2.16 Corrective Action

Requirements for the development and implementation of a system to determine, document, and initiate appropriate corrective actions after encountering conditions adverse to quality at the WIPP are outlined in Part I, Section 3 of the QAPD. Conditions adverse to acceptable quality are documented and reported in accordance with corrective action procedures and corrected as soon as practical. Immediate action will be taken to control work performed under conditions adverse to acceptable quality, and its results, to prevent degradation in quality.

The EM manager or designee investigates any deficiencies in groundwater surveillance activities to determine if there is an underlying root cause. All such actions are documented and reported to the Q&RA Department.

4.2.17 Quality Assurance Records

Part I, Section 4 of the QAPD outlines the policy used at the WIPP regarding identification, preparation, collection, storage, maintenance, disposition, and permanent storage of QA records. The EM manager or designee is responsible for the preparation and distribution of records in accordance with appropriate DOE Orders, policies, and directives.

Records to be generated in the GSP are specified by procedure. QA records are identified. This is the basis for the labeling of records as "QA" on the EM Records Inventory and Disposition Schedule (RIDS).

QA records document the results of the GSP implementing procedures and are sufficient to demonstrate that all quality-related aspects are valid. The records will be identifiable, legible, and retrievable in accordance with WP 15-PR, WID Records Management Plan, and QA record procedures.

While in the custody of the GSP group, the records shall be stored in a UL listed, one-hour fire-resistant cabinet. The EM manager shall coordinate with WIPP Project Records Services (PRS) for both periodic and perpetual transfer of records to PRS.

4.2.18 Assessments

Provisions and responsibilities for assessments are outlined in Part III, Sections 9 and 10, of the QAPD. Periodic, independent assessments of the GSP shall be scheduled, planned, and performed to verify that work is performed in accordance with specified requirements. The Independent Assessment Section has the responsibility and oversight authority for appraising GSP activities for compliance with applicable environmental statutes. Assessment teams will not include members of the GSP staff. Assessments are performed in accordance with applicable assessment procedures.

5.0 GSP WATER QUALITY SAMPLING PLAN

5.1 Scope

This section of the WIPP GSP Plan serves as the controlling document for the WQSP. The WQSP is a subprogram of the GSP.

The WQSP was initiated in January 1985. The objective of the program is to collect representative and reproducible groundwater samples from water-bearing zones in the area of the WIPP site. The purpose of the program is to provide defensible data for meeting the requirements of site characterization, performance assessment, regulatory compliance, and permitting. A program plan that defined the basic structure and operational activities of the program was initially developed by Colton and Morse (1985). The program plan was replaced in 1987 by WP 07-2, Waste Isolation Pilot Plant Water Quality Sampling Manual. In 1991 the WQSP manual was replaced by WP 02-1, Waste Isolation Pilot Plant Groundwater Monitoring Program Plan and Procedures Manual.

5.1.1 General

From 1984 to 1990, the WQSP was designed to characterize the physical and chemical characteristics of representative groundwater samples occurring within and immediately surrounding the WIPP site. Various wells were serially sampled, three times each, to determine the representative character of the groundwater present at each location. Data collected were supplied to the ES&H Department and used to develop a baseline of water quality data as part of the Radiological Baseline Program. A nonradiological database was developed to support the background water quality characterization report. Data were also supplied to and used by Sandia National Laboratories (SNL) for site characterization and performance assessment. By the close of 1990, the groundwater of interest had been characterized, and the objective of the program shifted from characterization to surveillance.

On October 1, 1988, the ES&H Department assumed responsibility for the WQSP. Water quality sampling activities were coordinated with the Environmental Monitoring Program.

Collection of groundwater quality data continues to assist the DOE in meeting performance assessment, regulatory compliance, and permitting requirements. The data also provide:

- Radiological and nonradiological water quality input to the WIPP Environmental Monitoring Program.
- A means to comply with future groundwater inventory and monitoring regulations.
- Input for making land-use decisions (i.e., designing long-term active and passive institutional controls for the site).

Groundwater exists both above and below the WIPP repository, but no hydrologic continuity exists between the repository and the groundwater. Groundwater below the repository occurring in the sandstones of the Delaware Mountain Group (Powers, et al., 1978) is isolated by bedded salt deposits in the lower part of the Salado Formation and in the underlying Castile Formation. Groundwater below the repository is not being monitored as part of this program.

Groundwater above the repository is being monitored. Groundwater exists in both the Dewey Lake Formation and the Rustler Formation. Zones monitored for background characterization within the Rustler are the Culebra and the Magenta members. These zones appear to be dolomite units isolated from one another by impermeable units. With the exception of excavated shafts at WIPP, these zones are isolated from the repository excavations by bedded salt deposits in the upper two thirds of the Salado Formation.

Postbackground surveillance is focused on the Culebra because it is the primary flow path within the Rustler formation. Databases are maintained for the Magenta so that if the need arises surveillance of the Magenta can be resumed.

The Culebra is areally persistent, but quantity and quality of water vary considerably from place to place. The dolomite is vuggy, fractured, and commonly associated with anhydride (Lambert and Mercer, 1977). The Culebra has a low hydraulic conductivity. It is a fractured unit that is best modeled as a dual-porosity media. Water yields are small and saline (Powers et al., 1978).

The Magenta is finely crystalline and dense. Like the Culebra, the Magenta has a low hydraulic conductivity through fractures and contains limited amounts of poor quality water (Powers et al., 1978).

The Dewey Lake Redbeds consist of orange-red silt stone, mud stone, and some sandstone. The Dewey Lake Redbeds do not form an aquifer, but some permeable sand lenses are present and those yield limited quantities of fresh water to a few

private wells in the area around the WIPP site (Powers et al., 1978). One such sand lens has been identified within the WIPP boundary and is scheduled for surveillance as part of the WQSP.

5.2 Surveillance Well Construction

Many of the WIPP surveillance wells were drilled and completed prior to 1980. As the WIPP Project progressed, additional monitoring wells were completed in the vicinity of the site. Drilling of the bulk of WIPP surveillance wells began in 1976 and continued into 1988.

In general, all of these wells were drilled as part of the geologic site characterization and resource evaluation programs. Most WIPP surveillance wells were drilled and completed using oil field techniques. Surveillance wells at the site have been completed, generally, using two types of installations. One installation requires drilling the well to some depth below the base of the Culebra and then casing the well to the bottom of the hole. The interval of the Culebra or Magenta is then perforated to allow access to the formation for testing or sampling purposes. The second type of installation consists of drilling the hole to a depth just above the top of the Culebra, installing well casing to the bottom of the drilled hole, and coring or drilling through the Culebra interval, leaving the Culebra interval open to the formation.

These types of well completions presented problems in collecting undisturbed and representative samples from the water-bearing units. The open-hole completions have, in some cases, resulted in sediments below the Culebra being exposed in the sampling interval. In some cases, these sediments are rich in halite or other evaporite minerals, causing the water chemistry in the well bore and the water-bearing unit surrounding the well to be altered. Often, during drilling and completion of surveillance wells, fluids containing fresh water, saturated brine, and drilling fluids containing petroleum products have been introduced into the well bore. In some cases, these fluids were left standing in the well bore for extended periods of time, resulting in contamination of the surrounding formation (Crawley 1988).

Standard oil field steel well casings have been used during completion of the WIPP surveillance wells. This type of casing is easily corroded by the brackish to brine water found in the WIPP area. Based on serial sampling results, it appears that the products of well casing corrosion migrate from the well bore into the formation, resulting in a halo or plume of groundwater with altered chemistry surrounding the surveillance wells. Obtaining a representative sample has required that the surveillance wells be pumped for long periods of time to remove the contamination.

Well drilling and completion techniques such as those described above are usually not used for installation of monitoring wells employed in RCRA or other groundwater sampling programs, due to the likelihood of aquifer contamination. These practices

required that the WQSP use extensive groundwater pumping in order to obtain uncontaminated water samples.

The difficulty in obtaining representative groundwater samples, due to the design of the wells used by the WQSP, necessitated the use of a serial sampling technique. Serial sampling and the associated equipment are discussed later in this section.

Seven observation wells were completed after the baseline was established using EPA recommended drilling methods and casing materials that have the potential to meet RCRA monitoring standards. Six of the wells were completed in the Culebra; one well in the Dewey Lake formation. Two years of sampling are scheduled prior to the anticipated receipt of waste. The data gathered from these wells will be compared to the existing database and the existing background data will be appended as appropriate.

The configuration of the seven new observation wells may well preclude the necessity to perform serial sampling. However, sampling of a portion of the older surveillance wells may be necessary in years to come. Therefore, a discussion of serial sampling techniques is included in this document.

5.3 Sampling Program Description

The WQSP has employed two types of sampling procedures at the WIPP: serial sampling and final sampling.

5.3.1 Serial Sampling

Serial sampling is the collection of sequential samples for the purpose of determining when the water chemistry stabilizes or reaches a steady state. Ideally, when the water chemistry stabilizes, it is assumed that the chemistry is representative of the native formation fluid, and a final sample is collected. However, in reality, serial sampling leads to the collection of water samples with reproducible chemistries which may or may not be representative of the undisturbed groundwater. The water samples may still be impacted by well construction practices and effects from the installation of downhole pumping and sampling equipment.

During the background characterization phase of the WQSP serial sample, field parameters were monitored on a daily basis. After completion of the background characterization phase, monitoring of serial sample parameters was modified by pumping each well for 48 hours prior to the start of serial sampling then comparing the serial sampling analysis results to the average last day serial sample results for previous sampling rounds. A 95 percent confidence interval was established for comparison standards.

The field analytical parameters found to be the most useful in identifying a steady state condition of the water chemistry include chloride, divalent cations (hardness), and alkalinity, which are analyzed by classic wet chemistry bench methods (titration). Total iron has also been found to be a useful indicator and is analyzed using spectrophotometric methods. Other serial sampling parameters analyzed in the field include measurement of pH, Eh, temperature, specific conductance, and specific gravity. Procedures for collection and analysis of serial samples are processed, approved, and maintained by the site documentation process.

5.3.2 Final Samples

Final groundwater samples are collected once evidence from serial sampling indicates that the pumped groundwater has reached a chemical steady state. Final samples are forwarded to a contract analytical laboratory for analysis.

Final samples are collected in the appropriate type of container for the specific analysis to be performed. For each parameter analyzed, a sufficient volume of sample is collected to satisfy the volume requirements of the analytical laboratory. This includes an additional volume of sample water necessary for maintaining quality control standards. All final samples are treated, handled, and preserved as required for the specific type of analysis to be performed. Details about sample collection, preservation, and volumes required for individual types of analyses are found in the applicable procedures generated, approved, and maintained by the site documentation process.

Splits of the final sample are provided to oversight agencies and WIPP stakeholders as requested by the DOE. A split of the sample is also placed in storage within the ES&H Environmental Sample storage area and held until final reports from the contract analytical laboratory have been evaluated and approved. When the final laboratory report has been approved the samples are removed from storage and destroyed.

Detailed protocols, in the form of procedures, assure that samples are collected in a consistent and repeatable fashion. Procedures applicable to water quality sampling are generated, approved, and maintained by the site documentation process.

The serial sampling process will probably not be needed with the wells completed specifically for water quality sampling. However, during the first two years of sampling, the wells will be serially sampled using an abbreviated method. It is anticipated that changes in the water chemistry from stagnated to representative will occur within the first 24 hours of the purging process. Whereas, this change usually occurred over a seven-day period with the old wells.

During the first two or three years of sampling, these wells will be serially sampled with the first sample being analyzed as soon as possible after the pump is turned on

and daily there after for a period of four days or until the field parameters (chloride, divalent cations, alkalinity and iron) stabilize. Eh, pH, and conductance will be monitored continuously by using a flow cell with ion-specific electrodes and a real-time readout. After two years of sampling data have been accumulated, a decision will be made to determine if the serial sampling process can be eliminated. If serial sampling is removed from the water quality sampling well protocol, the decision to collect samples will be based on the number of well bore volumes purged and the results of continuous monitoring of temperature, Eh, pH, and conductance.

5.4 Groundwater Pumping and Sampling Systems

The water-bearing units at the WIPP are highly variable in their ability to yield water to surveillance wells. The Culebra, the most transmissive hydrologic unit in the WIPP area, exhibits transmissivities that range many orders of magnitude across the site area and has been the primary focus of the GSP. The Magenta has a lower transmissivity and yields very small quantities of water to wells. Because the water-yielding characteristics of the hydrologic units at the WIPP are variable, different types of pumping equipment are used during water quality sampling activities.

The groundwater pumping and sampling systems used to collect a groundwater sample are designed to provide continuous and adequate production of water so that a representative groundwater sample can be obtained. The wells used for water quality sampling vary in yield, depth, and pumping lift. These factors affect the duration of pumping as well as the equipment required at each well. Based upon expected yields, the wells monitored at WIPP can be divided into three categories according to flow rate: (1) high flow rate of 10 to 25 gallons per minute (gpm); (2) medium flow rate or 1 to 10 gpm; and (3) low flow rate of less than 1 gpm.

The high and medium flow rate wells may use a submersible pump-packer assembly. The low-volume wells may require a gas-driven piston pump-packer assembly. A discussion of the different pump-packer equipment is provided below.

The type of pumping and sampling system to be used in a well depends primarily on the aquifer characteristics and well construction. For example, if well construction is such that it yields contamination to the aquifer (i.e., metal casing) a packer is normally recommended to minimize purging time. If the aquifer yields adequate water to the well to be classified a high or medium production well, a submersible electric pump may be used. However, if the well is completed with the water-bearing unit uncased, a gas piston pump may be needed to minimize stress to the formation walls to prevent collapse of the formation.

Wells that are completed to water quality standards are cased and screened through the production interval with materials that do not yield contamination to the aquifer or allow the production interval to collapse under stress. An electric, submersible pump installation without the use of a packer is an acceptable installation in this instance.

The largest amount of discharge from the submersible pump takes place from a discharge pipe. In addition to this main discharge pipe a dedicated nylon sample line, running parallel to the discharge pipe, is also used. Flow through the pipe is regulated on the surface by a flow control valve and/or variable speed drive controller. Cumulative flow is measured using a totalizing flow meter. Flow from the discharge pipe is routed to a discharge tank for disposal.

The dedicated nylon sampling line is used to collect the water sample that will undergo analysis. By using a dedicated nylon sample line, the water is not contaminated by the metal discharge pipe. The sample line branches from the main discharge pipe a few inches above the pump. Flow from the sample line is routed into the sample collection area. Flow through the sample collection line is regulated by a flow-control valve. The sample line is insulated at the surface to minimize temperature fluctuations.

A gas-driven pump and sampling system can be used on any volume well. When used, the pump intake is set at a predetermined depth near or in the production zone. The pumping rate is adjusted to maintain the water level in the well above the pump intake.

The flow rate for gas driven pumps is controlled by regulating the air pressure on the pump intake or by a flow control valve. Water is continuously discharged into a water storage tank. Detailed protocol for assembling, installing, and controlling pumping and sampling systems is found in the procedures generated, approved, and maintained by the site documentation process.

5.5 Pressure Monitoring Systems

Regardless of which pump is used when sampling a well that was drilled for the geologic site characterization and resource evaluation program, a packer is used to isolate the pump intake from contaminated well-bore fluid that exists in the well above the sampling zone. If the packer seal is not good, contaminated water from above the packer can leak into the formation water being sampled and bias analytical results. If the packer has a good seal the pressure above the inflated packer should remain constant.

Pressure above the packer is monitored using transducers and/or bubblers to verify that the seal on the packer is good. Pressure below the packer is monitored to ensure that water levels do not fall below the pump intake. Periodic checks of the pressures are conducted during field sampling to verify packer seal integrity. These field checks are recorded on Field Activity Log Forms.

Wells drilled to water-quality specifications do not require the installation of a packer because sample biases due to well construction deficiencies are not present. However, pressures will be monitored in the formation to maintain water level above

the pump intake. Procedures governing the installation and use of pressure monitoring devices are generated, approved, and maintained by the site documentation process.

5.6 Sample Analysis

The mobile field laboratory provides a work place for conducting field sampling and analyses. The laboratory is positioned near the wellhead, is climate controlled, and contains the necessary equipment, reagents, glassware, and deionized water for conducting the various analyses.

Two types of water samples are collected: serial samples and final samples. Serial samples are taken at regular intervals and analyzed in the mobile laboratory for various physical and chemical parameters (called field parameters). The serial sample data are used to determine the chemical steady state conditions of the groundwater, as a direct function of the volume of the water being pumped from the well. Interpretation of the serial sampling data enables the TL to make a determination of when steady state conditions are attained in the pumped groundwater.

Final samples are collected when the serially sampled field parameters have achieved a steady state. If one or more of the field parameters do not stabilize, and there is reason to believe it will not, the TL may choose to collect the final sample regardless of this instability in the field parameter(s).

The objective of the serial sampling effort is to obtain representative water samples in a reproducible manner. By definition, a representative groundwater sample is a sample of undisturbed groundwater. A groundwater sample is considered to be representative of the undisturbed groundwater only if it is chemically identical to the undisturbed groundwater (i.e., completely unaltered by the effects of drilling, postdrilling processes and reactions, and sampling procedures). Obtaining a representative groundwater sample is a theoretical ideal. For example, the redox potential of the aquifer groundwater, Eh, is likely to change as a result of pressure decreases (gas loss) and contamination by atmospheric oxygen that occurs during the sampling process. The ratios between the different oxidation states of a multivariant element may change, and the total concentration of that element may also change during sampling due to precipitation.

To determine how close the pumped groundwater is to being representative, a comparison is made by monitoring the same selected field parameters which were used to initially define the background characteristics of the water. When these parameters appear stable, then the determination is made that the water sample is representative. Stability is usually defined as ± 5 percent of the average of preceding parameter measurements made on the final day of sampling for previous rounds.

When stability has been determined, a final sample is collected. The final sample is considered to be as representative a sample of the undisturbed groundwater as can possibly be obtained considering the analytical and technical means at hand.

5.6.1 Serial Samples

Serial samples are collected and analyzed in the mobile laboratory to detect and monitor the chemical variation of the groundwater as a function of the volume of water pumped. The purpose of implementing this rigorous serial sampling and analysis program is to ascertain when the pumped groundwater has reached a chemical steady state. Once serial sampling begins, the frequency at which serial samples are collected and analyzed is left to the discretion of the TL. The serial sampling frequency is based upon the site-specific conditions existing at each well, but usually is performed a minimum of three times during a sampling round.

The three field parameters of temperature, Eh, and pH are determined by either an "in-line" technique, using a self-contained flow cell, or an "off-line" technique, in which the samples are collected from a nylon sample line at atmospheric pressure. The iron, divalent cation, chloride, alkalinity, specific conductance, and specific gravity samples are collected from the nylon sample line at atmospheric pressure.

New polyethylene containers are used to collect the serial samples from the nylon sample line. Serial sampling water collected for solute and specific conductance determinations is filtered through a 0.45 μm filter membrane using a stainless steel, in-line filter holder. Filtered water is used to rinse the sample bottle prior to serial sample collection. Unfiltered groundwater is used when determining temperature, pH, Eh, and specific gravity. Sample bottles are properly identified and labeled.

The filtered sample collected for solute analyses is immediately analyzed for iron and alkalinity, as these two solution parameters are extremely sensitive to changes in the ambient water-sample pressure and temperature. The sample aliquot needed for the other chemical parameter analyses may be taken from a second filtered sample bottle. Temperature, pH, and Eh, when not measured in a flow cell, are measured at the approximate time of serial sample collection; these samples are collected from the unfiltered sample line.

Experience gained from the serial sampling of wells has shown that samples to be analyzed for chloride and divalent cations can be stored for one week prior to analysis with confidence that the analytical results will not be altered.

Upon completion of the collection of the final sample suite, the serial sample bottles accrued throughout the duration of the pumping of the well are discarded. No serial sample bottles will be reused for sampling purposes of any sort. However, serial samples may be archived for a period of time depending upon the need. Procedures

for sample collection and analysis are generated, approved, and maintained by the site documentation process.

5.6.2 Final Samples

The final sample is collected once the pumped groundwater has achieved a chemically steady state. A serial sample is also collected and analyzed for each day of final sampling. Sample preservation, handling, and transportation methods are designed to maintain the integrity and representativeness of the final samples.

Prior to collecting the final samples, the collection team must consider the analyses to be performed so that proper shipping or storage containers can be assembled.

Final samples are sent to contract laboratories and analyzed for general chemistry, radionuclides, metals, and selected volatile organic compounds that are specific to the waste anticipated to arrive at WIPP. Gases and redox-couples were analyzed during the baseline study, but these data are not needed for environmental monitoring and are no longer obtained on a routine basis.

Water samples are collected at atmospheric pressure using either the filtered or unfiltered nylon sampling lines branching from the main sample line. The samples are collected in new and unused glass and plastic containers.

Before the final sample is taken, all plastic and glass containers are rinsed with the pumped groundwater, either filtered or unfiltered, dependent upon analysis protocol. When the rinsing procedure is completed, the final sample is collected.

5.7 Sample Preservation, Tracking, Packaging and Transportation

Many of the chemical constituents that are measured are not chemically stable and need to be preserved. Samples requiring acidification are treated with either high purity hydrochloric acid, nitric acid, or sulfuric acid (ULTREX or equivalent), depending upon the standard method of treatment required for the particular parameter suite.

The procedure used by the contract laboratory to which the samples are being sent prescribes the type and amount of preservative which should be used. This information is recorded on the Final Sample Checklist for use by field personnel when final samples are being collected.

The sample tracking system at WIPP uses uniquely numbered Chain of Custody Forms and Request for Analysis Forms. The primary consideration for storage or transportation is that samples must be analyzed within the prescribed holding times for the parameters of interest. Procedures for sample tracking and preservation are generated, approved, and maintained by the site documentation process.

The prescribed transport temperature for the organic samples is four degrees Celsius. This temperature must be maintained until the sample reaches the contracted laboratory.

Insulated shipping containers packaged with reusable blue ice are used to keep the samples cool during transport to the contract laboratory. Hold times for specific analytical parameters require samples to be shipped by express air freight. The coolers are packaged to meet Department of Transportation and International Air Transportation Association commercial carrier regulations.

5.8 Quality Assurance, Records Management and Document Control

All aspects of quality assurance, records management, and control of documents generated as a result of WQSP are governed by the QAPD; WP 15-PR, Records Management Plan; and implementing procedures generated, approved, and maintained by the site documentation process.

A chemistry laboratory notebook is maintained in the mobile laboratory to record the overall conditions at the well, the analytical difficulties or problems experienced, and any information which may be pertinent to future interpretation and scientific use of the field data. The original notebook is kept in the field laboratory. A copy of the notes made for each sampling round is kept in a fire-resistant file cabinet.

All field data collected are organized into a data book. The typical field data book contains the following:

- A copy of all of the notes entered into the laboratory notebook concerning the sampling round.
- A copy of all chain of custody forms and request for analysis forms used to distribute the final samples.
- A copy of the completed final sample checklist.
- A copy of all standardization forms.
- A hard copy printout of all computer data entries.
- A copy of all of the Serial Sampling Report Forms submitted for the sampling round.
- A copy of all worksheets used to prepare the data for entry into the computer.

- A written summary report containing a description of the well completion data, a brief summary of serial sampling results, and general observations.
- A copy of all Field Sketch Plan Forms.
- A copy of all Field Activity Log Forms.
- A computer printout of all data logger information, if a data logger was used.
- Validated Check Print copies of all data sheets.

A contract laboratory data book is made for each contract laboratory used to analyze samples from a particular well. The contract laboratory data book contains at a minimum:

- A copy of the contract laboratory analytical report.
- A copy of the computer data generated.

Data collected as a result of WQSP activities are summarized and reported on an annual basis in the Site Environmental Report. Raw data are stored in fireproof cabinets in the EM Section for a period of two years and then turned over to PRS for storage in accordance with the RIDS.

5.9 Calibration Requirements

The equipment used to collect data for the WQSP is to be calibrated in accordance with WP 10-AD, WIPP Maintenance Administrative Procedures Manual. The metrology laboratory is responsible for calibrating needed equipment on schedule, in accordance with written procedures. The EM Section is responsible for maintaining current calibration records for each piece of equipment.

6.0 WATER LEVEL MONITORING PLAN

6.1 Scope

This section of the WIPP GSP serves as the controlling document for the WLMP. The WLMP is a subprogram of the GSP. The quality assurance activities of the WLMP are in strict accordance with the QAPD and the quality assurance implementing procedures specific to environmental monitoring are found in WP 02-3, Environmental Monitoring Procedures Manual.

Water level monitoring will continue through the postoperational phase of the WIPP. This plan addresses the activities of the WLMP during the preoperational and operational phases of the WIPP. Postoperational activity plans will be formulated at a

later date and will address the objectives of water level monitoring as required at the time of decommissioning.

6.2 Introduction

This program will continue the collection and documentation of water level data initiated by the U.S. Geological Survey (Richey, 1987) and SNL (Stensrud et al., 1988) as part of the WIPP Site Characterization Program.

As currently planned, water level measurements will be conducted using hydrologic test wells that were constructed for the site characterization and WQSP. These test wells are distributed geographically both within and surrounding the WIPP site. The frequency of measurement is subjectively defined by the need to record the dynamic nature of the potentiometric surface through time.

On October 1, 1988, the ES&H Department assumed responsibility for Groundwater Level Monitoring Activities. At that time a WLMP plan was still being developed. In June of 1989, an initial plan was finalized entitled WP 07-2, WIPP Water Level Monitoring Program Plan, IT Corp. (June 1989). WP 07-2 was subsequently replaced in 1990 by WP 02-1, Groundwater Monitoring Program Plan and Procedures Manual.

Collection of groundwater-level data assists the DOE in meeting performance assessment, regulatory compliance, and permitting requirements. These data also provide:

- Data collection as required by the Environmental Monitoring Plan.
- A means to fulfill commitments made in the FEIS.
- A means to comply with future groundwater inventory and monitoring regulations.
- Input for making land use decisions, (i.e., designing long-term active and passive institutional controls for the site).
- Assistance in understanding any changes to readings from the water-pressure transducers installed in each of the shafts to monitor water conditions behind the liners.
- An understanding of whether or not the horizontal and vertical gradients of flow are changing over time.

6.3 Objective

The objective of the WLMP is to extend the documented record of water-level fluctuations in the Culebra and Magenta members of the Rustler Formation in the vicinity of the WIPP facility. Water-level data will also be collected from wells completed in other water-bearing zones overlying and underlying the WIPP repository horizon when access to those zones is possible. This includes, but is not limited to, the Bell Canyon Formation, the Forty Niner member of the Rustler, the contact zone between the Rustler and Salado Formations, and the Dewey Lake Red Beds, when access to these zones is possible.

The scope of the program is subject to change depending upon the following:

- Data trends
- Performance assessment program needs
- Environmental Monitoring Program needs
- Regulatory compliance needs

Water level measurements will be taken monthly in at least one accessible completed interval at each available well pad. At well pads with two or more wells completed in the same interval, quarterly measurements will be taken in the redundant wells.

Water level monitoring will continue through the life of the WIPP Project. It may be deemed necessary to temporarily increase the frequency of monitoring to effectively document naturally occurring or artificial perturbations that may be imposed on the hydrologic systems at any point in time. This will be conducted in selected key wells by increasing the frequency of the manual water-level measurements or by monitoring water pressures with the aid of electronic pressure transducers and remote data-logging systems.

One of the postulated contaminate pathways to the biosphere in the event of a release is believed to be in the water-bearing zones of the Rustler Formation, more specifically, the Magenta and Culebra members. The Culebra is believed to be the more conductive of the two (Mercer, 1983) and has received the most attention in site characterization studies. Other water bearing zones in the vicinity of the WIPP site, in which a limited number of hydrologic test wells have been completed, include the Dewey Lake Red Beds, the Rustler/Salado Contact, the Forty Niner Member of the Rustler, and the Bell Canyon Formation. All of the above listed zones will be monitored as part of this program plan, subject to availability.

Water level fluctuations of confined water bearing units may result from a variety of hydrologic phenomena (Freeze and Cherry, 1979) and (Davis and DeWeist, 1966). These include:

- Changes in groundwater storage (e.g., groundwater recharge)
- Changes in atmospheric pressure
- Deformation of the water bearing zone (e.g., earthquakes and earth tides)
- Disturbances within or adjacent to a well (e.g., groundwater pumping and shaft construction)

Interpretation of water level measurements and corresponding fluctuations over time is complicated at the WIPP by spatial variation in fluid density both vertically in well bores and areally from well to well. To monitor the hydraulic gradients of the hydrologic flow systems at the WIPP accurately, actual water level measurements and the densities of the fluids in the well bores must be known. When both of these parameters are known, equivalent freshwater heads can be calculated. The concept of freshwater head is discussed in Lusczynski (1961) where the following definition is provided:

> Fresh water head at a given point in groundwater of variable density is defined as the water level in a well filled with fresh water from that point to a level high enough to balance the existing pressure at that point. Fresh water heads define hydraulic gradients along a horizontal.

A discussion explaining the calculation of freshwater heads from midformation depth at WIPP can be found in Haug, et al. (1987).

A Pressure Density Survey Program (PDSP) has been conducted to determine the actual variation in density gradients existing in the test wells. The PDSP measured the actual midformation pressures of the Culebra. Data from this program have identified those wells in which some adjustment to measured water level values must be accounted for in order to calculate the measured water levels accurately in terms of equivalent freshwater heads.

6.4 Field Methods

To obtain an accurate groundwater level measurement, a calibrated water level measuring device is lowered into a test well and the depth to water is recorded from a known reference point. When using an electrical conductance probe, the depth to water can be determined by reading the appropriate measurement markings on the embossed measuring tape when the alarm is activated at the surface. Specific procedures regarding the specific activities governing the Water Level Monitoring Program are generated, approved, and maintained by the site documentation process.

6.5 Records and Document Control

All incoming data will be processed in a timely manner to assure data integrity. The data management process for water level measurements begins with completion of the field data sheets. Date, time, tape measurement, equipment identification number, calibration due date, initial of the field personnel, and equipment/comments are recorded on the field data sheets. If, for some unexpected reason, a measurement is not possible (i.e., a test is under way that blocks entry to the well bore), then a notation as to why the measurement was not taken is recorded in the comment column. Personnel also use the comment column to report any security observations (i.e., well lock missing).

Data recorded on the field data sheets and submitted by field personnel are subject to guidelines outlined in WP 02-3, Environmental Procedures Manual. The data are entered onto a computerized worksheet. The worksheet calculates water level in both feet and meters relative to the top of casing and also relative to mean sea level.

A check print is made of the worksheet printout. The check print is used to verify that data taken in the field is properly reported on the database printout. A minimum of 10 percent of the spreadsheet calculations are randomly verified on the check print to ensure that calculations are being performed correctly. If errors are found, the worksheet is corrected. The data contained on the computerized worksheet are translated into a database file. A printout is made of the database file. The data each month are then compiled into report format and transmitted to the appropriate agencies as requested by the DOE.

A computerized database file is maintained for all groundwater level data. Monthly and quarterly data are appended into a yearly file. Upon verification that the yearly database is free of errors, it is appended into the project database file. A printed copy of the project database is maintained in the ES&H EM fire-resistant storage area current through December of the preceding year.

6.6 Reporting

Data collected from this program are reported in the Annual Site Environmental Report (ASER). The ASER includes all applicable information that may affect the comparison of water level data through time. This information will include but is not limited to:

- Well configuration changes that may have occurred from the time of the last measurement (i.e., plug installation and removal, packer removal and reinstallation, or both; and the type and quantity of fluids that may have been introduced into the test wells).

- Any pumping activities that may have taken place since publication of the last annual report (i.e., water quality sampling, hydraulic testing, and shaft installation or grouting activities).

6.7 Calibration Requirements

The equipment used in taking groundwater level measurements is to be calibrated in accordance with WP 10-AD, WIPP Maintenance Administrative Procedures Manual. The WID metrology laboratory is responsible for calibrating needed equipment on schedule, in accordance with written procedures. The EM Section is responsible for maintaining current calibration records for each piece of equipment.

WQSP 1 THROUGH WQSP 6 AND WQSP 6A

WQSP-1 - Well WQSP-1 was drilled between September 13 and 16, 1994 to a total depth of 737.0 ft below ground surface (bgs). The borehole was drilled through the Culebra and extends 15.0 ft into the unnamed lower member of the Rustler Formation. The well was drilled to a depth of 693 ft bgs using compressed air as the drilling fluid. The interval from 693 to 737 ft bgs (the total depth) was drilled using air mist with a foaming agent as the drilling fluid. WQSP-1 was drilled to 695.6 ft bgs using a 9⅞-inch drill bit and was cored from 695.6 to 737.0 ft bgs using a 5¼-inch core bit to cut 4-inch-diameter core. After coring, WQSP-1 was reamed to 9⅞ inches in diameter to total depth. WQSP-1 was cased from the surface to 737 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen across the Culebra interval from 702 to 727 ft bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 640 to 651 ft bgs and with 8/16 Brady gravel from 651 to 737 ft bgs. Based on core log results, the Culebra is located from 699.0 to 722.0 ft bgs.

WQSP-2 - WQSP-2 was drilled between September 6 and 12, 1994 to a total depth of 846.0 ft bgs. The borehole was drilled through the Culebra and extends 12.3 ft into the unnamed lower member of the Rustler Formation. The well was drilled to a depth of 800 ft bgs with a 9⅞-inch drill bit using compressed air as the drilling fluid. The interval from 800 to 845 ft bgs (the total depth) was drilled with a 5¼-inch core bit to cut 4-inch-diameter core using air mist with a foaming agent as the drilling fluid. After coring, WQSP-2 was reamed to 9⅞ inches in diameter to total depth. WQSP-2 was cased from the surface to 846 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen across the Culebra interval from 811 to 836 ft bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 790 to 793 ft bgs and with 8/16 Brady gravel from 793 to 846 ft bgs. Based on core log results, the Culebra is located from 810.1 to 833.7 ft bgs. See

WQSP-3 - Well WQSP-3 was drilled between October 21 and 26, 1994, to a total depth of 880.0 ft bgs. The borehole was drilled through the Culebra and extends 8.6 ft into the unnamed lower member of the Rustler Formation. The well was drilled to a total depth of 880 ft bgs using compressed air as the drilling fluid. The borehole was cleaned using air mist with a foaming agent. WQSP-3 was drilled to 833 ft bgs using a 9⅞-inch drill bit and was cored from

833.0 to 879.0 ft bgs using a 5¼-inch core bit to cut 4-inch-diameter core. After coring, WQSP-3 was reamed to 9⅞ inches in diameter to a total depth of 880 ft bgs. WQSP-3 was cased from the surface to 880 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen across the Culebra interval from 844 to 869 ft bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 827 to 830 ft bgs and with 8/16 Brady gravel from 830 to 880 ft bgs. Based on core log results, the Culebra is located from 844.0 to 870.4 ft bgs. See Figure 5-3.

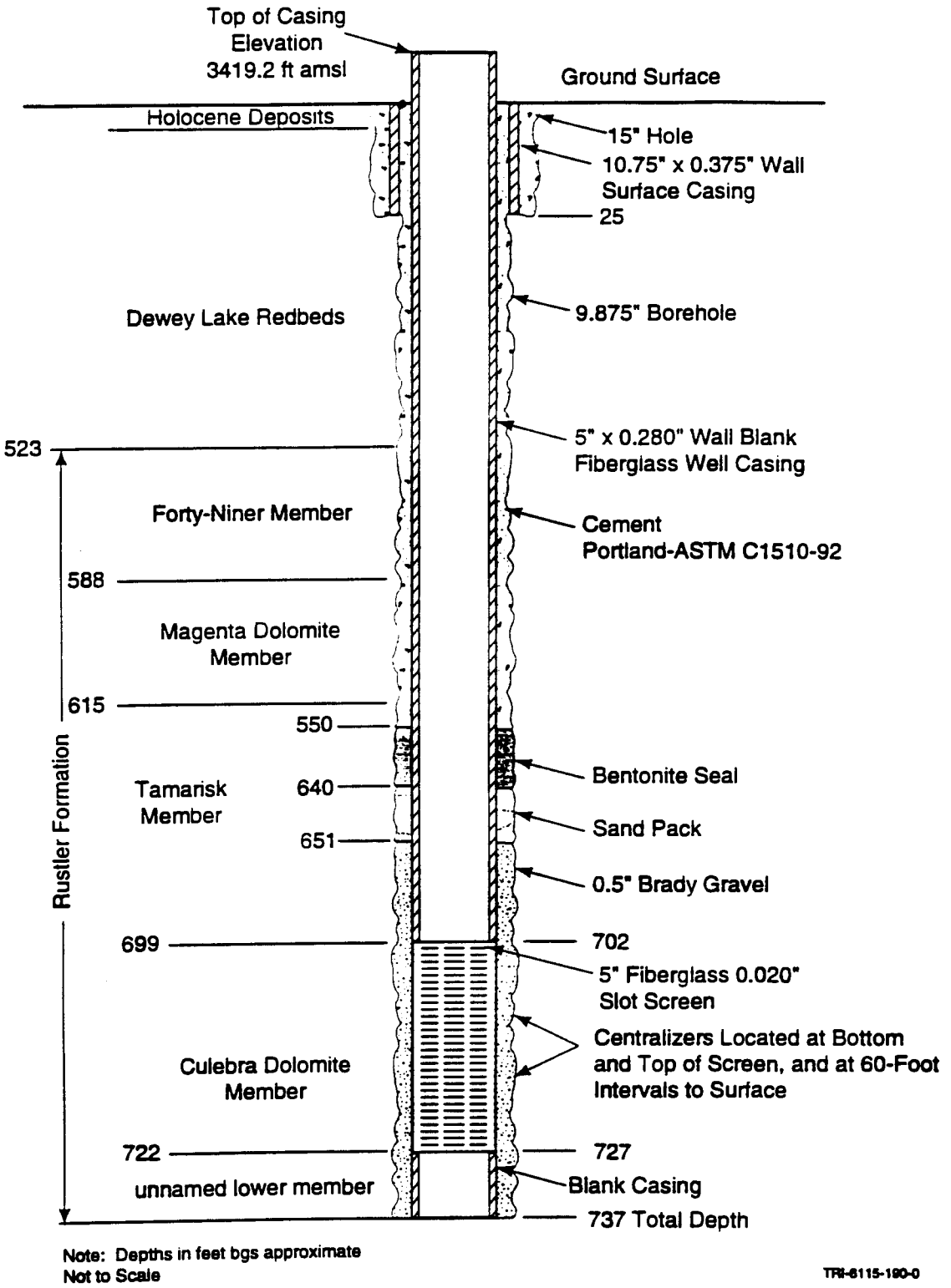
WQSP-4 - Well WQSP-4 was drilled between October 5 and 10, 1994, to a total depth of 800.0 ft bgs. The borehole was drilled through the Culebra and extends 9.2 ft into the unnamed lower member of the Rustler Formation. The well was drilled to a depth of 740.5 ft bgs with a 9⅞-inch drill bit using compressed air as the drilling fluid. The interval from 740.5 to 798.0 ft bgs was cored using a 5¼-inch core bit to cut 4-inch-diameter core using air mist with a foaming agent as the drilling fluid. After coring, WQSP-4 was reamed to 9⅞ inches in diameter to a total depth of 800 ft bgs. WQSP-4 was cased from the surface to 800 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen across the Culebra interval from 764 to 789 ft bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 752 to 755 ft bgs and with 8/16 Brady gravel from 755 to 800 ft bgs. Based on core log results, the Culebra is located from 765.6 to 790.8 ft bgs. See Figure 5-4.

WQSP-5 - Well WQSP-5 was drilled between October 12 and 19, 1994, to a total depth of 681.0 ft bgs. The borehole was drilled through the Culebra and extends 10.6 ft into the unnamed lower member of the Rustler Formation. The well was drilled to a depth of 676 ft bgs using compressed air as the drilling fluid. The borehole was cleaned using air mist with a foaming agent. WQSP-5 was drilled to 648 ft bgs using a 9⅞-inch drill bit and was cored from 648.0 to 676.0 ft bgs using a 5¼-inch core bit to cut 4-inch-diameter core. After coring, WQSP-5 was reamed to 9⅞ inches in diameter to a total depth of 681 ft bgs. WQSP-5 was cased from the surface to 681 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen across the Culebra interval from 646 to 671 ft bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 623 to 626 ft bgs and with 8/16 Brady gravel from 626 to 681 ft bgs. Based on core log results, the Culebra is located from 648.0 to 674.4 ft bgs. See Figure 5-5.

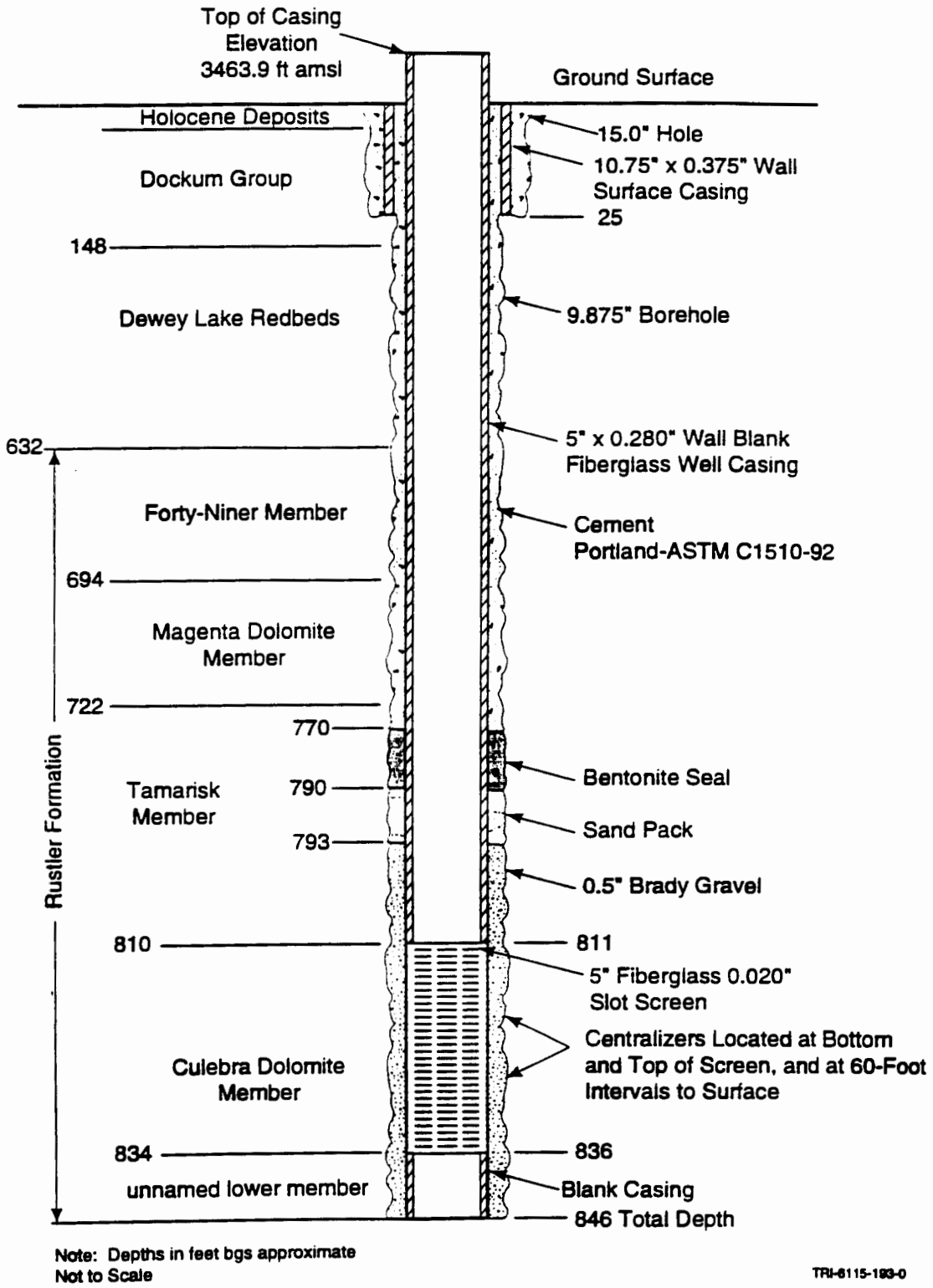
WQSP-6 - Well WQSP-6 was drilled between September 26 and October 3, 1994, to a total depth of 616.6 ft bgs. The borehole was drilled through the Culebra and extends 9.7 ft into the

unnamed lower member of the Rustler Formation. The well was drilled to a depth of 367 ft bgs using compressed air as the drilling fluid. The interval from 367 to 616 ft bgs (the total depth) was drilled using brine as the drilling fluid. WQSP-6 was drilled to 568 ft bgs using a 9⅞-inch drill bit and was cored from 568 to 616 ft bgs using a 5¼-inch core bit to cut 4-inch-diameter core. After coring, WQSP-6 was reamed to 9⅞ inches in diameter to a total depth of 616.6 ft bgs. WQSP-6 was cased from the surface to 616 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen across the Culebra interval from 581 to 606 ft bgs. The annulus between the borehole wall and casing/screen is packed with sand from 567 to 570 ft bgs and with 8/16 Brady gravel from 570 to 616.6 ft bgs. Based on core log results, the Culebra is located from 582.0 to 606.9 ft bgs. See Figure 5-6.

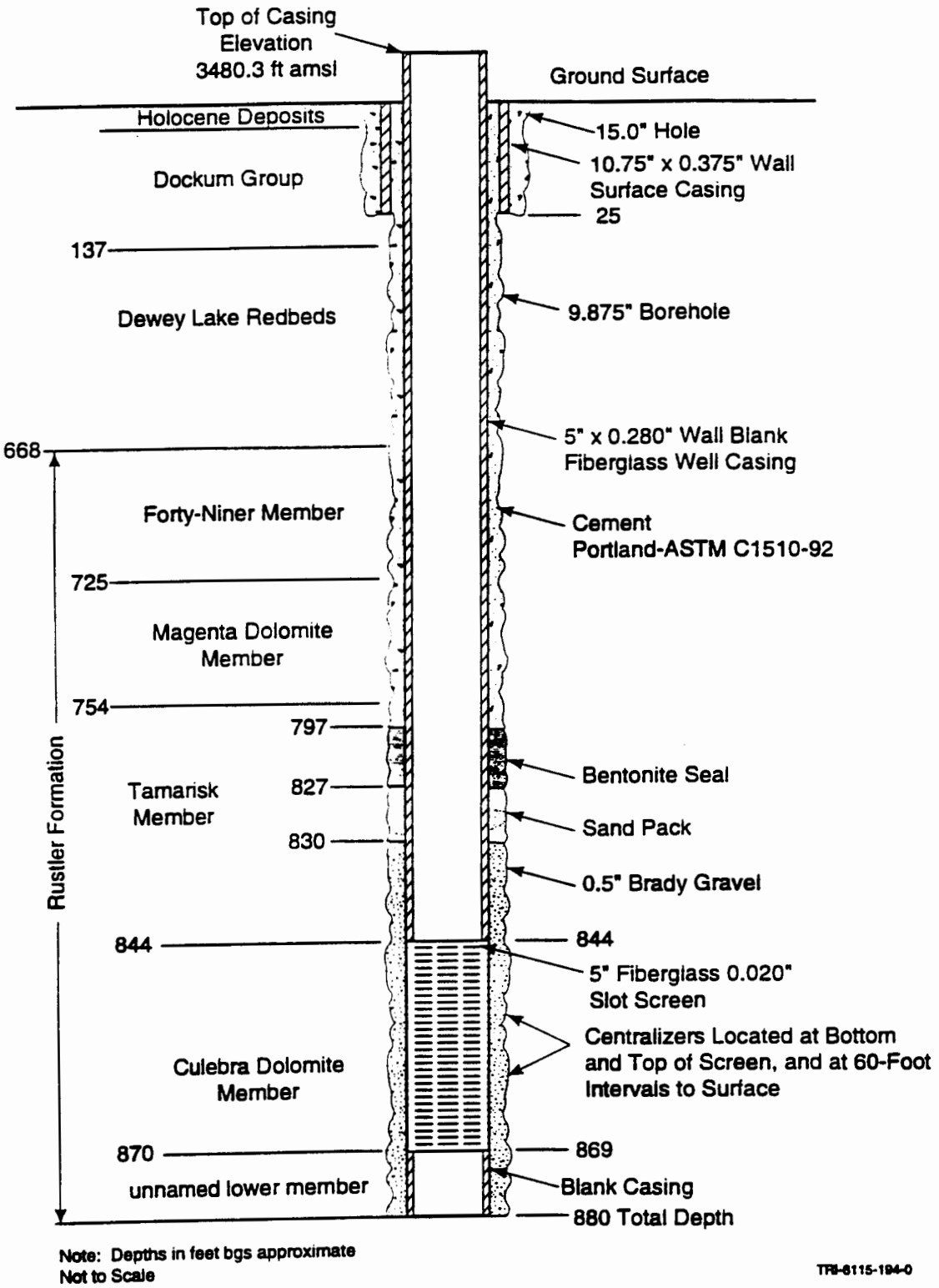
WQSP-6A - Well WQSP-6A was drilled between October 31 and November 1, 1994, to a total depth of 225.0 ft bgs. The borehole was drilled through a water-producing zone in the Dewey Lake Redbeds that had been previously encountered while drilling well WQSP-6. The well was drilled to a depth of 225 ft bgs using compressed air as the drilling fluid. The borehole was cleaned using air mist with a foaming agent. WQSP-6A was drilled to 160 ft bgs using a 9⅞-inch drill bit and was cored from 160.0 to 220.0 ft bgs using a 5¼-inch core bit to cut 4-inch-diameter core. After coring, WQSP-6A was reamed to 9⅞ inches in diameter to a total depth of 225 ft bgs. WQSP-6A was cased from the surface to 225 ft bgs with 5-inch (0.280-inch wall) blank fiberglass casing with in-line 5-inch-diameter fiberglass 0.020-inch slotted screen from 190 to 215 ft bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 172 to 175 ft bgs and with 8/16 Brady gravel from 175 to 225 ft bgs. See Figure 5-7.



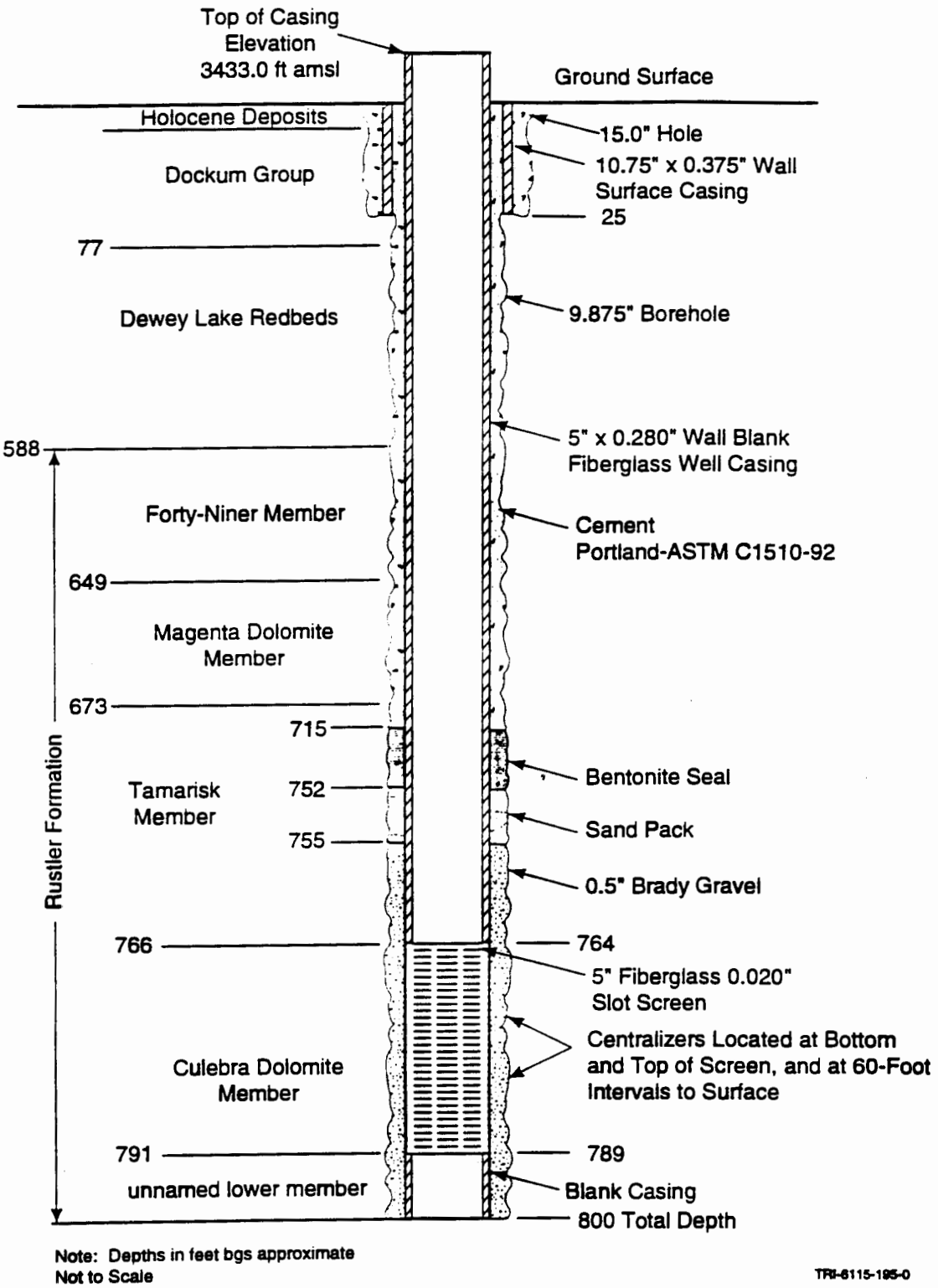
As-built configuration of well WQSP-1.



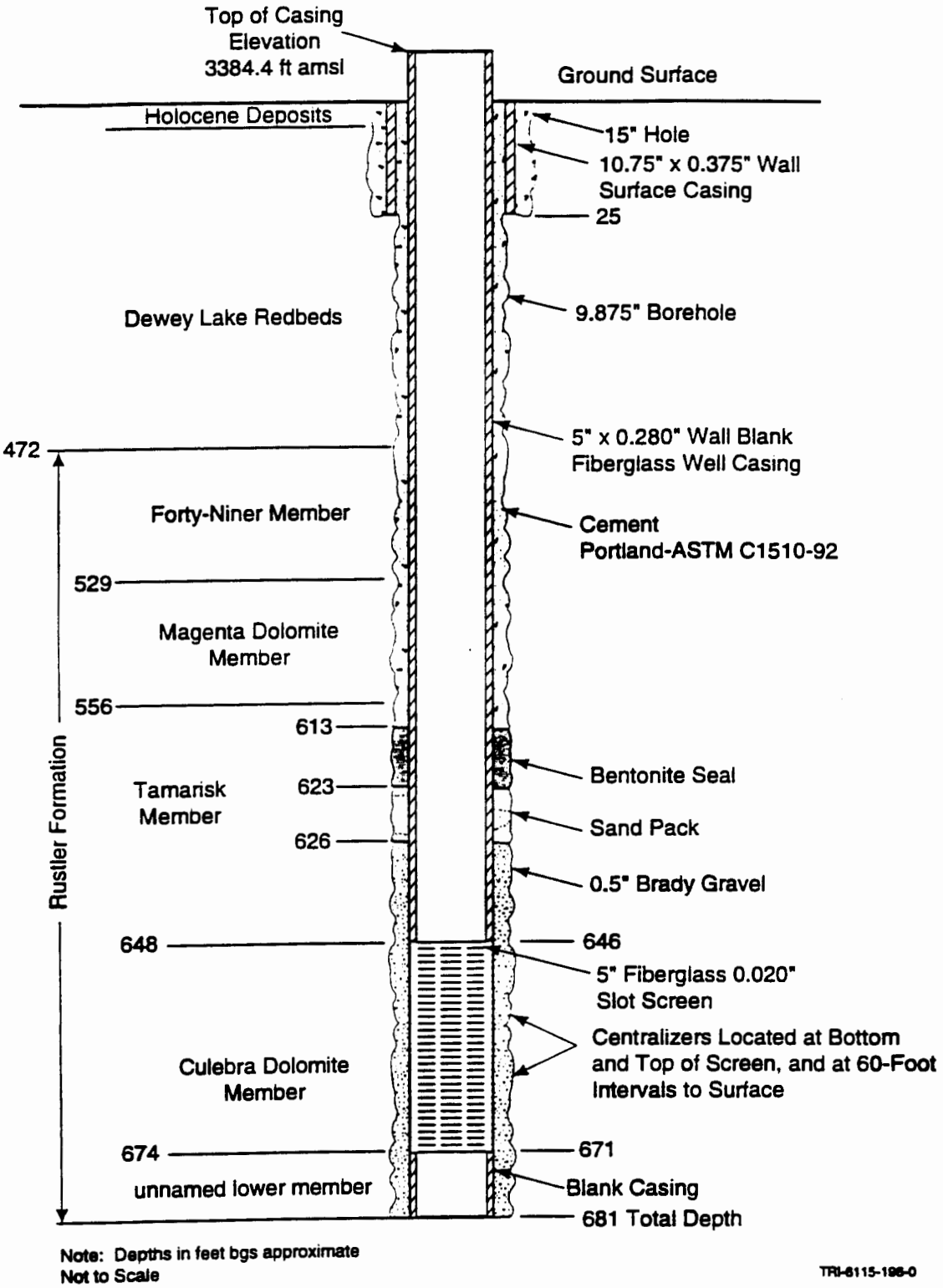
As-built configuration of well WQSP-2.



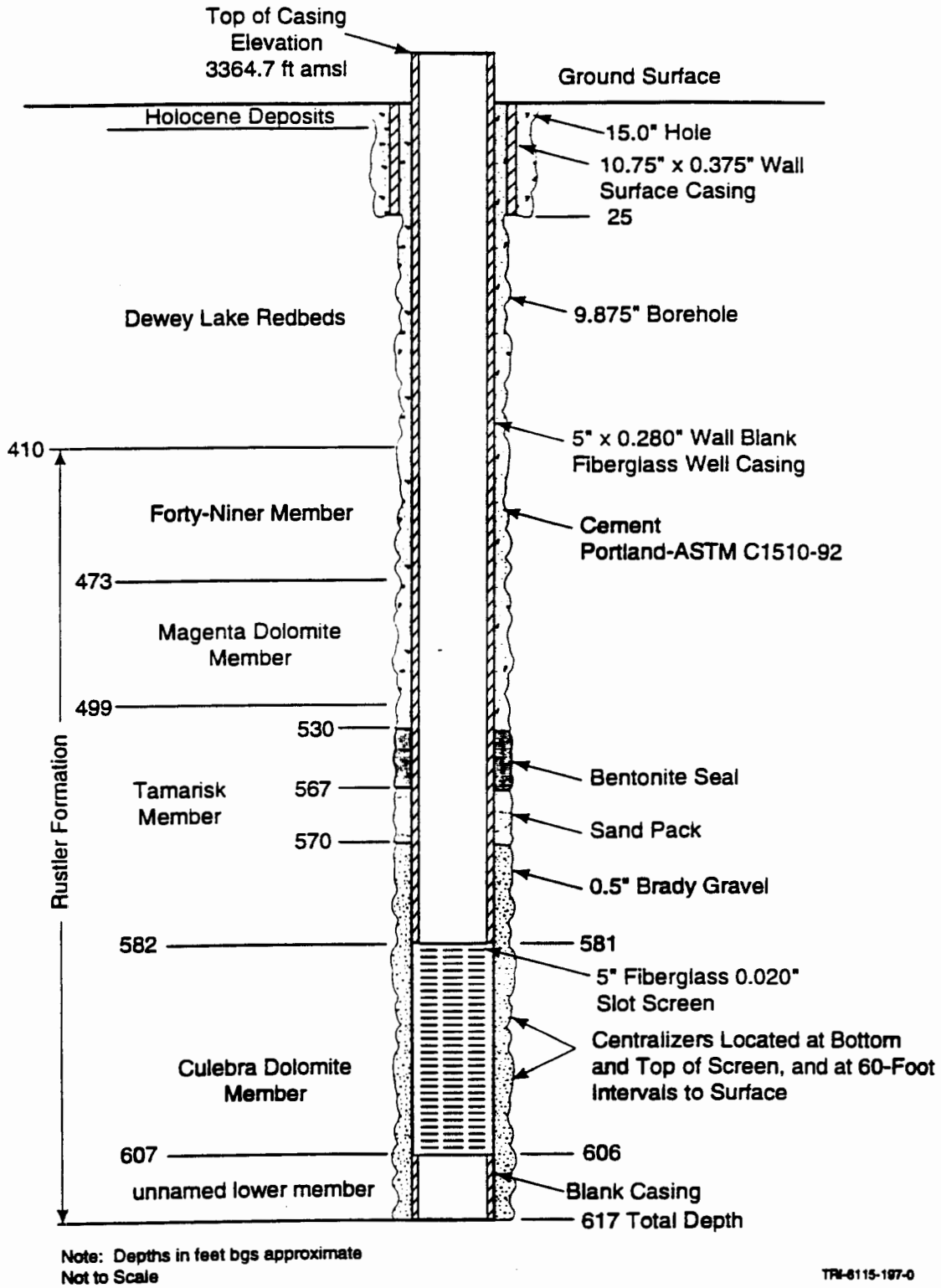
As-built configuration of well WQSP-3.



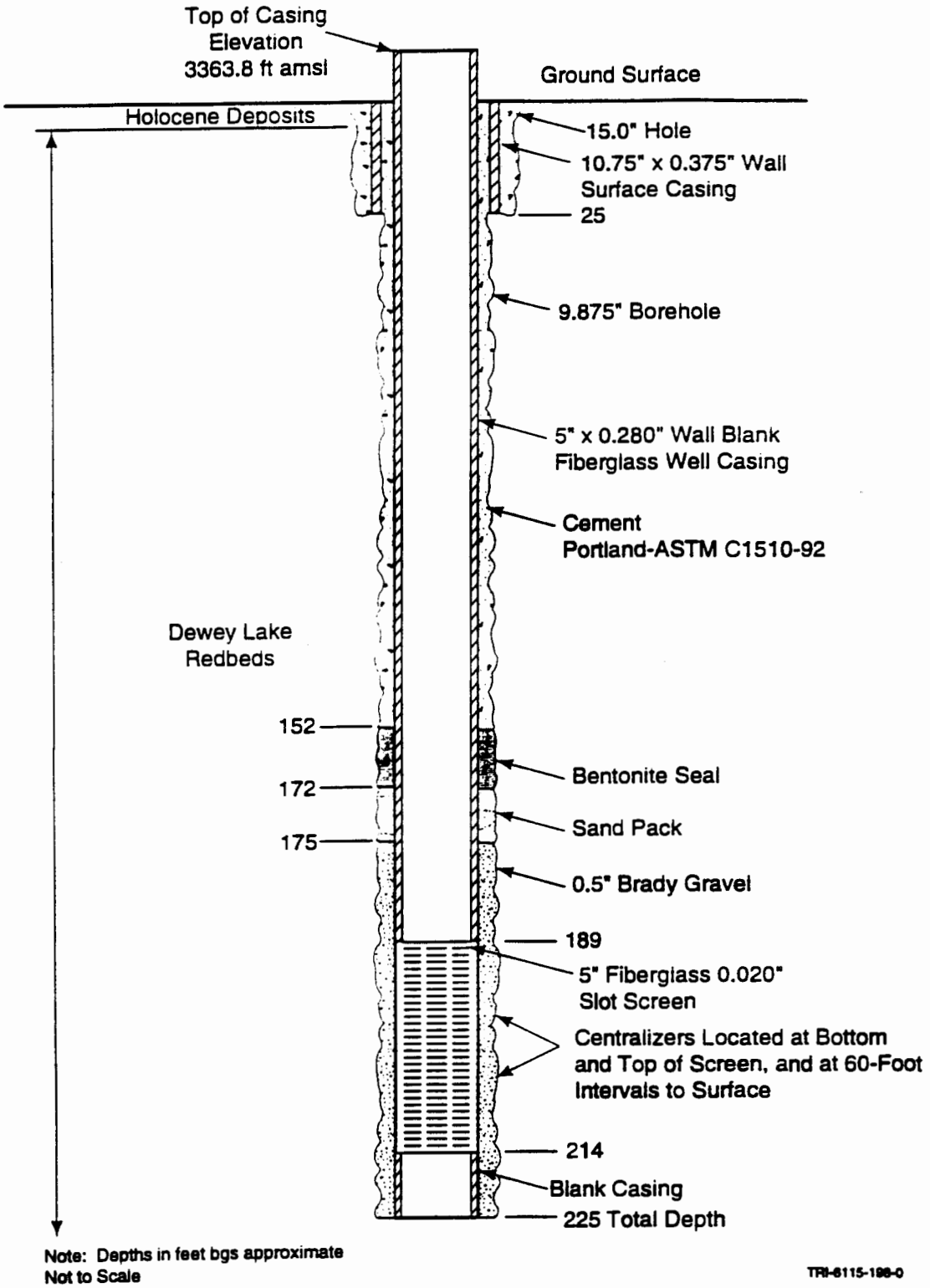
As-built configuration of well WQSP-4.



As-built configuration of well WQSP-5.



As-built configuration of well WQSP-6.



As-built configuration of well WQSP-6A.

Rev 6.2

APPENDIX D18
WIPP GROUNDWATER MONITORING PROGRAM PLAN

APPENDIX D18
WIPP GROUNDWATER MONITORING PROGRAM PLAN

TABLE OF CONTENTS

List of Tables D18-v

List of Figures D18-vii

List of Abbreviations/Acronyms D18-ix

D18-1 Introduction D18-1
D18-1a Site Location D18-1
D18-1b Purpose D18-2
D18-1c Geologic and Hydrologic Characteristics D18-2
D18-1c(1) Geology D18-2
D18-1c(2) Groundwater Hydrology D18-3

D18-2 General Regulatory Requirements D18-7

D18-3 WIPP Groundwater Monitoring Program—Overview D18-7
D18-3a Scope D18-7
D18-3b Historic WIPP WQSP D18-8
D18-3b(1) Background D18-8
D18-3b(2) Well Construction D18-8
D18-3c Current WIPP GWSP D18-9
D18-3d WIPP GMP D18-10
D18-3d(1) GMP Well Construction Specification D18-11

D18-4 Project Organization and Responsibilities D18-13
D18-4a Environmental Monitoring Manager D18-13
D18-4b Team Leader D18-14
D18-4c Field Team D18-14
D18-4d Safety Manager D18-14
D18-4e Analytical Laboratory D18-14
D18-4f Q&RA Manager D18-15

D18-5 QA Requirements D18-15
D18-5a QA Program—Overview D18-15
D18-5b DQOs D18-15
D18-5b(1) Accuracy D18-16
D18-5b(2) Precision D18-16

D18-5b(3) Contamination D18-17
D18-5b(4) Completeness D18-17
D18-5b(5) Representativeness D18-17
D18-5b(6) Comparability D18-18
D18-5c Design Control D18-18
D18-5d Instructions, Procedures, and Drawings D18-18
D18-5e Document Control D18-18
D18-5f Control of Processes D18-18
D18-5g Inspection and Surveillance D18-19
D18-5h Control of Monitoring and Data Collection Equipment D18-19
D18-5i Control of Nonconforming Conditions D18-19
D18-5j Corrective Action D18-19
D18-5k Quality Assurance Records D18-19
D18-6 Monitoring Program Description D18-20
D18-6a Monitor Well Locations D18-20
D18-6b Monitoring Frequency D18-21
D18-6c Analytical Parameters D18-21
D18-6d Sample Collection and Laboratory Analysis D18-22
D18-6d(1) Serial Samples D18-22
D18-6d(2) Final Samples D18-23
D18-6d(3) Laboratory Analysis D18-24
D18-6e Statistical Analysis of Laboratory Data D18-25
D18-6e(1) Temporal and Spatial Analysis D18-25
D18-6e(2) Distributions and Descriptive Statistics D18-26
D18-6e(3) Data Anomalies D18-26
D18-6e(4) Comparisons and Reporting D18-27

D18-7 Sample Documentation and Custody D18-27
D18-7a Sample Numbers and Labels D18-27
D18-7b Custody Seals D18-28
D18-7c Sample Tracking Logbook D18-28
D18-7d Request for Analysis and Chain of Custody D18-28

D18-8 Field Operations D18-28
D18-8a Water Quality Sampling Technique D18-29
D18-8a(1) Serial Sampling D18-29
D18-8a(2) Final Samples D18-29
D18-8a(3) Groundwater Pumping and Sampling Systems D18-30
D18-8a(4) Pressure Monitoring Systems D18-31
D18-8b Water-Level Monitoring Methodology D18-31
D18-8b(1) Scope D18-31

D18-8b(2) Introduction D18-32
D18-8b(3) Objective D18-32

D18-8b(4) Field Methods D18-34
D18-8b(5) Records and Document Control D18-34

D18-8b(6) Reporting D18-35
D18-8c Calibration D18-35
D18-8c(1) Sampling Equipment Calibration Requirements D18-35
D18-8c(2) Water-Level Monitoring Equipment Calibration Requirements D18-35
D18-8d Well Evacuation Procedures D18-35
D18-8e Sample Preservation, Tracking, Packaging and Transportation D18-35

D18-9 Reporting D18-36
D18-9a Laboratory Data Reports D18-36
D18-9b Statistical Analysis and Reporting of Results D18-36
D18-9c Annual Site Environmental Report D18-36

D18-10 Records Management D18-37

D18-11 References D18-38

Attachment A—WIPP Procedures for Sample Collection, Water-Level Monitoring, QA/QC, and Records Control
Attachment B—Contract Laboratory Operating Procedures
Attachment C—GMP Water Quality Data to Date

This page left blank intentionally

List of Tables

Table	*Title*
D18-1	Hydrologic Parameters for Rock Units Above the Salado at WIPP
D18-2	WIPP Groundwater Monitoring Program Sample Collection and Water-Level Measurement Frequency
D18-3	Analytical Parameter List for the WIPP Detection Monitoring Program

This page left blank intentionally

List of Figures

Figure	*Title*
D18-1	General Location of the WIPP Facility
D18-2	WIPP Facility Boundaries Showing 16-Square-Mile Land Withdrawal Boundary and Point of Compliance
D18-3	Site Geologic Column
D18-4	Generalized Stratigraphic Cross-Section Above Bell Canyon Formation at the WIPP Site
D18-5	Schematic North-South Cross-Section Through the North Delaware Basin
D18-6	Culebra Freshwater-Head Contour Surface
D18-7	Total Dissolved Solids Distribution in the Culebra
D18-8	WQSP Monitor Well Locations
D18-9	WIPP GMP Monitor Well Locations and Potentiometric Surface of the Culebra Dolomite Member of the Rustler Formation Near the WIPP Site as of 12/96 (Adjusted to Equivalent Freshwater Head)
D18-10	As-Built Configuration of Well WQSP 1
D18-11	As-Built Configuration of Well WQSP 2
D18-12	As-Built Configuration of Well WQSP 3
D18-13	As-Built Configuration of Well WQSP 4
D18-14	As-Built Configuration of Well WQSP 5
D18-15	As-Built Configuration of Well WQSP 6
D18-16	As-Built Configuration of Well WQSP 6A
D18-17a	Chain-of-Custody Record

LIST OF FIGURES *(CONTINUED)*

D18-17b **Request for Analysis**

D18-18 **Water-Level Monitoring Locations**

List of Abbreviations/Acronyms

ASER	Annual Site Environmental Report
Bell Canyon	Bell Canyon Formation
bgs	below ground surface
Castile	Castile Formation
cm	centimeter(s)
Culebra	Culebra member of the Rustler Formation
°C	degree(s) Celsius
DI	deionized
DOE	U.S. Department of Energy
DQO	data quality objectives
EM	Environmental Monitoring
EPA	U.S. Environmental Protection Agency
ES&H	Environmental Health and Safety Department
FEIS	Final Environmental Impact Statement
ft	foot (feet)
ft^2	square foot (feet)
GMP	Groundwater Monitoring Program
GWSP	Groundwater Surveillance Program
HASP	Health and Safety Plan
HWMU	hazardous waste management unit(s)
km	kilometer(s)
km^2	square kilometer(s)
$lb/in.^2$	pound(s) per square inch
LCS	laboratory control samples
LD	limit of detection
LWA	Land Withdrawal Act
m	meter(s)
M&DC	monitoring and data collection
m^2	square meter(s)
mg/L	milligram(s) per liter
mi	mile(s)
mi^2	square mile(s)
MPa	megapascal(s)
mV	millivolt(s)
NIST	National Institute for Standards and Technology
NMED	New Mexico Environment Department
PDSP	Pressure Density Survey Program
Q&RA	Quality and Regulatory Assurance
QA/QC	quality assurance/quality control
QAPD	Quality Assurance Program Description

LIST OF ABBREVIATIONS/ACRONYMS *(CONTINUED)*

RCRA	Resource Conservation and Recovery Act
RFA and CofC	request for analysis/chain of custody
RIDS	Records Inventory and Disposition Schedule
RPD	relative percent difference
Rustler	Rustler Formation
Salado	Salado Formation
SC	specific conductance
SNL	Sandia National Laboratories
SOP	Standard Operating Procedure
STLB	sample tracking logbook
TRU	transuranic
TSDF	treatment, storage, and disposal facilities
VOC	volatile organic compound
WID	Waste Isolation Division
WIPP	Waste Isolation Pilot Plant
WLMP	WIPP Groundwater Level Monitoring Program
WQSP	Water Quality Sampling Program
µg/L	microgram(s) per liter

APPENDIX D18
WIPP GROUNDWATER MONITORING PROGRAM PLAN

D18-1 Introduction

The Waste Isolation Pilot Plant (WIPP) project is a geologic repository for the disposal of transuranic (TRU) waste. The disposal horizon is located 2,150 feet (ft) (655.3 meters [m]) below the land surface in the bedded salt of the Salado Formation (hereinafter referred to as the Salado). At the WIPP, water-bearing units occur both above and below the disposal horizon. Groundwater monitoring of the uppermost aquifer below the facility is not proposed at the WIPP because that water-bearing unit (the Bell Canyon Formation) is not considered a credible pathway for a release from the repository. The underlying water-bearing sandstones of the Bell Canyon Formation are separated from the repository horizon by over 2000 ft (609.6 m) of very low-permeability evaporite sediments (Appendices E1 and D6). No credible pathway has been established for contaminant transport to aquifers below the repository horizon as there is no hydrologic communication between the two. The U.S. Environmental Protection Agency (EPA) concluded in 1990 that vertical communication does not exist based on their review of the numerous studies (EPA, 1990). The disposal horizon is isolated from underlying water-bearing units by extremely low-permeability layers of rock. Furthermore, drilling boreholes for groundwater monitoring through the Salado and the Castile Formation (hereinafter referred to as the Castile) into that aquifer would act to compromise the isolation properties of the repository medium and, therefore, is not allowed.

Disposal of transuranic (TRU) mixed waste in the WIPP facility is subject to regulation under Title 20 of the New Mexico Administrative Code, Chapter 4, Part 1 (20 NMAC 4.1), Subpart V. As required by 20 NMAC 4.1, Subpart V, §264.601, the U.S. Department of Energy (DOE) must demonstrate that the environmental performance standards for a miscellaneous unit, which are applied to the hazardous waste management units (HWMU) in the underground, will be met.

Groundwater monitoring at the WIPP in the past has focused on the Culebra member of the Rustler Formation (hereinafter referred to as the Culebra) because it represents the most significant hydrologic contaminant migration pathway to the accessible environment. The Culebra is the nearest and most significant water-bearing unit lying above the repository. Modeling of groundwater movement in the Culebra, based on the concept of a groundwater basin, is discussed in detail in Appendix D6, Section D6-2a(1).

D18-1a Site Location

The WIPP site is located in Eddy County in southeastern New Mexico (Figure D18-1) within the Pecos Valley section of the southern Great Plains physiographic province (Powers et al., 1978). The site is 26 miles (mi) (42 kilometers [km]) southeast of Carlsbad in an area known as Los Medaños (the dunes). Los Medaños is a relatively flat, sparsely inhabited plateau with little water and limited land uses.

The WIPP site (Figure D18-2) consists of 16 sections of federal land in Township 22 South, Range 31 East. The 16 sections of federal land were withdrawn from the application of public land laws by the WIPP Land Withdrawal Act (LWA), Public Law 102-579, that was signed on October 30, 1992. The WIPP LWA transferred the responsibility for the administration of the 16 sections from the Department of Interior, Bureau of

Land Management, to the DOE. This law specified that mining and drilling for purposes other than support of the WIPP project are prohibited within this 16 section area with the exception of Section 31. Oil and gas drilling activities are restricted in Section 31 from the surface down to 6,000 feet.

D18-1b Purpose

This monitoring plan provides instructions for sample collection, water-level monitoring, data management, and reporting of annual data; identifies analytical parameters selected for assessing the quality of the groundwater; and establishes personnel responsibilities for the WIPP groundwater monitoring program. Because quality assurance is an integral component of the groundwater sampling, analysis, and reporting process, quality assurance/quality control (QA/QC) elements and associated data acceptance criteria are included in this plan.

Detailed instruction for performing field activities that will be conducted in conjunction with this sampling and analysis plan are provided in field operating procedures, referenced in Sections D18-6 and D18-8 and included as Attachment A to this plan. Detailed procedures are provided for each aspect of the groundwater sampling process, including water-level measurement, sampling equipment decontamination and installation, purge requirements, field water-quality measurements, and sample collection. These procedures are not intended to replace proper field sampling expertise. Samples will be collected by trained personnel under the supervision and direction of qualified engineers, scientists, or other technical personnel.

D18-1c Geologic and Hydrologic Characteristics

D18-1c(1) Geology

The WIPP site is situated within the Delaware Basin, which is part of the larger Permian Basin, located in the south-central region of North America. During the Permian period, which came to a close about 245 million years ago, ancient seas covered the basin. Their later evaporation resulted in the deposition of a thick sequence of evaporites. Appendix D6 presents a detailed discussion of the regional geologic history. Three major evaporite-bearing formations were deposited in the Delaware Basin (see Figures D18-3 and D18-4):

- The Castile, which formed through evaporation of the Permian Sea, consists of interbedded anhydrites and halite. Its upper boundary is at a depth of about 2,825 ft (861 m) below ground surface (bgs), and its thickness at the WIPP facility is 1,250 ft (381 m) (see Appendix D6).

- The repository is located in the Salado, which overlies the Castile and resulted from prolonged desiccation that produced predominantly halite, with some carbonates, anhydrites, and clay seams. Its upper boundary is at a depth of about 850 ft (259 m) bgs, and it is about 2,000 ft (610 m) thick in the repository area (see Appendix D6).

- The Rustler Formation (hereinafter referred to as the Rustler) was deposited in a lagoonal environment during a major freshening of the basin and consists of carbonates, anhydrites, and halites. Its beds consist of clay and anhydrite and contain small amounts of brine.

The Rustler's upper boundary is about 500 ft (152 m) bgs, and it ranges up to 350 ft (107 m) in thickness in the area (see Appendix D6).

These evaporite-bearing formations lie between two other formations significant to the geology and hydrology of the WIPP site. The Dewey Lake overlying the Rustler is dominated by nonmarine sediments and consists almost entirely of mudstone, claystone, siltstone, and interbedded sandstone (Appendix D6). This formation forms a 500-ft- (152-m) thick barrier of fine-grained sediments that retard the downward percolation of water into the evaporite units below.[1] The Bell Canyon Formation (hereinafter referred to as the Bell Canyon)—the first water-bearing unit below the repository (Appendix D6)—is confined by the thick evaporite sequences of the Castile above. It consists of 1,200 ft (366 m) of interbedded sandstone, shale, and siltstone.

The Salado was selected to host the WIPP repository for several reasons. First, it is regionally extensive, underlying an area of more than 36,000 square mi (mi^2) (93,240 square kilometers [km^2]). Second, its permeability is extremely low, and fluids within it are effectively immobile. Third, salt behaves mechanically in a plastic manner under pressure (the pressure at the disposal horizon is more than 2,000 pounds per square inch [$lb/in.^2$] or 13.8 megapascals [MPa]) and moves to fill any opening (referred to as creep). Fourth, any fluid remaining in small fractures or openings is saturated with salt, is incapable of further salt dissolution, and has remained in place for millions of years. Finally, the Salado lies between the Rustler and the Castile (Figure D18-5), which contain highly impermeable layers that further confine and isolate waste within and isolate water outside the WIPP repository (Appendix D6).

D18-1c(2) Groundwater Hydrology

The Salado was selected as the host geologic unit because it is hydrologically isolated and because of the plastic nature of the salt. The general hydrogeology of the area surrounding the WIPP facility is described in this section starting with the first geologic unit below the Salado. Appendix D6 provides more detailed discussions of the local and regional hydrogeology. Relevant hydrological parameters for the various rock units above the Salado at the WIPP are summarized in Table D18-1.

The Castile

The Castile is a basin-filling evaporite sequence of sediments surrounded by the Capitan Reef. The Castile represents a major regional groundwater aquiclude that effectively prevents upward migration of water from the underlying Bell Canyon. Fluid present in the Castile is very restricted because evaporites do not readily maintain pore space, solution channels, or open fractures at depth. Drill-stem tests conducted in the Castile during construction of the WIPP facility found its permeability to be lower than detection limits; however, the hydraulic conductivity has been conservatively estimated to be less than 10^{-8} ft (3×10^{-9} m) per day. A description of the Castile brine reservoirs outside the WIPP area is provided in Appendix D6.

---

[1]While there may be some uncertainty over the amount of vertical recharge occurring within the Rustler, the issue is only of significance to long-term performance calculations in which releases from the repository occur through the creation of a migration pathway resulting from drilling (inadvertently) in the WIPP area. The consequences of vertical recharge are bounded in the modeling by assuming that under future climate conditions (which are assumed to be cooler and wetter), the groundwater level (water table) moves to the land surface.

The Salado

The Salado is an evaporite sequence that filled the remainder of the Delaware Basin and lapped extensively over the Capitan Reef and the back-reef sediments beyond. The Salado consists of approximately 2,000 ft (610 m) of bedded halite, with interbeds or seams of anhydrite, clay, and polyhalite. It acts hydrologically as a regional confining bed and does not contain circulating fluids. The porosity of the Salado is very low and interconnected pores are virtually nonexistent at the depth of the disposal horizon. Fluids associated with the Salado occur mainly as very small fluid inclusions in the halite crystals and also occur between crystal boundaries (interstitial fluid) of the massive crystalline salt formation; fluids also occur in clay seams and anhydrite beds. Permeabilities measured from the surface in the area of the WIPP facility range from 0.01 to 25 microdarcies. The most reliable value, 0.3 microdarcy, was obtained from well DOE-2. The results of permeability testing at the disposal horizon are within the range of 0.001 to 0.01 microdarcy. As a comparison, the permeability of the Salado is roughly a thousand times more restrictive than that of the lower clay liner required of surface impoundments and landfills, assuming similar thicknesses. The results of recent testing suggest that permeabilities in undisturbed rock salt may approach zero (see Appendix D6).

The Rustler

The Rustler has been the subject of extensive characterization activities because it contains the most transmissive hydrologic units overlying the Salado (specifically the Culebra). Within the Rustler, five members have been identified. Of these the Culebra is the most transmissive and has been the focus of most of the Rustler hydrologic studies (see Appendix D6).

The Culebra is the first continuous water-bearing zone above the Salado and ranges up to 30 ft (9 m) in thickness. Water in the Culebra is usually present in fractures and is confined by overlying gypsum or anhydrite and underlying clay and anhydrite beds. Its hydraulic gradient in the area of the WIPP facility is approximately 20 ft per mi (6.1 m per km) and becomes much flatter south and southwest of the site (Figure D18-6). Transmissivities in the Nash Draw range up to 1,250 square ft (ft^2) (116 square m [m^2]) per day; closer to the WIPP facility, they are as low as 0.007 to 74 ft^2 (0.00065 to 7.0 m^2) per day. The Culebra is hydrologically confined and testing indicates no significant leakage between it and other units (see Appendix D6).

The two primary types of field tests that are being used to characterize the flow and transport characteristics of the Culebra are hydraulic tests and tracer tests.

The hydraulic testing consists of pumping, injection, and slug testing of wells across the study area (e.g., Beauheim 1987a). The most detailed hydraulic test data exist for the WIPP hydropads (e.g., H-19). The hydropads generally comprise a network of three or more wells located within a few tens of meters of each other. Long-term pumping tests have been conducted at hydropads H-3, H-11, and H-19 and at well WIPP-13 (Beauheim 1987b, 1987c; Beauheim et al. 1995). These pumping tests provided transient pressure data at the hydropad and over a much larger area. Tests often included use of automated data-acquisition systems, providing high-resolution (in both space and time) data sets. In addition to long-term pumping tests, slug tests and short-term pumping tests have been conducted at individual wells to provide pressure data that can be used to interpret the transmissivity at that well (Beauheim 1987a). (Additional short-term pumping tests have been conducted in the WQSP wells [Stensrud 1995]). Detailed cross-hole hydraulic testing has recently been conducted at the H-19 hydropad (Kloska et al. 1995).

The hydraulic tests are designed to yield pressure data for the interpretation of such characteristics as transmissivity, permeability, and storativity. The pressure data from long-term pumping tests and the interpreted transmissivity values for individual wells are used for the generation of transmissivity fields in flow modeling. Some of the hydraulic test data and interpretations are also important for the interpretation of transport characteristics. For instance, the permeability values interpreted from the hydraulic tests at a given hydropad are needed for interpretations of tracer test data at that hydropad.

There is strong evidence that the permeability of the Culebra varies spatially and varies sufficiently that it cannot be characterized with a uniform value or range over the region of interest to the WIPP. The transmissivity of the Culebra varies spatially over six orders of magnitude from east to west in the vicinity of the WIPP (see Figure D6-30). Over the site, Culebra transmissivity varies over three to four orders of magnitude. Figure D6-30 shows variation in transmissivity in the Culebra in the WIPP region. Transmissivities are from 1×10^{-3} square feet per day (1×10^{-9} square meters per second) at well P-18 east of the WIPP site to 1×10^{3} square feet per day (1×10^{-3} square meters per second) at well H-7 in Nash Draw.

Transmissivity variations in the Culebra are believed to be controlled by the relative abundance of open fractures rather than by primary (that is, depositional) features of the unit. Lateral variations in depositional environments were small within the mapped region, and primary features of the Culebra show little map-scale spatial variability, according to Holt and Powers 1988. Direct measurements of the density of open fractures are not available from core samples because of incomplete recovery and fracturing during drilling, but observation of the relatively unfractured exposures in the WIPP shafts suggests that the density of open fractures in the Culebra decreases to the east. Qualitative correlations have been noted between transmissivity and several geologic features possibly related to open-fracture density, including (1) the distribution of overburden above the Culebra, (2) the distribution of halite in other members of the Rustler, (3) the dissolution of halite in the upper portion of the Salado, and (4) the distribution of gypsum fillings in fractures in the Culebra.

Measured matrix porosities of the Culebra vary from 0.03 to 0.30. Fracture porosity values have not been measured directly, but interpreted values from tracer tests at the H-3, H-6, and H-11 hydropads vary from 5×10^{-4} to 3×10^{-3}. Data are insufficient to determine whether the average porosity of the matrix and fractures varies significantly on a regional scale.

Geochemical and radioisotope characteristics of the Culebra have been studied. There is considerable variation in groundwater geochemistry in the Culebra. The variation has been described in terms of different hydrogeochemical facies that can be mapped in the Culebra. A halite-rich hydrogeochemical facies exists in the region of the WIPP site and to the east, approximately corresponding to the regions in which halite exists in units above and below the Culebra, and in which a large portion of the Culebra fractures are gypsum filled. An anhydrite-rich hydrogeochemical facies exists west and south of the WIPP site, where there is relatively less halite in adjacent strata and where there are fewer gypsum-filled fractures. Radiogenic isotopic signatures suggest that the age of the groundwater in the Culebra is on the order of 10,000 years or more (see, for example, Lambert 1987, Lambert and Carter 1987, and Lambert and Harvey 1987).

The radiogenic ages of the Culebra groundwater and the geochemical differences provide information potentially relevant to the groundwater flow directions and groundwater interaction with other units and are important constraints on conceptual models of groundwater flow. Previous conceptual models of the

Culebra (see for example, Chapman 1986, Chapman 1988, LaVenue et al. 1990) have not been able to consistently relate the hydrogeochemical facies, radiogenic ages, and flow constraints (that is, transmissivity, boundary conditions, etc.) in the Culebra.

The groundwater basin modeling that has been conducted, although it did not model solute transport processes, provides flow fields that reasonably explain observed hydrogeochemical facies and radiogenic ages. The groundwater basin model combines and tests three fundamental processes: (1) it calculates vertical leakage, which may carry solutes into the Culebra; (2) it calculates lateral fluxes in the Culebra (directions as well as rates); and (3) it calculates a range of possible effects of climate change. The presence of the halite facies is explained by vertical leakage of solutes into the Culebra from the overlying halite-containing Tamarisk by advective or diffusive processes. Because lateral flow rates here are low, even slow rates of solute transport into the Culebra can result in high solute concentration. Vertical leakage occurs slowly over the entire model region, and thus the age of groundwater in the Culebra is old, consistent with radiogenic information. Lateral fluxes within the anhydrite zone are larger because of higher transmissivity, and where the halite and anhydrite facies regions converge, the halite facies signature is lost by dilution with relatively large quantities of anhydrite facies groundwater.

Groundwater levels in the Culebra in the WIPP region have been measured continuously for several decades. Water-level rises have been observed in the WIPP region and are attributed to three causes as discussed below. The extent of water-level rise observed at a particular well depends on several factors, but the proximity of the observation point to the cause of the water-level rise appears to be a primary factor.

In the vicinity of the WIPP site, water-level rises are unquestionably caused by recovery from drainage into the shafts. Drainage into shafts has been reduced by a number of grouting programs over the years, most recently in 1993 around the AIS. Northwest of the site, in and near Nash Draw, water levels appear to fluctuate in response to effluent discharge from potash mines. Correlation of water-level fluctuation with potash mine discharge cannot be proven because sufficient data on the timing and volumes of discharge are not available. Head distribution in the Culebra (see Figure D6-31) is consistent with groundwater basin modeling results indicating that the generalized directional flow of groundwater is north to south. However, caution should be used when making assumptions based on groundwater-level data alone. Studies in the Culebra have shown that fluid density variations in the Culebra can affect flow direction. One should also be aware that the fractured nature of the Culebra, coupled with variable fluid densities, can also cause localized flow patterns to differ from general flow patterns. Water-level rises in the vicinity of the H-9 hydropad, about 6.5 miles south of the site, are thought to be caused by either WIPP activities or potash mining discharge. They remain unexplained. The DOE continues to monitor groundwater levels throughout the region, but only water level changes at or near the site have the potential to affect performance.

Inferences about vertical flow directions in the Culebra have been made from well data collected by the DOE. Beauheim (1987a) reported flow directions towards the Culebra from both the unnamed lower member and the Magenta over the WIPP site, indicating that the Culebra acts as a drain for the units around it. This indication is consistent with results of groundwater basin modeling. Recent simulations to enhance the conceptual understanding of the geohydrology of the Rustler can be found in Corbet and Knupp 1996.

Use of water from the Culebra is quite limited because of its varying yields and high salinity. The Culebra is not used for water supply in the immediate WIPP site vicinity. Its nearest use is approximately 7 mi (11 km) southwest of the WIPP facility, where salinity is low enough to allow its use for livestock watering (shown, for example, as Well H-8 in Figure D18-7). Appendix D6 provides a detailed discussion of the hydrologic characteristics of the other Rustler members and the Dewey Lake, which lie above the Rustler. The Culebra will be the focus of future groundwater monitoring at WIPP because it is the most transmissive continuous water-bearing zone at WIPP and is the most likely pathway for contaminant migration.

D18-2 General Regulatory Requirements

Because geologic repositories such as the WIPP facility are defined under the Resource Conservation and Recovery Act (RCRA) as land disposal facilities and as miscellaneous units, the groundwater monitoring requirements of 20 NMAC 4.1, Subpart V, §§264.600 through 264.603, must be addressed. 20 NMAC 4.1, Subpart V, §§264.90 through 264.101, applies to miscellaneous unit treatment, storage, and disposal facilities (TSDF) only if groundwater monitoring is needed to satisfy 20 NMAC 4.1, Subpart V, §§264.601 through 264.603, environmental performance standards.

The New Mexico Environment Department (NMED) has concluded that groundwater monitoring in accordance with 20 NMAC 4.1, Subpart V, §264.90 at the WIPP is necessary to meet the requirements of 20 NMAC 4.1, Subpart V, §§264.601 through 264.603.

D18-3 WIPP Groundwater Monitoring Program—Overview

D18-3a Scope

The DOE has established this "Groundwater Monitoring Program (GMP) Plan" to define and protect groundwater resources at the WIPP. In part, the purpose of the GMP is to determine background or existing conditions of groundwater quality and quantity around the WIPP facility area, and to maintain a groundwater quality database. The policy driver for this program is 20 NMAC 4.1, Subpart V, §§264.600 to 264.603, which requires monitoring for miscellaneous units. The format adopted for this plan is 20 NMAC 4.1, Subpart V, §§264.00 through 264.101, which provides instruction on the monitoring of groundwater that is or could be affected by waste management activities at WIPP. DOE Order 5400.1 (IV-10[c]) also instructs that "where appropriate, groundwater monitoring programs shall be designed and implemented in accordance with 40 CFR Part 264 Subpart F or 40 CFR 265 Subpart F."

One of the objectives of the WIPP groundwater monitoring program is to establish, by means of groundwater sampling and analysis events, an accurate and representative groundwater database that is scientifically defensible and supports regulatory compliance. This plan governs all groundwater sampling events and ensures that all such data are gathered in accordance with 20 NMAC 4.1, Subpart V, §§264.90 through 264.101 and other applicable requirements. The groundwater quality data generated by monitoring activities will provide a comprehensive background database against which future analytical results can be compared during the detection monitoring program.

Groundwater monitoring at WIPP has been historically conducted by several programs including the WIPP Site Characterization Program, the WIPP Water Quality Sampling Program (WQSP), and recently the WIPP Groundwater Surveillance Program (GWSP). Groundwater quality and water-level data have been collected

by these programs for over 12 years at the WIPP. The WQSP and WIPP GWSP are briefly described below to provide historical information. Data from the WQSP wells (which are widely distributed across the area) will be used to continually define changes in the area's potentiometric surface and groundwater flow directions. The new monitoring wells of the WIPP GWSP have been constructed to the specifications provided in the RCRA Ground-Water Monitoring Technical Enforcement Guidance Document (EPA, 1986a) and are now being used to establish background water quality and water levels in accordance with 20 NMAC 4.1, Subpart V, §264.94(b)(4). These WIPP GWSP wells will be used as the monitoring installations for the WIPP Detection Groundwater Monitoring Program as required by 20 NMAC 4.1, Subpart V, §§264.90 through 264.101. Justification for the locations of these wells (3 upgradient and 4 downgradient) is presented subsequently.

D18-3b Historic WIPP WQSP

The WQSP was first initiated in January 1985. The objective of the program was to collect representative and reproducible groundwater samples from water-bearing zones in the area of the WIPP site. The purpose of the program was to provide defensible data for meeting the requirements of site characterization and provide initial background water-quality data to be used as a foundation for meeting future regulatory compliance and permitting criteria.

D18-3b(1) Background

From 1985 to 1990, the WQSP characterized the physical and chemical characteristics of representative groundwater samples occurring within and immediately surrounding the WIPP site. Various wells were serially sampled three times to determine the representative character of the groundwater present at each location. Data collected were supplied to the Environment, Safety and Health Department and used to develop a baseline of water-quality data as part of the Radiological Baseline Program. A nonradiological database was developed to support the background water quality characterization report (Appendix D17). Data were also supplied to and used by Sandia National Laboratories (SNL) for site characterization.

During the initial phase of the WQSP, data were collected from existing wells that were drilled as part of the geologic site characterization and resource evaluation programs and made available to the WQSP. Figure D18-8 shows the monitoring locations used by the WQSP in the past.

D18-3b(2) Well Construction

The bulk of these wells were drilled between 1976 and 1980 using standard oil field drilling technology. Standard oil field steel casing either K-55 or J-55 were used in well construction. Surveillance wells were completed using two primary methods.

The first installation method required drilling the well to some depth below the zone of interest and then casing the well to the bottom of the hole. The zone of interest was then perforated using either shot or knife perforation methods.

The second installation method required drilling and casing the well to some point above the zone of interest then reentering the well bore and core drilling it through the zone of interest and leaving it open to the formation.

These types of well completions presented numerous problems in collecting useful data. The open hole completions resulted in halite rich sediments of lower formations being exposed to the sampling zone in some cases. In most cases the sampled zone collapsed from the stress of numerous pumping events filling in the interval with debris to the bottom of the cased portion of the well.

The casings soon began to corrode after exposure to the brackish-to-brine water found in the WIPP area. Based on sampling results, it appeared that products of well casing corrosion migrated from the well bore into the formation resulting in a halo or plume of groundwater altering the chemistry immediately surrounding the surveillance wells. Obtaining usable data required the use of packers to isolate the sampled formation from contaminated water standing in the casing above the formation and took unusually long periods of time to purge the well prior to obtaining representative samples.

D18-3c Current WIPP GWSP

After careful evaluation of the data gathered for background reports it became apparent that for compliance and permitting purposes, a system of new wells designed specifically for the purpose of collecting water-quality data needed to be installed.

Based on water-level surface elevation data (Richey, 1987) (Stensrud et al., 1988) and pressure density survey data (Crawley, 1988) it was recognized that spatial, variable densities of groundwater presented misleading flow gradients when potentiometric maps were produced based on surface elevations alone.

Davies (1989) identified multiple flow paths extending across the WIPP site based on a groundwater flow model using groundwater surface elevation data adjusted arithmetically to freshwater equivalent heads. This method eliminated the bias created by spatial variability of groundwater density. The new surveillance program wells were sited assuming the WIPP shafts to be the release point and based on the hydrologic characteristics presented by these programs.

Surveillance program wells were sited following the general guidance in the EPA Groundwater Monitoring Technical Enforcement Guidance Document and the groundwater flow characteristics at the site. Wells WQSP-1, WQSP-2, and WQSP-3 were located directly upgradient of the WIPP shaft area. The locations of the three upgradient wells were selected to be representative of the flow vectors of groundwater moving downgradient onto the WIPP site. Figure 34 of Davies, 1989, shows the simulation of direction and magnitude of groundwater flow. The upgradient wells were located based on the flow vectors resulting from this model simulation.

WQSP-4, WQSP-5, and WQSP-6 were located downgradient of the WIPP shaft area in concert with the flow vectors shown by this model simulation. All three downgradient wells (WQSP-4, 5, and 6) were sited based on the greatest velocity magnitude of groundwater flow leaving the shaft area as shown on Figure 34 of Davies, 1989, and upgradient of the WIPP groundwater monitoring compliance point. WQSP-4 was also specifically located to monitor the zone of higher transmissivity around wells DOE-1 and H-11, which may represent faster flow path away from the WIPP shaft area to the compliance point (DOE 1996).

After careful consideration given to the construction materials available, the decision was made that epoxy resin fiberglass casing afforded the most resistance to the corrosive waters found in the vicinity of WIPP.

Based on the flow paths identified by Davies (1989) and research conducted for construction materials, a proposal was written that addressed the following:

1. Monitor well siting
2. Construction practices and
3. Well construction materials.

The proposal was presented to members of Westinghouse Waste Isolation Division (WID), DOE, Environmental Evaluation Group, EPA Region 6, NMED and SNL in May of 1994 and a consensual agreement was reached among the attendees regarding each of the three issues presented, including monitor well location, monitoring network configuration, and hydrologic unit to be monitored.

Construction of the WIPP GWSP wells began in September of 1995 and was completed in November 1995. Sampling of the WIPP GWSP wells to establish the water quality background began in the spring of 1996.

The original WQSP observation wells have been and will continue to be used as piezometer wells to support collection of groundwater levels and groundwater flow modeling data to support regulatory compliance and permitting activities. Groundwater level monitoring data also are used to provide:

- Data required by the WIPP Environmental Monitoring Plan
- A means to fulfill commitments made in the Final Environmental Impact Statement (FEIS)
- A means to comply with future groundwater inventory and monitoring regulations
- Input for making land use decisions (i.e., designing long-term active and passive institutional controls for the WIPP)
- Assistance in understanding whether or not the horizontal and vertical gradients of the regional groundwater flow systems are changing over time.

Current flow patterns in the Culebra suggest the configuration of the WIPP GWSP system wells will provide sound, defensible data throughout the detection monitoring period. The new wells constructed for the WIPP GWSP (Figure D18-9) will be used as the monitoring network during operational detection monitoring as required by 20 NMAC 4.1, Subpart V, §§264.90 through 264.101.

D18-3d <u>WIPP GMP</u>

The WIPP GMP, for which this plan has been written, is a continuation of the current WIPP GWSP. The monitoring installations used during recent groundwater surveillance (WQSP 1–6A) will serve as the monitoring location during background water-quality characterization and the detection monitoring program (Figure D18-9).

The Culebra has been selected for the focus of the GMP due to it being regionally extensive and exhibiting the most significant transmissivity of the water-bearing units at WIPP. The Culebra has been extensively

studied during all past hydrologic characterization programs and found to be the most likely hydrologic pathway to the accessible environment or compliance point for any potential contamination.

At WIPP, the compliance point has been defined as the edge of the dual units 16 mi^2 land withdrawal boundary (Figure D18-9). This boundary represents the location where the nearest potential human receptor can reasonably be expected to be chronically exposed to potential contaminants.

The GMP wells were located to intercept all likely downgradient flow vectors away from the WIPP shafts area based on current density corrected potentiometric surfaces (Figure D18-9). The monitoring wells were also located to specifically allow early detection of contaminant migration by being installed upgradient of the compliance point. Based on natural contours of the potentiometric surface (Figure D18-9) the selected well placement locations are directly downgradient of the general flow direction from the shaft area. Transport modeling of contaminant migration throughout the Culebra to the compliance point has shown that travel times are on the order of thousands of years if, under worst case conditions, hazardous constituents could migrate from the sealed repository (see Section D18-9b[1][c] for repository modeling details). This time frame is well in excess of the regulatory compliance requirements of postclosure care. If contaminants were to migrate from the disposal facility, they would be detected by the GMP wells long before they could reach the facility compliance point.

Potentiometric surfaces and groundwater flow directions defined prior to large-scale pumping in the WIPP area and the excavation of WIPP shafts suggests that flow was generally to the south-southeast from the waste disposal and shaft areas (Mercer, 1983; Davies, 1989). Recent (December 1996) potentiometric surface maps of the Culebra adjusted for density differences show very similar characteristics (Figure D18-9). The GMP wells have been located directly downgradient of the waste emplacement areas according to present-day adjusted potentiometric surfaces.

Potentiometric surfaces that have not been corrected for density differences and that contain transient relics of previous pumping-drawdown events do not reflect accurate natural groundwater flow directions and should not be used to assess the adequacy of groundwater monitoring locations. Previous potentiometric surface maps showing a potentiometric low and hydrologic gradient toward the area between WQSP-3 and WQSP-4 had not been adjusted to freshwater head equivalents, and had also been influenced by the long-term pumping at well H-19, and do not represent natural Culebra flow directions or gradients.

D18-3d(1) <u>GMP Well Construction Specification</u>

<u>WQSP-1</u>—Well WQSP-1 was drilled between September 13 and 16, 1994, to a total depth of 737 ft (225 m) bgs. The borehole was drilled through the Culebra and extends 15 ft (5 m) into the unnamed lower member of the Rustler. The well was drilled to a depth of 693 ft (211 m) bgs using compressed air as the drilling fluid. The interval from 693 to 737 ft (225 to 211 m) bgs (the total depth) was drilled using air mist with a foaming agent as the drilling fluid. WQSP-1 was drilled to 695.6 ft (212 m) bgs using a 9⅞-in. drill bit and was cored from 695.6 to 737 ft (212 to 225 m) bgs using a 5¼-in. core bit to cut 4-in.- (0.1-m) diameter core. After coring, WQSP-1 was reamed to 9⅞ in. (0.3 m) in diameter to total depth. WQSP-1 was cased from the surface to 737 ft (224.6 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-centimeter (cm)] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen across the Culebra interval from 702 to 727 ft (214 to 222 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 640 to 651 ft (195 to 198 m) bgs and with 8/16 Brady gravel from

651 to 737 ft (198 to 225 m) bgs. Based on core log results, the Culebra is located from 699 to 722 ft (213 to 220 m) bgs (see Figure D18-10).

WQSP-2—Well WQSP-2 was drilled between September 6 and 12, 1994, to a total depth of 846 ft (257.9 m) bgs. The borehole was drilled through the Culebra and extends 12.3 ft (3.7 m) into the unnamed lower member of the Rustler. The well was drilled to a depth of 800 ft (244 m) bgs with a 9⅞-in. drill bit using compressed air as the drilling fluid. The interval from 800 to 846 ft (244 to 258 m) bgs (the total depth) was drilled with a 5¼-in. core bit to cut 4-in.- (0.1-m) diameter core using air mist with a foaming agent as the drilling fluid. After coring, WQSP-2 was reamed to 9⅞ in. (0.3 m) in diameter to total depth. WQSP-2 was cased from the surface to 846 ft (258 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-cm] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen across the Culebra interval from 811 to 836 ft (247 to 255 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 790 to 793 ft (241 to 242 m) bgs and with 8/16 Brady gravel from 793 to 846 ft (242 to 258 m) bgs. Based on core log results, the Culebra is located from 810.1 to 833.7 ft (247 to 254 m) bgs (see Figure D18-11).

WQSP-3—Well WQSP-3 was drilled between October 21 and 26, 1994, to a total depth of 880 ft (268 m) bgs. The borehole was drilled through the Culebra and extends 8.6 ft (2.6 m) into the unnamed lower member of the Rustler. The well was drilled to a depth of 880 ft (268 m) bgs using compressed air as the drilling fluid. The borehole was cleaned using air mist with a foaming agent. WQSP-3 was drilled to 833 ft (254 m) bgs using a 9⅞-in. drill bit and was cored from 833 to 879 ft (254 to 268 m) bgs using a 5¼-in. core bit to cut 4-in.- (0.1-m) diameter core. After coring, WQSP-3 was reamed to 9⅞ in. (0.3 m) in diameter to total depth of 880 ft (268 m) bgs. WQSP-3 was cased from the surface to 880 ft (268 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-cm] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen across the Culebra interval from 844 to 869 ft (257 to 265 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 827 to 830 ft (252 to 253 m) bgs and with 8/16 Brady gravel from 830 to 880 ft (253 to 268 m) bgs. Based on core log results, the Culebra is located from 844 to 870 ft (257 to 265 m) bgs (see Figure D18-12).

WQSP-4—Well WQSP-4 was drilled between October 5 and 10, 1994, to a total depth of 800 ft (244 m) bgs. The borehole was drilled through the Culebra and extends 9.2 ft (2.8 m) into the unnamed lower member of the Rustler. The well was drilled to a depth of 740 ft (226 m) bgs with a 9⅞-in. drill bit using compressed air as the drilling fluid. The interval from 740.5 to 798 ft (225.7 to 243 m) bgs was cored with a 5¼-in. (0.13-m) core bit to cut 4-in.- (0.1-m) diameter core using air mist with a foaming agent as the drilling fluid. After coring, WQSP-4 was reamed to 9⅞ in. (0.3 m) in diameter to total depth of 800 ft (244 m) bgs. WQSP-4 was cased from the surface to 800 ft (244 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-cm] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen across the Culebra interval from 764 to 789 ft (233 to 241 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 752 to 755 ft (229 to 230 m) bgs and with 8/16 Brady gravel from 755 to 800 ft (230 to 244 m) bgs. Based on core log results, the Culebra is located from 766 to 790.8 ft (233 to 241 m) bgs (see Figure D18-13).

WQSP-5—Well WQSP-5 was drilled between October 12 and 19, 1994, to a total depth of 681 ft (208 m) bgs. The borehole was drilled through the Culebra and extends into the unnamed lower member of the Rustler. The well was drilled to a depth of 676 ft (206 m) bgs using compressed air as the drilling fluid. The borehole was cleaned using air mist with a foaming agent. WQSP-5 was drilled to 648 ft (198 m) bgs using a 9⅞-in. drill bit and was cored from 648 to 676 ft (198 to 206 m) bgs using a 5¼-in. core bit to cut 4-in.-

(0.1-m) diameter core. After coring, WQSP-5 was reamed to 9⅞ in. (0.3 m) in diameter to total depth of 681 ft (208 m) bgs. WQSP-5 was cased from the surface to 681 ft (208 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-cm] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen across the Culebra interval from 646 to 671 ft (197 to 205 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 623 to 626 ft (190 to 191 m) bgs and with 8/16 Brady gravel from 626 to 681 ft (191 to 208 m) bgs. Based on core log results, the Culebra is located from 648 to 674.4 ft (198 to 205.6 m) bgs (see Figure D18-14).

WQSP-6—Well WQSP-6 was drilled between September 26 and October 3, 1994, to a total depth of 616.6 ft (187.9 m) bgs. The borehole was drilled through the Culebra and extends 9.7 ft (3 m) into the unnamed lower member of the Rustler. The well was drilled to a depth of 367 ft (112 m) bgs using compressed air as the drilling fluid. The interval from 367 to 616 ft (112 to 188 m) bgs (the total depth) was drilled using brine as the drilling fluid. WQSP-6 was drilled to 568 ft (173 m) 4-in.- (0.1-m) ft bgs using a 9⅞-in. drill bit and was cored from 568 to 616 ft (173 to 188 m) bgs using a 5¼-in. core bit to cut 4-in.- (0.1-m) diameter core. After coring, WQSP-6 was reamed to 9⅞ in. (0.3 m) in diameter to total depth of 616.6 ft (188 m) bgs. WQSP-6 was cased from the surface to 616 ft (188 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-cm] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen across the Culebra interval from 581 to 606 ft (177 to 185 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 567 to 570 ft (173 to 173.7 m) bgs and with 8/16 Brady gravel from 570 to 616.6 ft (174 to 188 m) bgs. Based on core log results, the Culebra is located from 582 to 606.9 ft (177 to 185 m) bgs (see Figure D18-15).

WQSP-6A—Well WQSP-6A was drilled between October 31 and November 1, 1994, to a total depth of 225 ft (69 m) bgs. The borehole was drilled through a water-producing zone in the Dewey Lake Redbeds that had been previously encountered while drilling well WQSP-6. The well was drilled to a depth of 225 ft (69 m) bgs using compressed air as the drilling fluid. The borehole was cleaned using air mist with a foaming agent. WQSP-6A was drilled to 160 ft (49 m) bgs using a 9⅞-in. drill bit and was cored from 160 to 220 ft (49 to 67 m) bgs using a 5¼-in. core bit to cut 4-in.- (0.1-m) diameter core. After coring, WQSP-6A was reamed to 9⅞ in. (0.3 m) in diameter to total depth of 225 ft (69 m) bgs. WQSP-6A was cased from the surface to 225 ft (69 m) bgs with 5-in. (0.1-m) (0.28-in. [0.7-cm] wall) blank fiberglass casing with in-line 5-in.- (0.1-m) diameter fiberglass 0.02-in. (0.1-cm) slotted screen from 190 to 215 ft (58 to 66 m) bgs. The annulus between the borehole wall and the casing/screen is packed with sand from 172 to 175 ft (52 to 53 m) bgs and with 8/16 Brady gravel from 175 to 225 ft (53 to 69 m) bgs (see Figure D18-16).

D18-4 Project Organization and Responsibilities

D18-4a Environmental Monitoring Manager

The Environmental Monitoring (EM) Manager is responsible for the overall design and implementation of the GMP. The EM Manager develops and approves specific procedures for the conduct of all GMP activities, and reviews and approves programmatic reports. The EM Manager provides oversight of appropriate levels of cooperation and consultation between the EM Section and the State of New Mexico regarding environmental monitoring and revises the QA section of the GMP.

The EM Manager and staff are responsible for achieving and maintaining quality in the GMP. All GMP data is reviewed and approved by the EM Manager, or designee, prior to release.

The EM Manager establishes minimum qualification criteria and training requirements for all GMP personnel. The EM Manager assures that position descriptions for assigned GMP personnel are adequately prepared. The EM Manager and/or Team Leader assures that training is performed on an individual basis to maintain an acceptable level of proficiency by all new or temporary GMP staff and by all permanent Groundwater Surveillance Program staff. The EM Manager assures that documents detailing all staff training are current and properly filed. Copies of training records are on file in the WID Technical Training Section.

The EM Manager appoints a GMP Team Leader and Field Team, assigning the following responsibilities described below.

D18-4b Team Leader

The Team Leader will coordinate and oversee field sampling activities, ensuring that sampling and associated procedures are followed and that QA/QC and safety guidelines are met. The Team Leader directs the GMP per written approved procedures, and initiates the review of programmatic plans and procedures. The Team Leader reviews and evaluates sample data, prepares and reviews programmatic reports, and assures that appropriate samples are collected and analyzed. The Team Leader assures that adequate technical support is provided to the Quality and Regulatory Assurance (Q&RA) Department, when required during audits of vendor facilities. Any nonconformances or project changes will be immediately communicated to the Team Leader.

D18-4c Field Team

The Field Team members consist of one or more scientists, engineers, or technicians, who are responsible for sample collection, handling, shipping, and preparation and maintenance of appropriate data sheets, and completion of sample tracking documentation under the direction of the Team Leader, in accordance with this GMP and associated field procedures. The field team will inspect, maintain, and ensure proper calibration of equipment prior to use at each site, while ensuring that site health and safety requirements are met at all times. The sampling team will communicate any nonconformances, malfunctions, or project changes to the Team Leader immediately.

D18-4d Safety Manager

The Safety Manager is responsible for ensuring that the GMP WIPP Safety Manual (WP12-1) addresses health and safety concerns associated with sampling and analysis activities. The Safety Manager is responsible for ensuring that field team members and any other personnel on site are operating under the WIPP Safety Manual and the personnel have appropriate training to operate on the site. The Safety Manager will conduct periodic health and safety audits (minimum annually) and initiate corrective action where deficiencies are identified. Health and safety audits will be documented and submitted to the EM Manager.

D18-4e Analytical Laboratory

The analytical laboratory used for sample analyses is responsible for supplying most sample collection containers and sample shipping containers to the field sampling team. Sample collection containers supplied by the laboratory will be certified as clean by either the laboratory or their supplier. WID

supplies containers for radiological samples. The analytical laboratory is responsible for performing analyses in accordance with this Groundwater Monitoring Program Plan and regulatory requirements. The laboratory will maintain documentation of sample handling and custody, analytical results, and internal QC data. Additionally, the laboratory will analyze QC samples in accordance with this plan and its own internal QC program for indicators of analytical accuracy and precision. Data generated outside laboratory acceptance limits will trigger an investigation and, if appropriate, corrective action, as directed by the EM Manager. The laboratory will report the results of the environmental sample and QC sample analyses and any necessary corrective actions that were performed. In the event that more than one analytical laboratory is used (e.g., for different analyses), each one has the responsibilities described above.

D18-4f Q&RA Manager

The Q&RA Manager provides independent oversight of the GMP, via the assigned cognizant Q&RA engineer, to verify that quality objectives are defined and achieved. The Q&RA Manager ensures objective, independent assessments of the GMP quality performance and the quality performance of the contract analytical laboratory. The Q&RA Manager has been delegated authority by the WID General Manager to access work areas, identify quality problems, initiate or recommend corrective actions, verify implementation of corrective actions, and ensure that work is controlled or stopped until adequate disposition of an unsatisfactory condition has been implemented.

D18-5 QA Requirements

QA requirements for WIPP are defined in the WID Quality Assurance Program Description, WP 13-1. Requirements specific to the GMP are presented in this section.

D18-5a QA Program—Overview

This program was developed to assure that the integrity and quality are maintained for all samples collected and that the integrity and quality of equipment and records are maintained in accordance with EPA guidance. The QA Program identifies data quality objectives (DQO), processes for assuring sample quality, and processes for generating and maintaining quality records.

D18-5b DQOs

DQOs are qualitative and quantitative statements that specify the quality of data required to support project decisions. DQOs are established to ensure that the data collected are of a sufficient and known quality for their intended uses. The overall DQO for this project is to collect accurate and defensible data of known quality that are sufficient to assess the concentrations of constituents in the groundwater underlying the WIPP area. The data generated thus far by the GMP has been used to establish a baseline groundwater quality database. For the purpose of this monitoring program, DQOs for measurement data will be specified in terms of accuracy, precision, completeness, representativeness, and comparability. Measurements of data quality in terms of accuracy and precision will be derived from the analysis of QC samples generated in the field and laboratory. Appropriate QC procedures will be used so that known and acceptable levels of accuracy and precision are maintained for each data set. This section defines the acceptance criteria for each QC analysis performed. The following subsections define each DQO.

D18-5b(1) Accuracy

Accuracy is the closeness of agreement between a measurement and an accepted reference value. When applied to a set of observed values, accuracy is a combination of a random component and a common systematic error (bias) component. Measurements for accuracy will include analysis of calibration standards, laboratory control samples, matrix spike samples, and surrogate spike samples. The bias component of accuracy is expressed as %R. Percent recovery is expressed as follows:

$$\%R = \frac{(\textit{measured sample concentration})}{\textit{true concentration}} \times 100$$

D18-5b(1)a Accuracy Objectives for Field Measurements

Field measurements will include pH, specific conductance (SC), temperature, Eh, and static water level. Field measurement accuracy will be determined using calibration check standards. Thermometers used for field measurements are calibrated to National Institute for Standards and Technology (NIST) traceable standard on an annual basis to assure accuracy. Accuracy of water-level measurements will be made before each measurement period by verifying calibration of the device within the specified schedule. Detailed instructions for water-level measurement, instrument calibration and field water quality measurements are detailed in WIPP procedures. Acceptable accuracy for all field analyses and measurements is ±5 percent.

D18-5b(1)b Accuracy Objectives for Laboratory Measurements

Analytical system accuracy will be quantified using the following laboratory accuracy QC checks: calibration standards, laboratory control samples (LCS), laboratory blanks, matrix and surrogate spike samples. Single LCSs and matrix spike and surrogate spike sample analyses will be expressed as %R. Laboratory analytical accuracy is parameter dependent and is prescribed in the laboratory SOQs (Attachment B).

D18-5b(2) Precision

Precision is the agreement among a set of replicate measurements without assumption or knowledge of the true value. Precision data will be derived from duplicate field and laboratory measurements. Precision will be expressed as relative percent difference (RPD), which is calculated as follows:

$$RPD = \frac{|(\textit{measured value sample } 1 - \textit{measured value sample } 2)|}{\textit{average of measured samples } 1 + 2} \times 100$$

D18-5b(2)a Precision Objectives for Field Measurements

Precision of field measurements of water-quality parameters will meet or exceed required reporting levels. SC, pH, temperature, and optionally Eh are measured during well purging and after sampling. SC

measurements will be precise to ±10% pH to 0.10 standard unit, and temperature to 0.10 degrees Celsius (°C), Eh to 10 millivolts (mV). Turbidity is not measured because the water drawn for the samples are typically very clear so there is no need to measure turbidity.

D18-5b(2)b Precision Objectives for Laboratory Measurements

Precision of laboratory analyses will be assessed by analyzing duplicate LCSs with each analytical batch assessed at a minimum frequency of 1 in 20 groundwater samples for nonradiological parameters, and 1 in 10 for radiological parameters. Analytical precision control limits will be determined by the laboratory by performing replicate analyses of control samples. Precision measurements will be expressed as RPD. Laboratory analytical precision is also parameter dependent and is prescribed in laboratory SOQs (Attachment B).

D18-5b(3) Contamination

In addition to measurements of precision and bias, QC checks for contamination will be performed. QC samples including trip blanks, field blanks, and method blanks will be analyzed to assess and document contamination attributable to sample collection equipment, sample handling and shipping, and laboratory reagents and glassware. Trip blanks are used to assess volatile organic compound (VOC) sample contamination during shipment and handling, field blanks are used to assess field sample collection methods, and method blanks are used to assess contamination resulting from the analytical process. Evaluation of sample blanks will be performed following EPA Functional Guidelines for Data Validation. Detection of analytes of interest in blank samples may be used to disqualify some samples, requiring resampling and additional analyses, on a case-by-case basis.

D18-5b(4) Completeness

Completeness is defined as a measure of the amount of usable valid data resulting from a data collection activity, given the sample design and analysis. Completeness may be affected by unexpected conditions that may occur during the data collection process.

Occurrences that reduce the amount of data collected include sample container breakage in the laboratory and data generated while the laboratory was operating outside prescribed QC limits. All attempts will be made to minimize data loss and to recover lost data whenever possible. The completeness objective for noncritical measurements (i.e., field measurements) is 90 percent and 100 percent for critical measurements (i.e., compliance data). If the completeness objective is not met, the WIPP EM Manager will determine the need for resampling on a case-by-case basis. Numerical expression of the completeness (%C) of data is as follows:

$$\%C = \frac{\textit{number of accepted samples}}{\textit{total number of samples collected}} \times 100$$

D18-5b(5) Representativeness

Representativeness is the degree to which sample analyses accurately and precisely represent the media they are intended to represent. Data representativeness for this sampling program will be accomplished

through implementing approved sampling procedures and the use of validated analytical methods. Sampling procedures are designed to minimize factors affecting the integrity of the samples. Groundwater samples will only be collected after well purging criteria have been met. The analytical methods selected are those that will most accurately and precisely represent the true concentration of analytes of interest.

D18-5b(6) Comparability

Comparability is the extent to which one data set can be compared to another. Comparability will be achieved through reporting data in consistent units and collection and analysis of samples using consistent methodology. Aqueous samples will consistently be reported in units of measures dictated by the analytical method. Units of measure include:

- Milligrams per liter (mg/L) for alkalinity, inorganic compounds and metals
- Micrograms per liter (µg/L) for VOCs.

Water-level measurements will be expressed as equivalent freshwater elevation in feet above mean sea level.

D18-5c Design Control

The groundwater monitoring system was designed to meet specifications described in 20 NMAC 4.1, Subpart V, §§264.97 and 264.601 through 264.603.

D18-5d Instructions, Procedures, and Drawings

Provisions and responsibilities for the preparation and use of instructions and procedures at the WIPP are outlined in Part II, Section 4 of the Quality Assurance Program Description (QAPD). Quality-affecting activities performed by or on behalf of groundwater monitoring are required to be performed in accordance with documented and approved procedures which meet the intent of 20 NMAC 4.1, Subpart V, §264.90.

Technical procedures were developed for each quality-affecting function performed for groundwater monitoring. The technical procedures unique to the GMP are maintained in the Environment Safety and Health Department (ES&H) at the WIPP. The procedures are sufficiently detailed and include, when applicable, quantitative or qualitative acceptance criteria.

Procedures were prepared in accordance with requirements in Section 4 of the QAPD.

D18-5e Document Control

Requirements for the control of documents are outlined in Part II, Section 4 of the WID QAPD. Controls ensure that the latest approved versions of procedures are used in performing groundwater monitoring functions and that obsolete materials are removed from work areas.

D18-5f Control of Processes

Process control requirements, defined in the QAPD, are met for this GMP.

D18-5g Inspection and Surveillance

Inspection and surveillance activities are conducted as outlined in Part II, Section 10 of the QAPD. The Q&RA Department is responsible for performing the applicable inspections and surveillance on the scope of work. EM section personnel are responsible for performance checks as defined in applicable procedures and determined by WID metrology laboratory personnel. Performance checks for the GMP are designed to determine the acceptability of purchased items and to assess degradation that occurs during use.

D18-5h Control of Monitoring and Data Collection Equipment

Monitoring and data collection (M&DC) equipment is controlled and calibrated according to WP 10-AD, WIPP Maintenance Administrative Procedures Manual, to ensure continued accuracy of groundwater monitoring data. Results of calibrations, maintenance, and repair are documented. Calibration records identify the reference standard and the relationship to national standards or nationally accepted measurement systems. Records are maintained to track uses of M&DC equipment. If M&DC equipment is found to be out of tolerance, the equipment is tagged and it is not used until corrections are made.

D18-5i Control of Nonconforming Conditions

Part II, Section 8 of the WID QAPD describes the system used at the WIPP for ensuring that appropriate measures are established to control nonconforming conditions. Nonconforming conditions connected to the GMP are identified in and controlled by documented procedures. Equipment that does not conform to specified requirements is controlled to prevent use. The disposition of defective items is documented on records traceable to the affected items. Prior to final disposition, faulty items are tagged and segregated. Repaired equipment is subject to the original acceptance inspections and tests prior to use.

D18-5j Corrective Action

Requirements for the development and implementation of a system to determine, document, and initiate appropriate corrective actions after encountering conditions adverse to quality at the WIPP are outlined in Part I, Section 3 of the QAPD. Conditions adverse to acceptable quality are documented and reported in accordance with corrective action procedures and corrected as soon as practical. Immediate action will be taken to control work performed under conditions adverse to acceptable quality and its results to prevent quality degradation.

D18-5k Quality Assurance Records

Part I, Section 4 of the QAPD outlines the policy used at the WIPP regarding identification, preparation, collection, storage, maintenance, disposition, and permanent storage of QA records.

Records to be generated in the groundwater monitoring program are specified by procedure. QA and RCRA operating records are identified. This is the basis for the labeling of records as "QA" or "RCRA operating" on the EM Records Inventory and Disposition Schedule (RIDS).

QA records document the results of the groundwater monitoring program implementing procedures and are sufficient to demonstrate that all quality-related aspects are valid. The records will be identifiable, legible, and retrievable in accordance with WP 15-PR, WID Records Management Plan, and QA record procedures.

D18-6 Monitoring Program Description

The WIPP GMP has been designed to meet the groundwater monitoring requirements of 20 NMAC 4.1 Subpart V §§264.90 through 264.101. The following sections of the monitoring plan describe the specific components of the GMP.

D18-6a Monitor Well Locations

Groundwater exists both above and below the WIPP repository, but no hydrologic continuity exists between the repository and the groundwater. Groundwater below the repository occurring in the sandstones of the Delaware Mountain Group (Powers, et al., 1978) is isolated by bedded salt deposits in the lower part of the Salado and in the underlying Castile. Groundwater below the repository is not being monitored as part of this program.

Groundwater above the repository is being monitored. Groundwater exists in both the Dewey Lake and the Rustler. Zones monitored for background characterization within the Rustler are the Culebra and the Magenta members. These zones are dolomite units isolated from one another by impermeable layers. With the exception of excavated shafts at WIPP, these zones are isolated from the repository excavations by bedded salt deposits in the upper two thirds of the Salado.

Postbackground monitoring will be focused on the Culebra because it is the primary flow path within the Rustler. Databases are maintained for the Magenta so that if the need arises monitoring of the Magenta can be resumed.

The Culebra is areally persistent, but quantity and quality of water vary considerably from place to place. The dolomite is vuggy, fractured, and commonly associated with anhydrite (Lambert and Mercer, 1977). The Culebra has a low hydraulic conductivity. It is a fractured unit that is best modeled as a dual-porosity media. Water yields are small and saline (Powers et al., 1978).

The Dewey Lake consists of orange-red siltstone, mudstone, and some sandstone. The Dewey Lake does not form an aquifer, but some permeable sand lenses are present and those yield limited quantities of fresh water to a few private wells in the area around the WIPP site (Powers et al., 1978). One such sand lens has been identified within the WIPP boundary and is scheduled for monitoring as part of the GMP.

Seven observation wells were completed after the WQSP baseline was established using EPA recommended drilling methods and casing materials that have the potential to meet RCRA monitoring standards. Six of the wells were completed in the Culebra; one well in the Dewey Lake. Two years of sampling are scheduled prior to the anticipated receipt of waste. The data gathered from these new wells will be compared to the existing database and the existing background data will be appended as appropriate. Of the six Culebra monitoring wells, (Figure D18-9) three were installed upgradient of the WIPP shaft areas and three wells installed downgradient of the facility in locations representative of the major groundwater flow directions (Davies, 1989). Well location surveys for each of the seven wells were performed by WID survey

personnel using the State Plane Coordinates-North American Datum Model 27 method. Results of the surveys are on file with the New Mexico State Engineers Department along with the associated extraction permits for each well.

Figure D18-9 shows the WIPP GMP well installation sites and the most recent freshwater head corrected potentiometric surface of the Culebra member of the Rustler.

D18-6b Monitoring Frequency

The GMP has been sampling the seven monitoring wells on a semiannual basis over the past two years to establish background water chemistry in accordance with 20 NMAC 4.1 Subpart V, §§264.97 and 264.98. Background water quality in both upgradient and downgradient wells will be established prior to receipt of waste at the WIPP.

As shown in Table D18-2, the GMP will collect groundwater quality samples for all seven wells on a semiannual basis during the life of the detection monitoring program.

From this point on, groundwater samples will be collected from the GMP wells at a frequency of four sample replicates collected semiannually from each well for the indicator parameters of pH, SC, TOC, and TOX to further establish background until detection monitoring in accordance with 20 NMAC 4.1, Subpart V (40 CFR §264.98) becomes applicable. Detection monitoring will start when the WIPP receives waste and continue through the postclosure phase as required by 20 NMAC 4.1, Subpart V (40 CFR §264.90[c]). During detection monitoring, one sample and one sample duplicate will be collected semiannually. 20 NMAC 4.1, Subpart V (40 CFR §264.97[g][2]) provides that an alternate sampling procedure to that provided in 264.98 may be proposed. Given the nature and rate of groundwater flow in the area surrounding WIPP, collecting and analyzing one sample semiannually will be protective of human health and the environment because any hazardous constituent leaving the underground disposal facility will not have the potential to substantially migrate beyond the compliance point in a one-year time frame. Groundwater flow characteristics are presented in detail in Appendices D6 and E1.

Groundwater levels will also be monitored in each of the seven wells on a monthly basis. The water level in each well will also be measured prior to each sampling event. Water-level measurements in the other existing WQSP wells will also be monitored on a monthly basis to supplement the area water-level database and to help define regional changes in groundwater flow directions and gradients. The characteristics of the groundwater monitoring program (frequency, location) will be evaluated if significant changes are observed in the groundwater flow direction or gradient. Any necessary changes will be identified and a permit modification sought as appropriate.

D18-6c Analytical Parameters

The analytes of interest that are now being measured to establish background concentrations include all indicator parameters and all other parameters listed in 20 NMAC 4.1 Subpart V, §264, Appendix IX. Samples that will be taken as part of the detection monitoring program during disposal operations include the most prevalent constituents in the waste as indicated in Table D18-3, plus metals (see Table C-9 in Chapter C and Appendix C6 for more detail). In addition, field measurements of pH, SC, temperature, chloride, Eh, total iron, and alkalinity are being taken during background sampling as specified in Section D18-8 of this plan. Alkalinity, chloride, and iron may be deleted during detection monitoring. Parameters such

as specific conductance, total dissolved solids, total suspended solids, density, pH, total organic carbon, and total organic halogens were chosen as indicator parameters because of their universal commonality to groundwater. Parameters such as chloride, alkalinity, calcium, magnesium, and potassium were chosen as matrix-specific general indicator parameters. Organic and inorganic compounds on the right hand side of Table E18-2 were chosen because they may occur in the waste to be disposed at the WIPP facility.

D18-6d Sample Collection and Laboratory Analysis

The mobile field laboratory provides a work place for conducting field sampling and analyses. The laboratory is positioned near the wellhead, is climate controlled, and contains the necessary equipment, reagents, glassware, and deionized water for conducting the various field analyses.

Two types of water samples are generally collected: serial samples and final samples. Serial samples are taken at regular intervals and analyzed in the mobile field laboratory for various physical and chemical parameters (called field parameters). The serial sample data are used to determine the chemical steady-state conditions of the groundwater as a direct function of the volume of the water being pumped from the well. Interpretation of the serial sampling data enables the Team Leader to make a determination of when steady-state conditions are attained in the pumped groundwater.

Final samples are collected when the serially sampled field parameters have achieved a steady state. If one or more of the field parameters do not stabilize—and there is reason to believe it will not—the Team Leader may choose to collect the final sample regardless of this instability in the field parameter(s).

The objective of the serial sampling effort is to obtain representative water samples in a reproducible manner. By definition, a representative groundwater sample is a sample of undisturbed groundwater. A groundwater sample is considered to be representative of the undisturbed groundwater only if it is chemically identical to the undisturbed groundwater (i.e., completely unaltered by the effects of drilling, postdrilling processes and reactions, and sampling procedures).

To determine how close the pumped groundwater is to being representative, a comparison is made by monitoring the same selected field parameters that were used to initially define the background characteristics of the water. When these parameters appear stable the determination is made that the water sample is representative.

D18-6d(1) Serial Samples

Serial samples are collected and analyzed in the mobile laboratory to detect and monitor the chemical variation of the groundwater as a function of the volume of water pumped. Once serial sampling begins, the frequency at which serial samples are collected and analyzed is left to the discretion of the Team Leader. The serial sampling frequency is based upon the site-specific conditions existing at each well, but usually is performed a minimum of three times during a sampling round.

The three field parameters of temperature, Eh, and pH are determined by either an "in-line" technique, using a self-contained flow cell, or an "off-line" technique, in which the samples are collected from

a nylon sample line at atmospheric pressure. The iron, divalent cation, chloride, alkalinity, specific conductance, and specific gravity samples are collected from the nylon sample line at atmospheric pressure.

New polyethylene containers are used to collect the serial samples from the nylon sample line. Serial sampling water collected for solute and specific conductance determinations is filtered through a 0.45 µm membrane filter using a stainless steel, in-line filter holder. Filtered water is used to rinse the sample bottle prior to serial sample collection. Unfiltered groundwater is used when determining temperature, pH, Eh, and specific gravity. Sample bottles are properly identified and labeled.

The filtered sample collected for solute analyses is immediately analyzed for iron and alkalinity because these two solution parameters are extremely sensitive to changes in the ambient water-sample pressure and temperature. A sample and duplicate of filtered water are collected and analyzed for solute parameters (alkalinity, chloride, divalent cations, and iron). Temperature, pH, and Eh, when not measured in a flow cell, are measured at the approximate time of serial sample collection. These samples are collected from the unfiltered sample line.

Experience gained from the serial sampling of wells has shown that samples to be analyzed for chloride and divalent cations (after preservation with nitric acid and stored at 4°C) can be stored for one week prior to analysis with confidence that the analytical results will not be altered.

Upon completion of the collection of the final sample suite, the serial sample bottles accrued throughout the duration of the pumping of the well are discarded. No serial sample bottles will be reused for sampling purposes of any sort. However, serial samples may be stored for a period of time depending upon the need. Procedures for sample collection and analysis are generated, approved, and maintained by the site documentation process.

D18-6d(2) Final Samples

The final sample is collected once the pumped groundwater has achieved a chemical steady state based on the indicator parameters discussed above. A serial sample is also collected and analyzed for each day of final sampling. Sample preservation, handling, and transportation methods are designed to maintain the integrity and representativeness of the final samples.

Prior to collecting the final samples, the collection team must consider the analyses to be performed so that proper shipping or storage containers can be assembled.

Final samples are sent to contract laboratories and analyzed for general chemistry, radionuclides, metals, and selected VOCs that are specific to the waste anticipated to arrive at WIPP.

Water samples are collected at atmospheric pressure using either the filtered or unfiltered nylon sampling lines branching from the main sample line. The samples are collected in new and unused glass and plastic containers.

Before the final sample is taken, all plastic and glass containers are rinsed with the pumped groundwater, either filtered or unfiltered, dependent upon analysis protocol. When the rinsing procedure is completed the final sample is collected.

The WIPP has not accepted waste for disposal and previous WQSP sample analyses have shown that requested hazardous constituents have not been introduced to the groundwater in the vicinity of WIPP by other activities.

The current monitoring system uses dedicated pumping systems and sample collection lines from the sampled formation to the well head. Nondedicated sample collection lines from the well head to the sample collection area are discarded after each use. Laboratory glassware are washed after each use with a solution of nonphosphorus detergent and deionized (DI) water and rinsed in DI water. Sample containers are new, certified clean containers that are discarded after one use. Groundwater level measurement devices are rinsed with fresh water after each use. Nondedicated sample collection manifold assemblies will be rinsed with two gallons of fresh water, then rinsed with five gallons of 5 percent nitric acid solution and rinsed with five gallons of DI water after each use. The exposed ends will be capped off during storage. Prior to the next use of the sampling manifold, it will be rinsed a second time with DI water and a blank rinsate sample will be collected to verify decontamination.

Resulting wastes are disposed of in accordance with the WID "Site Generated, Non-Radioactive Hazardous Waste Management Plan," WP 02-RC.01.

D18-6d(3) Laboratory Analysis

Analysis of samples are performed by a commercial laboratory that participates in EPA's contract laboratory program. Methods are specified in procurement documents and are selected to be consistent with EPA recommended procedures in SW 846 (EPA, 1986b). Data analysis is to provide an objective and reliable means for interpreting data in relation to the objectives of the data collection program. For the GMP the principal goal of data analyses is the comparison of a data point or data set to equivalent data collected at another location and time (such as preoperational background data or data collected at a control location) or to a fixed standard. Additional detail on analytical techniques and methods are given in Attachment B, Laboratory SOPs.

Several levels of analyses are required for each parameter before statistically valid interpretation can be achieved. The type of analysis used at each level varies among parameters due to the particular characteristics of parameters and the specific objectives of monitoring. Five general levels of data analyses are described here. Analyses at each of these levels is considered for each parameter. The levels are:

(1) Determination of accuracy for each point measurement by quantification and control of precision and bias

(2) Evaluation of the effects of auto-correlation on the expected value of the point measurement due to location and time of sampling

(3) Identification of the appropriate model of variability (i.e., a probability density distribution) for each point measurement and the calculation of descriptive statistics based on the chosen model

(4) Treatment of data anomalies

(5) Interpretation of data through statistically valid comparisons (tests) and trend analysis.

The DOE performs analytical monitoring on groundwater samples through a contractual relationship with a laboratory qualified to perform analyses in accordance with EPA protocols. WIPP establishes the criteria for laboratory selection, including the stipulation that the laboratory follow the procedures specified in SW 846 and that the laboratory follow EPA protocols. The selected laboratory must demonstrate, through laboratory SOPs, that it will follow appropriate EPA SW 846 requirements and the requirements specified by the EPA protocols. The laboratory must also provide documentation to WIPP describing the sensitivity of laboratory instrumentation. Instrumentation sensitivity needs to be considered due to regulatory requirements governing constituent concentrations in groundwater, and due to the complexity of brines associated with the WIPP repository.

Once the initial qualification criteria, as described above, have been met, WIPP selects a laboratory based upon competitive bid. The selected laboratory will perform analytical work for WIPP for a predetermined period of time, as specified in the contract between WIPP and the selected laboratory. As this period of performance comes to an end, a new laboratory selection/competitive bid process is initiated by WIPP. The same or a different laboratory may be selected for the new contract period. The SOPs for the laboratory currently under contract to WIPP are included in Attachment B. This monitoring plan will be revised as appropriate and new laboratory SOPs added if the contract laboratory changes.

D18-6e Statistical Analysis of Laboratory Data

As required by 20 NMAC 4.1, Subpart V §264.97, water-quality data collected during the background characterization program and the operational detection monitoring program will be evaluated using appropriate statistical techniques. The following describes the statistical analysis to be performed by the GMP.

D18-6e(1) Temporal and Spatial Analysis

Environmental parameters vary with space and time. The effect of one or both of these two factors on the expected value of a point measurement is statistically evaluated through spatial analysis and time series analysis; however, these methods often require extensive sampling efforts that are in excess of the practical requirements of the WIPP GMP. The application of these methods to a particular parameter must, therefore, be limited by consideration of its significance in the final interpretation of the data.

In particular, spatial analysis has limited use in this program, although the effect of spatial auto-correlation on the interpretation of the data is considered for each parameter. Spatial variability is accounted for by the use of predetermined key sampling locations. Data analysis is performed on a location-specific basis, or data from different locations is combined only when the data have been determined to be statistically homogeneous.

Time series analysis plays a more important role in data analysis for the GMP. Parameters are reported as time series, either in tabular form or as time plots. For key time series parameters, these plots are in the form of control charts on which control levels will be identified based on preoperational database, fixed standards, control location databases, or other standards for comparison. Where significant

seasonal changes in the expected value of the parameter are identified in the preoperational database or in the control locations, corrections in the control levels which reflect the seasonal change are made.

D18-6e(2) Distributions and Descriptive Statistics

For data sets which include more than ten data points that are homogeneous in space and time (including seasonal homogeneity) and have less than ten percent missing data, a test for conformance to the normal distribution is performed. A probability plot is an accepted method for performing this test; however, more powerful tests of normality, such as the W Test, or D'Agostino's Test (Gilbert, 1987) are more accurate. Any standard best fit test is acceptable, provided the assumptions of the tests are met.

If normality is not met, the data will be log-transformed and retested for normality. If the transformed data fit a normal distribution, the original data will be accepted as having lognormal distribution. If normality is still not found, two courses may be taken. One is to continue to test the fit to standard families of distributions, such as the gamma, beta, and Weibull, with proper modifications to subsequent analyses based on these results. The other course is to use nonparametric methods of data analysis.

For data sets smaller than ten, but homogeneous and complete, the lognormal distribution is assumed. Data sets with more than ten percent missing data are analyzed using nonparametric methods. Nonhomogeneous data sets are subdivided into homogeneous sets and each of these analyzed individually.

Descriptive statistics are calculated for each homogeneous data set. At a minimum, these include a central value and a range of variation. The central value is the arithmetic mean of the untransformed data if the data are not censored at either end. If the data are censored, either a trimmed mean or the median is used as the central value (which may be within the censored range). If the data set is greater than ten and is uncensored, the standard deviation is calculated and used as a basis for the reported range in variation. If these criteria are not met, the range between the 0.25 and 0.75 quartiles is used.

D18-6e(3) Data Anomalies

Data anomalies include data points reported as being below the limit of detection (LD) or otherwise censored over a specific range of values, missing data points occurring randomly in the data set, and outliers that cannot be ascribed to a known source of variation.

Whenever possible, values which are below detection limits are obtained and incorporated into the database for statistical analysis. When values are not available, alternative methods of analysis, as described in previous sections, are used. In particular, the use of nonparametric statistics is required.

Missing data points comprising less than 10 percent of the data set do not affect data analyses. Results based on data in which more than 10 percent is missing are identified as such at the time of reporting. Consideration of the potential effect of missing data must be made when the majority of the data are missing from a discrete time span.

An outlier is defined as any data point occurring in either extreme upper or lower range of the data distribution for which there is less than 0.01 probability of occurrence. For normally distributed data this is roughly 2.3 or more standard deviations above or below the mean. When no probability model is identified, outliers may only be found through visual inspection of the data.

If an outside source of variation is not identified to account for outliers in a data set, it is included in the data set and all subsequent analyses. If the inclusion of such outliers is found to affect the final results of the analyses significantly, both results (with and without outliers) are reported.

D18-6e(4) Comparisons and Reporting

Prior to waste receipt, two duplicate measurements will have been made of each indicator and other parameters required by 40 CFR 265.92 (pH, SC, TOC, TOX) at every GMP groundwater monitoring well during each of the four background sampling events. Following completion of the four sampling events, the arithmetic mean and variance must then be calculated by the field supervisor or designee for each site. These measurements will then serve as a background value against which statistical values for subsequent sampling events during detection monitoring will be compared. Statistical analysis and comparison will be accomplished using Cochran's Approximation to the Behrens-Fisher students' t-test at the 0.01 level of significance (described in Appendix IV to 40 CFR 264, and stipulated in 40 CFR 265.93[b]). If the comparisons show a significant increase at any monitoring site, the site must be resampled and an analysis performed as soon as possible. The results of the statistical comparison will be reported annually in the ASER.

D18-7 Sample Documentation and Custody

To ensure the integrity of samples from the time of collection through reporting date, sample collection, handling, and custody must be documented. Sample custody and documentation procedures for EM sampling and analysis activities are detailed in the "Administrative Processes for Environmental Monitoring Programs" (WP02-EM3001). These procedures will be strictly followed throughout the course of each sample collection and analysis event.

Standardized forms used to document samples include sample identification numbers, sample labels, custody tape, the sample tracking log books, and the request for analysis/chain of custody (RFA and CofC) form. The forms are briefly defined in the following subsections.

All sample documentation will be completed for each sample and reviewed by the Team Leader for completeness and accuracy.

D18-7a Sample Numbers and Labels

A unique sample identification number will be assigned to each sample sent to the laboratory for analysis. The Team Leader will assign the numbers prior to sample collection. The sample identification numbers are used to track the sample from the time of collection through data reporting. Every sample container sent to the laboratory for analysis will be identified with a label affixed to it. Sample label information will be completed in permanent, indelible ink and will contain the following information: sample identification number with sample matrix type; sample location; analysis requested; time and date of collection; preservative(s), if any; and the sampler's name or initials.

D18-7b Custody Seals

Custody seals are used to detect unauthorized sample tampering from collection through analysis. The custody seals are adhesive-backed strips that are destroyed when removed or when the container is opened. The seal is dated, initialed, and affixed to the sample container in such a manner that it is necessary to break the seal to open the container. Seals are affixed to sample containers in the field immediately after collection. Upon receipt at the laboratory, the laboratory custodian will inspect the seal for integrity; a broken seal invalidates the sample.

D18-7c Sample Tracking Logbook

A sample tracking logbook (STLB) form will be completed for each sample collected. The STLB includes the following information: C of C number; Request for Analysis No.; date sample(s) were sent to the lab; laboratory name; acknowledgement of receipt or comments; well name and round number. Sample codes indicate the well location; the geologic formation where the water was collected from, the sampling round number; and the sample number. The code is broken down as follows:

$$WQ6^{1}C^{2}R2^{3}N1^{4}$$

[1]Well identification (e.g., WQSP-6 in this case)
[2]Geologic formation (e.g., the Culebra in this case)
[3]Sample round no. (Round 2)
[4]Sample no. (N1)

STLB information is completed in the field by the sampling team and checked by the Team Leader. When samples are shipped, the STLB remains in the custody of the EM Section for sample tracking purposes.

D18-7d Request for Analysis and Chain of Custody

A request for analysis (RFA) and chain of custody (CofC) form will be completed during or immediately following sample collection and will accompany the sample through analysis and disposal. An example of the RFA and CofC form is presented in Figures D18-17a and 17b. The RFA and CofC form is signed and dated each time the sample custody is transferred. A sample is considered to be in a person's custody if: the sample is in his/her physical possession; the sample is in his/her unobstructed view; and/or the sample is placed, by the last person in possession of it, in a secured area with restricted access. During shipment, the carrier's airbill number serves as custody verification. Upon receipt of the samples at the laboratory, the laboratory sample custodian acknowledges possession of the samples by signing and dating the RFA and CofC. The completed original (top page) of the RFA and CofC is returned to the Team Leader with the laboratory analytical report and becomes part of the permanent record of the sampling event. The RFA and CofC form also contains specific instructions to the laboratory for sample analysis, potential hazards, and disposal instructions.

D18-8 Field Operations

The new GMP monitoring wells are routinely sampled and monitored following established standard operating procedures (SOP). These monitoring related activities include static water level measurements, serial

and final sampling, equipment installation, equipment calibration, field water quality analyses, sample handling and shipment, and waste management. The following subsections describe the routine field procedures employed by the GMP.

D18-8a Water Quality Sampling Technique

The GMP has employed two types of sampling procedures at the WIPP: serial sampling and final sampling.

D18-8a(1) Serial Sampling

Serial sampling is the collection of sequential samples for the purpose of determining when the water chemistry stabilizes or reaches a steady state. Ideally, when the water chemistry stabilizes, it is assumed that the chemistry is representative of the native formation fluid, and a final sample is collected. However, the water samples may still be impacted by well construction practices and effects from the installation of downhole pumping and sampling equipment.

The field analytical parameters found to be the most useful in identifying a steady state condition of the water chemistry include chloride, divalent cations (hardness), and alkalinity, which are analyzed by classic wet chemistry bench methods (titration) in the mobile field laboratory. Total iron has also been found to be a useful indicator and is analyzed using spectrophotometric methods. Other serial sampling parameters analyzed in the field include measurement of pH, Eh, temperature, specific conductance, and specific gravity. Collection and analysis of serial samples are performed in accordance with approved procedures.

D18-8a(2) Final Samples

Final groundwater samples are collected once evidence from serial sampling indicates that the pumped groundwater has reached a chemical steady state. Final samples are forwarded to a contract analytical laboratory for analysis.

Final samples are collected in the appropriate type of container for the specific analysis to be performed. For each parameter analyzed, a sufficient volume of sample is collected to satisfy the volume requirements of the analytical laboratory (as specified by Laboratory SOPs). This includes an additional volume of sample water necessary for maintaining quality control standards. All final samples are treated, handled, and preserved as required for the specific type of analysis to be performed. Details about sample containers, preservation, and volumes required for individual types of analyses are found in the applicable procedures generated, approved, and maintained by the contract analytical laboratory (Attachment B).

Duplicates of the final sample are provided to WIPP oversight agencies as requested by the DOE. A duplicate of the sample is also preserved with nitric acid and placed in storage within the ES&H Environmental Sample storage area and held until final reports from the contract analytical laboratory have been evaluated and approved. When the final laboratory report has been approved the samples are removed from storage and destroyed.

Detailed protocols, in the form of procedures, assure that final samples are collected in a consistent and repeatable fashion. Procedure WP02-EM1006 defines the requirements for collection of final samples for analyses (Attachment A).

The serial sampling process will probably not be needed with the new wells completed specifically for the GMP and detection monitoring program. However, during the first two years of sampling, the wells will be serially sampled using an abbreviated method. It is anticipated that changes in the water chemistry from stagnated to representative will occur within the first 24 hours of the purging process. Whereas, this change usually occurred over a seven-day period with the old wells.

During the first two or three years of sampling, these wells will be serially sampled with the first sample being analyzed as soon as possible after the pump is turned on and daily thereafter for a period of four days or until the field parameters (chloride, divalent cations, alkalinity and iron) stabilize. Eh, pH, and conductance will be monitored continuously by using a flow cell with ion-specific electrodes and a real-time readout. After two years of sampling data have been accumulated, a decision will be made to determine if the serial sampling process can be eliminated. If serial sampling is removed from the water quality sampling well protocol, the decision to collect samples will be based on the number of well bore volumes purged and the results of continuous monitoring of temperature, Eh, pH, and conductance. Removal of serial sampling from the program will be accomplished through a permit modification and a modification to this plan.

D18-8a(3) Groundwater Pumping and Sampling Systems

The water-bearing units at the WIPP are highly variable in their ability to yield water to monitoring wells. The Culebra, the most transmissive hydrologic unit in the WIPP area, exhibits transmissivities that range many orders of magnitude across the site area and is the primary focus of the GMP. Because the water-yielding characteristics of the hydrologic units at the WIPP are variable, different types of pumping equipment have been used during water quality sampling activities.

The groundwater pumping and sampling systems used to collect a groundwater sample from the seven new GMP wells are designed to provide continuous and adequate production of water so that a representative groundwater sample can be obtained. The wells used for water quality sampling vary in yield, depth, and pumping lift. These factors affect the duration of pumping as well as the equipment required at each well.

The type of pumping and sampling system to be used in a well depends primarily on the aquifer characteristics of the Culebra and well construction. At the present time, the GMP wells are individually equipped with dedicated submersible pumping assemblies. Each well has a specific type of submersible pump, matched to the ability of the well to yield water during pumping. The down-hole submersible pumps are controlled by a variable electronic flow controller to match the production capacity of the formation at each well.

Wells that are completed for use as GMP installations are cased and screened through the production interval with materials that do not yield contamination to the aquifer or allow the production interval to collapse under stress (high epoxy fiberglass). Details of well construction are presented in Section D18-3c. An electric, submersible pump installation without the use of a packer is an acceptable installation in this instance. The largest amount of discharge from the submersible pump takes place from a discharge pipe. In addition to this main discharge pipe a dedicated nylon sample line, running parallel

to the discharge pipe, is also used. Flow through the pipe is regulated on the surface by a flow control valve and/or variable speed drive controller. Cumulative flow is measured using a totalizing flow meter. Flow from the discharge pipe is routed to a discharge tank for disposal.

The dedicated nylon sampling line is used to collect the water sample that will undergo analysis. By using a dedicated nylon sample line, the water is not contaminated by the metal discharge pipe. The sample line branches from the main discharge pipe a few inches above the pump. Flow from the sample line is routed into the sample collection area. Flow through the sample collection line is regulated by a flow-control valve. The sample line is insulated at the surface to minimize temperature fluctuations.

If pumping conditions change with time, GMP wells may be equipped with gas-driven pumps. A gas-driven pump and sampling system can be used on any volume well. When used, the pump intake is set at a predetermined depth near or in the production zone. The pumping rate is adjusted to maintain the water level in the well above the pump intake. The flow rate for gas driven pumps is controlled by regulating the air pressure on the pump intake or by a flow control valve. Water is continuously discharged into a water storage tank. Detailed protocol for assembling, installing, and controlling pumping and sampling systems is found in the procedures generated, approved, and maintained by the site documentation process.

D18-8a(4) Pressure Monitoring Systems

The GMP wells do not require the installation of a packer because sample biases due to well construction deficiencies are not present. However, pressures will be monitored using down hole automatic air line bubblers in the formation to maintain water level above the pump intake. Pressure transducers may be used in line with bubblers to provide continuous electronic monitoring through data acquisition systems. Procedures governing the installation and use of pressure monitoring devices are generated, approved, and maintained by the site documentation process.

D18-8b Water-Level Monitoring Methodology

The monitoring of water levels is not specifically requested in 20 NMAC 4.1, Subpart V, as part of the groundwater detection monitoring program. However, the NMED has determined that groundwater level monitoring is needed to comply with the standards for miscellaneous units found in 20 NMAC 4.1, Subpart V, §§264.600 through 264.603.

D18-8b(1) Scope

This section of the WIPP GMP describes the WIPP groundwater level monitoring program (WLMP). The WLMP is a subprogram of the GMP. The quality assurance activities of the WLMP are in strict accordance with the QAPD and the quality assurance implementing procedures specific to water level monitoring are found in WP 02-1, Groundwater Monitoring Program Plan and Procedures Manual.

Water level monitoring is in progress now and will continue through the postoperational phase of the WIPP. This section of the plan addresses the activities of the WLMP during the preoperational and operational phases of the WIPP. Postoperational activity plans will be formulated at a later date and will address the objectives of water level monitoring as required at the time of decommissioning.

D18-8b(2) Introduction

This program will continue the collection and documentation of water level data initiated by the U.S. Geological Survey (Richey, 1987) and SNL (Stensrud et al., 1988) as part of the WIPP Site Characterization Program.

As currently planned, water level measurements will be conducted using hydrologic test wells that were constructed for both the site characterization and the GMP. These test wells are distributed geographically both within and surrounding the WIPP site. The frequency of measurement is subjectively defined by the need to record the dynamic nature of the potentiometric surface through time.

On October 1, 1988, the ES&H assumed responsibility for groundwater level monitoring activities. At that time a WLMP plan was still being developed. In June of 1989, an initial plan was finalized entitled WP 07-2, WIPP Water-Level Monitoring Program Plan, IT Corp. (June 1989). WP 07-2 was subsequently replaced in 1990 by WP 02-1, Groundwater Monitoring Program Plan and Procedures Manual.

Collection of groundwater-level data assists the DOE in meeting regulatory compliance and permitting requirements. These data also provide:

- Data collection as required by the Environmental Monitoring Plan.
- A means to fulfill commitments made in the FEIS.
- A means to comply with future groundwater inventory and monitoring regulations.
- Input for making land use decisions, (i.e., designing long-term active and passive institutional controls for the site).
- Assistance in understanding any changes to readings from the water-pressure transducers installed in each of the shafts to monitor water conditions behind the liners.
- An understanding of whether or not the horizontal and vertical gradients of flow are changing over time.

D18-8b(3) Objective

The objective of the WLMP is to extend the documented record of water-level fluctuations in the Culebra and Magenta members of the Rustler in the vicinity of the WIPP facility and to meet the requirements of 20 NMAC 4.1, Subpart V, §§264.600 through 264.603. Water-level data will also be collected from wells completed in other water-bearing zones overlying and underlying the WIPP repository horizon when access to those zones is possible. This includes, but is not limited to, the Bell Canyon, the Forty-niner, the contact zone between the Rustler and Salado, and the Dewey Lake.

The scope of the program is subject to change depending upon the following:

- Data trends
- Performance assessment program needs
- Environmental Monitoring Program needs
- Regulatory compliance needs

Water level measurements will be taken monthly in at least one accessible completed interval at each available well pad. At well pads with two or more wells completed in the same interval, quarterly measurements will be taken in the redundant wells (well locations are shown in Figure D18-18).

Water level monitoring will continue through the life of the WIPP Project. It may be deemed necessary to temporarily increase the frequency of monitoring to effectively document naturally occurring or artificial perturbations that may be imposed on the hydrologic systems at any point in time. This will be conducted in selected key wells by increasing the frequency of the manual water-level measurements or by monitoring water pressures with the aid of electronic pressure transducers and remote data-logging systems.

Interpretation of water level measurements and corresponding fluctuations over time is complicated at the WIPP by spatial variation in fluid density both vertically in well bores and areally from well to well. To monitor the hydraulic gradients of the hydrologic flow systems at the WIPP accurately, actual water level measurements and the densities of the fluids in the well bores will be monitored. When both of these parameters are known, equivalent freshwater heads will be calculated. The concept of freshwater head is discussed in Lusczynski (1961).

A discussion explaining the calculation of freshwater heads from midformation depth at WIPP can be found in Haug, et al. (1987). Freshwater heads are useful in identifying hydraulic gradients in aquifers of variable density such as those existing at the WIPP site. Freshwater head at a given point is defined as the height of a column of freshwater that will balance the existing pressure at that point (Lusczynski, 1961).

Freshwater heads are indicative of heads above the center of the Culebra and freshwater elevations are indicative of elevations above mean sea level.

Measured water-level data can be converted to equivalent freshwater head from knowledge of the density

$$p = \rho g h$$

of the borehole fluid.
where

ρ = average density of the borehole fluid
h = fluid column height above the datum.

If the freshwater density is assumed to be 1.000 g/cm^3, then the equivalent freshwater head is equal to the fluid column height times the average borehole fluid density.

Detailed fluid-density logs have been developed for calculating the exact average borehole-fluid density (Crawley, 1988).

A Pressure Density Survey Program (PDSP) has been conducted to determine the actual variation in density gradients existing in the test wells. The PDSP measured the actual midformation pressures of the Culebra. Data from this program have identified those wells in which some adjustment to measured water level values must be accounted for in order to calculate the measured water levels accurately in terms of equivalent freshwater heads (Crawley, 1988).

D18-8b(4) Field Methods

To obtain an accurate groundwater level measurement, a calibrated water level measuring device is lowered into a test well and the depth to water is recorded from a known reference point. When using an electrical conductance probe, the depth to water can be determined by reading the appropriate measurement markings on the embossed measuring tape when the alarm is activated at the surface. Specific procedures regarding the specific activities governing the Water-Level Monitoring Program are generated, approved, and maintained by the site documentation process.

D18-8b(5) Records and Document Control

All incoming data will be processed in a timely manner to assure data integrity. The data management process for water level measurements begins with completion of the field data sheets. Date, time, tape measurement, equipment identification number, calibration due date, initial of the field personnel, and equipment/comments are recorded on the field data sheets. If, for some unexpected reason, a measurement is not possible (i.e., a test is under way that blocks entry to the well bore), then a notation as to why the measurement was not taken is recorded in the comment column. Personnel also use the comment column to report any security observations (i.e., well lock missing).

Data recorded on the field data sheets and submitted by field personnel are subject to guidelines outlined in WP 02-3, Environmental Procedures Manual. The data are entered onto a computerized worksheet. The worksheet calculates water level in both feet and meters relative to the top of casing and also relative to mean sea level. The worksheet also adjusts levels to equivalent freshwater heads.

A check print is made of the worksheet printout. The check print is used to verify that data taken in the field is properly reported on the database printout. A minimum of 10 percent of the spreadsheet calculations are randomly verified on the check print to ensure that calculations are being performed correctly. If errors are found, the worksheet is corrected. The data contained on the computerized worksheet are translated into a database file. A printout is made of the database file. The data each month are then compiled into report format and transmitted to the appropriate agencies as requested by the DOE.

A computerized database file is maintained for all groundwater level data. Monthly and quarterly data are appended into a yearly file. Upon verification that the yearly database is free of errors, it is appended into the project database file. A printed copy of the project database is maintained in the ES&H EM fire-resistant storage area current through December of the preceding year.

D18-8b(6) Reporting

Data collected from this program are reported in the Annual Site Environmental Report (ASER). The ASER includes all applicable information that may affect the comparison of water level data through time. This information will include but is not limited to:
1Well configuration changes that may have occurred from the time of the last measurement (i.e., plug installation and removal, packer removal and reinstallation, or both; and the type and quantity of fluids that may have been introduced into the test wells).

2Any pumping activities that may have taken place since publication of the last annual report (i.e., water quality sampling, hydraulic testing, and shaft installation or grouting activities).

D18-8c Calibration

D18-8c(1) Sampling Equipment Calibration Requirements

The equipment used to collect data for the WQSP is to be calibrated in accordance with WP 10-AD, WIPP Maintenance Administrative Procedures Manual. The EM Section is responsible for calibrating needed equipment on schedule, in accordance with written procedures. The EM Section is also responsible for maintaining current calibration records for each piece of equipment.

D18-8c(2) Water-Level Monitoring Equipment Calibration Requirements

The equipment used in taking groundwater level measurements is to be calibrated in accordance with WP 10-AD, WIPP Maintenance Administrative Procedures Manual. The EM Section is responsible for calibrating needed equipment on schedule, in accordance with written procedures. The EM Section is also responsible for maintaining current calibration records for each piece of equipment.

D18-8d Well Evacuation Procedures

Each GMP well is evacuated using the dedicated submersible pump until all serial sampling field parameters are stable. This evacuation procedure generally removes numerous well bore volumes of water from the well and assures representative water samples.

D18-8e Sample Preservation, Tracking, Packaging and Transportation

Many of the chemical constituents measured by the GMP are not chemically stable and require preservation and special handling techniques. Samples requiring acidification are treated with either high purity hydrochloric acid, nitric acid, or sulfuric acid (ULTREX or equivalent), depending upon the standard method of treatment required for the particular parameter suite or as requested by contract laboratory SOPs (Attachment B).

The procedure used by the contract laboratory to which the samples are being sent prescribes the type and amount of preservative, the container material type, and the required sample volumes that should be collected. This information is recorded on the Final Sample Checklist for use by field personnel

when final samples are being collected. WIPP will follow the EPA RCRA Groundwater Monitoring Technical Enforcement Guidance Document, Table 4-1, if laboratory SOPs do not specify sample container, volume, or preservation requirements.

The sample tracking system at WIPP uses uniquely numbered CofC Forms and RFA Forms. The primary consideration for storage or transportation is that samples must be analyzed within the prescribed holding times for the parameters of interest. Procedures for sample tracking and preservation are generated, approved, and maintained by the site documentation process.

Insulated shipping containers packaged with reusable ice packs are used to keep the samples cool during transport to the contract laboratory. Hold times for specific analytical parameters require samples to be shipped by express air freight. The coolers are packaged to meet Department of Transportation and International Air Transportation Association commercial carrier regulations.

D18-9 Reporting

D18-9a Laboratory Data Reports

Laboratory data are provided in electronic and hard copy reports to WIPP. Laboratory data reports will be forwarded to the Team Leader and contain the following information for each analytical report:

- A brief narrative summarizing laboratory analyses performed, date of issue, deviations from the analytical method, technical problems affecting data quality, laboratory quality checks, corrective actions (if any), and the project manager's signature approving issuance of the data report.

- Header information for each analytical data summary sheet including: sample number and corresponding laboratory identification number; sample matrix; date of collection, receipt, preparation and analysis; and analyst's name.

- Analytical parameter, analytical result, reporting units, reporting limit, analytical method used.

- Results of QC sample analyses for all concurrently analyzed QC samples.

D18-9b Statistical Analysis and Reporting of Results

Analytical results from semi-annual groundwater sampling activities will be compared and interpreted by the Team Leader through generation of statistical analyses as described in Section D18-6. The Team Leader or designee will perform statistical analyses; the results will be included in the ASER in summary form.

D18-9c Annual Site Environmental Report

Data collected from groundwater sampling activities will be reported to the EM Manager in the ASER. The ASER includes all applicable information that may affect the comparison of water level data through time. This information will include but is not limited to:

- Well configuration changes that may have occurred from the time of the last measurement (i.e., plug installation and removal, packer removal and reinstallation, or both; and the type and quantity of fluids that may have been introduced into the test wells).

- Any pumping activities that may have taken place since publication of the last annual report (i.e., water quality sampling, hydraulic testing, and shaft installation or grouting activities).

The GMP data used in generating the ASER will be maintained as part of the WIPP operating record and made available to the NMED for review as specified in the permit.

D18-10 Records Management

Records generated during groundwater sampling and water level monitoring events will be maintained in the form project files in the EM section. Project records include, but are not limited to:

- SAPs
- SOPs
- STLBs
- RFA and CofC forms
- Contract Analytical Laboratory Data Reports
- Variance Logs and Nonconformance Reports
- Corrective Action Reports.

All raw analytical records generated in conjunction with groundwater sampling and water level monitoring will be stored in fire resistant cabinets in the EM section according to the RIDs and will be made available for inspection upon request. These records will be transmitted to the WIPP PRS for long-term storage in accordance with the RIDS.

- Instrument maintenance and calibration records
- QC sample data
- Control charts and calculation
- Sample tracking and control documentation
- Raw analytical results.

D18-11 References

Beauheim, R.L., 1986. "Hydraulic-Test Interpretations for Well DOE-2 at the Waste Isolation Pilot Plant (WIPP) Site," *SAND86-1364*, Albuquerque, NM: Sandia National Laboratories, CCA Reference 40.

Beauheim, R.L., 1987a. "Analysis of Pumping Tests at the Culebra Dolomite Conducted at the H-3 Hydropad at the Waste Isolation Pilot Plant (WIPP) Site," *SAND86-2311*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Beauheim, R.L., 1987b. "Interpretation of Single Well Hydraulic Tests Conducted at and Near the Waste Isolation Pilot Plant (WIPP) Site, 1983–1987," *SAND87-0039*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Beauheim, R.L., 1987c. "Interpretation of the WIPP-13 Multipad Pumping Test of the Culebra Dolomite at the Waste Isolation Pilot Plant (WIPP) Site," *SAND87-2456*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Chapman, J.B., 1986. "Stable Isotopes in Southeastern New Mexico Groundwater: Implications for Dating Recharge in the WIPP Area," *EEG-35*, New Mexico Environmental Evaluation Group, Santa Fe, New Mexico.

Chapman, J.B., 1988. "Chemical and Radiochemical Characteristics of Groundwater in the Culebra Dolomite, Southeastern New Mexico," *Report EEG-39*," New Mexico Environmental Evaluation Group, Santa Fe, New Mexico.

Corbet, T.F., and P.M. Knupp, 1996. "The Role of Regional Groundwater Flow in the Hydrogeology of the Culebra Member of the Rustler Formation at the Waste Isolation Pilot Plant (WIPP), Southeastern New Mexico," *SAND96-2133*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Crawley, M.E., 1988. "Hydrostatic Pressure and Fluid-Density Distribution of the Culebra Member of the Rustler Formation Near the Waste Isolation Pilot Plant, Southeastern New Mexico," *DOE/WIPP 88-030*, U.S. Department of Energy, Carlsbad, New Mexico.

Davies, P.B., 1989. "Variable-Density Ground-Water Flow and Paleohydrology in the Waste Isolation Pilot Plant (WIPP) Region, Southeastern New Mexico," U.S. Geological Survey Open-File Report 88-490, Albuquerque, New Mexico.

Domenico, P.A., and F.W. Schwartz, 1990. "Physical and Chemical Hydrogeology," New York: John Wiley & Sons, Textbook.

Domski, P.S., D.T. Upton, and R.L. Beauheim, 1966. "Hydraulic Testing Around Room Q: Evaluation of the Effects of Mining on the Hydraulic Properties of Salado Evaporites," *SAND96-0435*, Albuquerque, NM: Sandia National Laboratories. CCA Reference 218.

Earlougher, E.C., Jr., 1977. "Advances in Well Test Analysis," Dallas, TX: Society of Petroleum Engineers of Aime. Textbook.

Haug, A., V.A. Kelly, A.M. LaVenue, and J.F. Pickens, 1987. "Modeling of Ground-Water Flow in the Culebra Dolomite at the Waste Isolation Pilot Plant (WIPP) Site: Interim Report," *SAND86-7167*, Sandia National Laboratories/New Mexico, Albuquerqua, New Mexico.

Holt, R.M., and D.W. Powers, 1988. "Facies Variability and Post-Deposition Alteration Within the Rustler Formation in the Vicinity of the Waste Isolation Pilot Plant, Southeastern New Mexico," *DOE-WIPP-88-04*, Carlsbad, New Mexico.

Jones, T.L., V.A. Kelley, J.F. Pickens, D.T. Upton, R.L. Beauheim, P.B. Davies, 1992. "Integration of Interpretation Results of Tracer Tests Performed in the Culebra Dolomite at the Waste Isolation Pilot Plant Site," *SAND92-1579*, Albuquerque, New Mexico.

Kloska, M.B., G.J. Saulnier, Jr., and R.L. Beauheim, 1995. "Culebra Transport Program Test Plan: Hydraulic Characterization of the Culebra Dolomite Member of the Rustler Formation at the H-19 Hydropad on the WIPP Site."

Lambert, S.J., and J.W. Mercer, 1977. "Hydrologic Investigations of Los Medaños Area, Southeast New Mexico," Sandia National Laboratories/New Mexico Report *SAND77-1401*, Albuquerque, New Mexico.

Lambert, S.J., 1987. "Stable-Isotope Studies of Groundwaters in Southeastern New Mexico," *SAND85-1978c*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico, in Chaturvedi, L., ed., "The Rustler Formation at the WIPP Site," *EEG-34*, New Mexico Environmental Evaluation Group, Santa Fe, New Mexico.

Lambert, S.J., and J.A. Carter, 1987. "Uranium-Isotope Systematics in Groundwaters of the Rustler Formation, Northern Delaware Basin, Southeastern New Mexico," *SAND87-0388*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Lambert, S.J., and D.M. Harvey, 1987. "Stable-Isotope Geochemistry of Groundwaters in the Delaware Basin of Southeastern New Mexico," *SAND87-0138*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico

LaVenue, A.M., T.L. Cauffman, and J.F. Pickens, 1990. "Groundwater Flow Modeling at the Culebra Dolomite, Volume I: Model Calibration," *SAND89-7068/1*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Lusczynski, N.J., 1961. "Head and Flow of Ground Water of Variable Density," Journal of Geophysical Research, Vol. 66, No. 12, pp. 4247–4256.

Powers, D.W., S.J. Lambert, S.E. Shaffer, L.R. Hill, and W.D. Weart, eds., 1978. "Geologic Characterization Report for the Waste Isolation Pilot Plant (WIPP) Site, Southeastern New Mexico," *SAND78-1596*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Richey, S.F., 1987. "Water-Level Data from Wells in the Vicinity of the Waste Isolation Pilot Plant, Southeastern New Mexico," *Open File Report 87-120*, Albuquerque, New Mexico.

Siegel et al., 1991.

Stensrud, W.A., M.A. Barne, K.D. Lantz, T.L. Cauffman, J.B. Palmer, G.J. Saulnier, 1988. "WIPP Hydrology Program, Waste Isolation Pilot Plant, Southeastern New Mexico," *SAND87-7166*, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Stensrud, W.A., 1995. "Culebra Transport Program Test Plan: Hydraulic Tests at Wells WQSP-1, WQSP-2, WQSP-3, WQSP-4, WQSP-5, WQSP-6, WQSP-6A at the Waste Isolation Pilot Plant (WIPP) Site."

U.S. Department of Energy (DOE), 1996. "United States Department of Energy Waste Isolation Pilot Plant Compliance Certification Application," *DOE/CAO-1996–2184*.

U.S. Environmental Protection Agency (EPA), 1990. "Background Documentation for the U.S. Environmental Protection Agency's Proposed Decision on the No-Migration Variance for U.S. Department of Energy's Waste Isolation Pilot Plant," U.S. Environmental Protection Agency, Washington, D.C.

U.S. Environmental Protection Agency (EPA), 1986a. "RCRA Ground-Water Monitoring Technical Enforcement Guidance Document," U.S. Environmental Protection Agency, Washington, D.C.

U.S. Environmental Protection Agency (EPA), 1986b. "Test Methods for Evaluating Solid Waste," *SW-846*, third ed., Office of Solid Waste and Emergency Response, Washington, D.C.

Westinghouse Electric Corporation, 1996. "WID Quality Assurance Program Description," *WP13-1*, Westinghouse Electric Corporation, Waste Isolation Division, Carlsbad, New Mexico.

Westinghouse Electric Corporation, 1991. "Waste Isolation Pilot Plant Groundwater Monitoring Program Plan and Procedures Manual," *WP02-01*, Waste Isolation Division (WID), Carlsbad, New Mexico.

Westinghouse Electric Corporation, 1987. "Waste Isolation Pilot Plant Water Quality Sampling Manual," *WP7-2*, Waste Isolation Division, Carlsbad, New Mexico.

TABLES

This page left blank intentionally.

TABLE D18-1
HYDROLOGICAL PARAMETERS FOR ROCK UNITS ABOVE THE SALADO AT WIPP

Unit		Hydraulic Conductivity	Storage Coefficient	Transmissivity	Permeability	Thickness	Hydraulic Gradient
Santa Rosa		2×10^{-8} to 2×10^{-6} m/s (1) (2)	Specific capacity 0.029 to 0.041 liters/sec/meters	6×10^{-7} to 6×10^{-5} m^2/s (3)	10^{-10} m^2	0 to 91 m	0.001 (5)
Dewey Lake		10^{-8} m/s	Specific storage 1×10^{-5} (1/m) (2)	2.8×10^{-6} to 2.8×10^{-4} m^2/s (4)	5.01×10^{-17} m^2	152 m (2.1.3.6)	0.001 (5)
Rustler	Forty-niner	1×10^{-13} to 1×10^{-11} m/s (anhydrite) 1×10^{-9} m/s (mudstone) (2)	Specific storage 1×10^{-5} (1/m) (2)	8×10^{-8} to 8×10^{-9} m^2/s	0 m^2	13 to 23 m	NA (6)
	Magenta	$1 \times 10^{-8.5}$ to $1 \times 10^{-6.5}$ m/s (2)	Specific storage 1×10^{-5} (1/m) (2)	4×10^{-4} to 1×10^{-9} m^2/s	6.31×10^{-14} m^2	7 to 8.5 m	3 to 6 m/km (2.2.1.4.1.4)
	Tamarisk	1×10^{-13} to 1×10^{-11} m/s (anhydrite) 1×10^{-9} m/s (mudstone) (2)	Specific storage 1×10^{-5} (1/m) (2)	$<2.7 \times 10^{-11}$ m^2/s	0 m^2	26 to 56 m	NA (6)
	Culebra	$1 \times 10^{-7.5}$ to $1 \times 10^{-5.5}$ m/s (2)	Specific storage 1×10^{-5} (1/m) (2)	1×10^{-3} to 1×10^{-9} m^2/s	2.1×10^{-14} m^2	4 to 11.6 m	0.003 to 0.007 (Figure 2-31) (5)
	Unnamed lower member	6×10^{-15} to 1×10^{-13} m/s 1.5×10^{-11} to 1.2×10^{-11} m/s (basal interval)	Specific storage 1×10^{-5} (1/m) (2)	2.9×10^{-10} to 2.2×10^{-13} m^2/s 2.9×10^{-10} to 2.4×10^{-10} m^2/s (basal interval)	0 m^2	29 to 38 m	NA (6)

TABLE D18-1 (Continued)
HYDROLOGICAL PARAMETERS FOR ROCK UNITS ABOVE THE SALADO AT WIPP

Table Notes:

(1) The Santa Rosa Formation is not present in the western portion of the WIPP site. It was combined with the Dewey Lake Red Beds in three-dimensional regional groundwater flow modeling (Corbet and Knupp, 1996), and the range of values entered here are those used in that study for the Dewey Lake/Triassic hydrostratigraphic unit.

(2) Values or ranges of values given for these entries are the values used in three-dimensional regional groundwater flow modeling (Corbet and Knupp, 1996). Values are estimated based on literature values for similar rock types, adjusted to be consistent with site-specific data where available. Ranges of values include spatial variation over the WIPP site and differences in values used in different simulations to test model sensitivity to the parameter.

(3) The range of values given here for transmissivity of the Santa Rosa is estimated for the center of the site. Transmissivity is the product of the thickness of the productive interval times its hydraulic conductivity. Thickness of the Santa Rosa is estimated to be 30 meters at the center of the WIPP site, and the range of derived transmissivities are based on the range of hydraulic conductivity values used by Corbet and Knupp (1996) for the combined Dewey Lake/Triassic unit.

(4) The range of values given here by transmissivity of the Dewey Lake is estimated for the center of the site. Transmissivity is the product of the thickness of the productive interval times its hydraulic conductivity. Thickness of the Dewey Lake is estimated to be 140 meters at the center of the WIPP site (CCA Figure 2-14), and the range of derived transmissivities are based on the range of hydraulic conductivity values used by Corbet and Knupp (1996) for the combined Dewey Lake/Triassic unit.

(5) Hydraulic gradient is a dimensionless term describing change in the elevation of hydraulic head divided by change in horizontal distance. Values given in these entries are determined from potentiometric surfaces. The range of values given for the Culebra reflects the highest and lowest gradients observed within the WIPP site boundary. Values for the Dewey Lake and Santa Rosa are assumed to be the same as the gradient determined from the water table. Note that the Santa Rosa Formation is absent or above the water table in most of the controlled area, and that the concept of a horizontal hydraulic gradient is not meaningful for these regions.

(6) Flow in units of very low hydraulic conductivity is slow, and primarily vertical. The concept of a horizontal hydraulic gradient is not applicable.

(7) These units are modeled as single-porosity porous media in the three-dimensional regional model (Corbet and Knupp, 1996). Matrix characteristics are described by standard hydraulic parameters (e.g., hydraulic conductivity, porosity). Fractures within these units are assumed for modeling purposes to have no effect on regional flow.

Sources: Beauheim, 1986; Domenico and Schwartz, 1990; Domski, Upton, and Beauheim, 1966; Earlough, 1977.

TABLE D18-2
WIPP GROUNDWATER MONITORING PROGRAM
SAMPLE COLLECTION AND WATER-LEVEL MEASUREMENT FREQUENCY

Installation	Frequency
Water Quality Sampling	
All WIPP surveillance wells	On special request only
GMP monitoring wells	Semiannually
Water-Level Monitoring	
All WIPP surveillance wells	Monthly
GMP monitoring wells	Monthly and prior to sampling events

TABLE D18-3
ANALYTICAL PARAMETER LIST FOR THE WIPP DETECTION MONITORING PROGRAM

Background Water Quality	Operational Detection Monitoring Water Quality
<u>Indicator Parameters</u> pH, SC, TOC, TOX <u>Parameters Listed in</u> 20 NMAC 4.1 Subpart V 264, Appendix IX <u>Field Analyses</u> pH, SC, temperature, chloride, Eh, alkalinity, total Fe	<u>Indicator Parameters</u> pH, SC, TOC, TOX <u>Organic Parameters</u> Carbon tetrachloride Chlorobenzene 1,1-dichloroethylene 1,1-dichloroethane Methylene chloride 1,1,2,2-tetrachloroethane Toluene 1,1,1-trichloroethane <u>Metals</u> TCLP metals Arsenic Barium Cadmium Chromium Lead Mercury Selenium Silver <u>Field Analyses</u> pH, SC, temperature, chloride, Eh, alkalinity, total Fe

FIGURES

This page left blank intentionally.

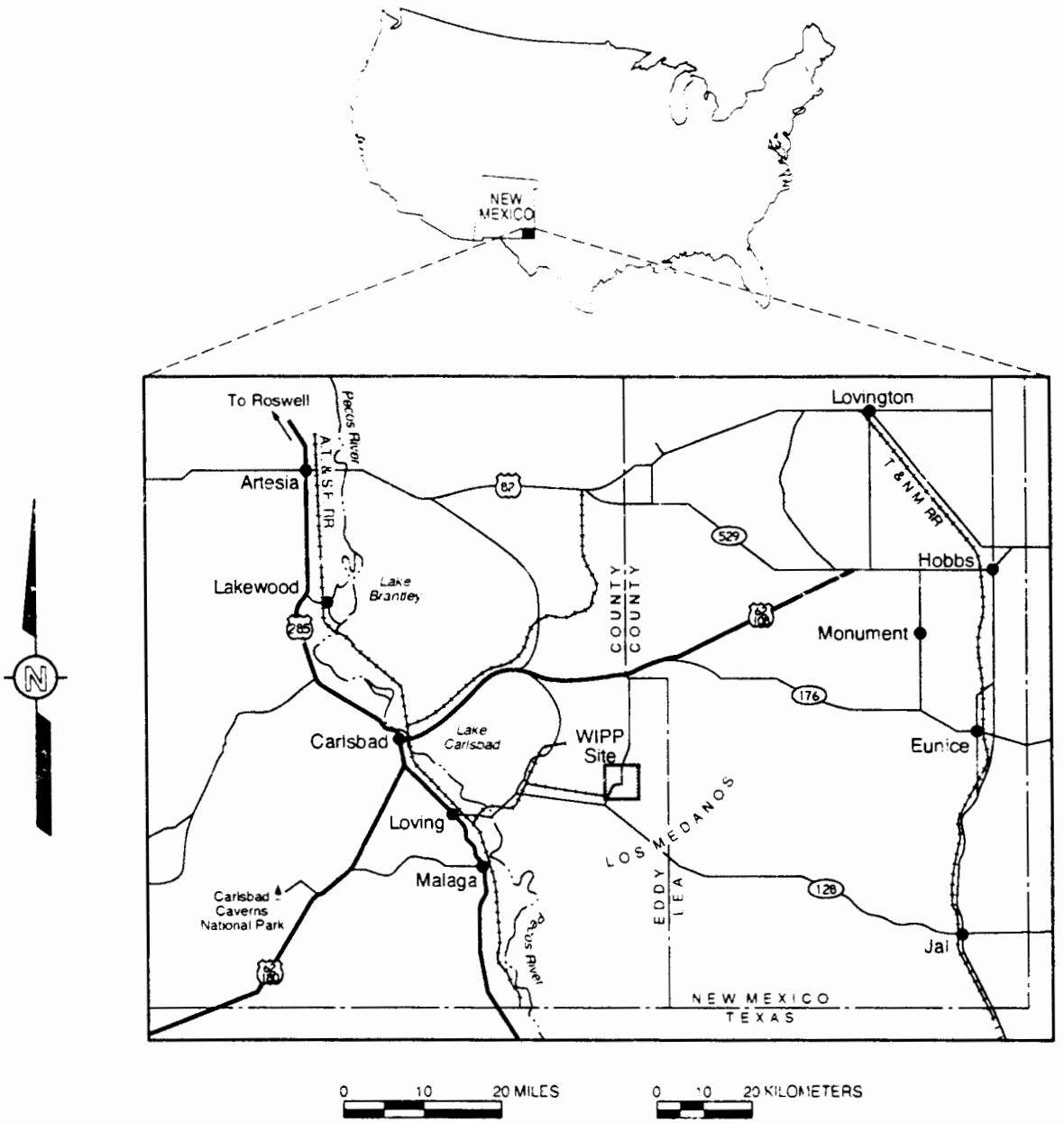


Figure D18-1
General Location of the WIPP Facility

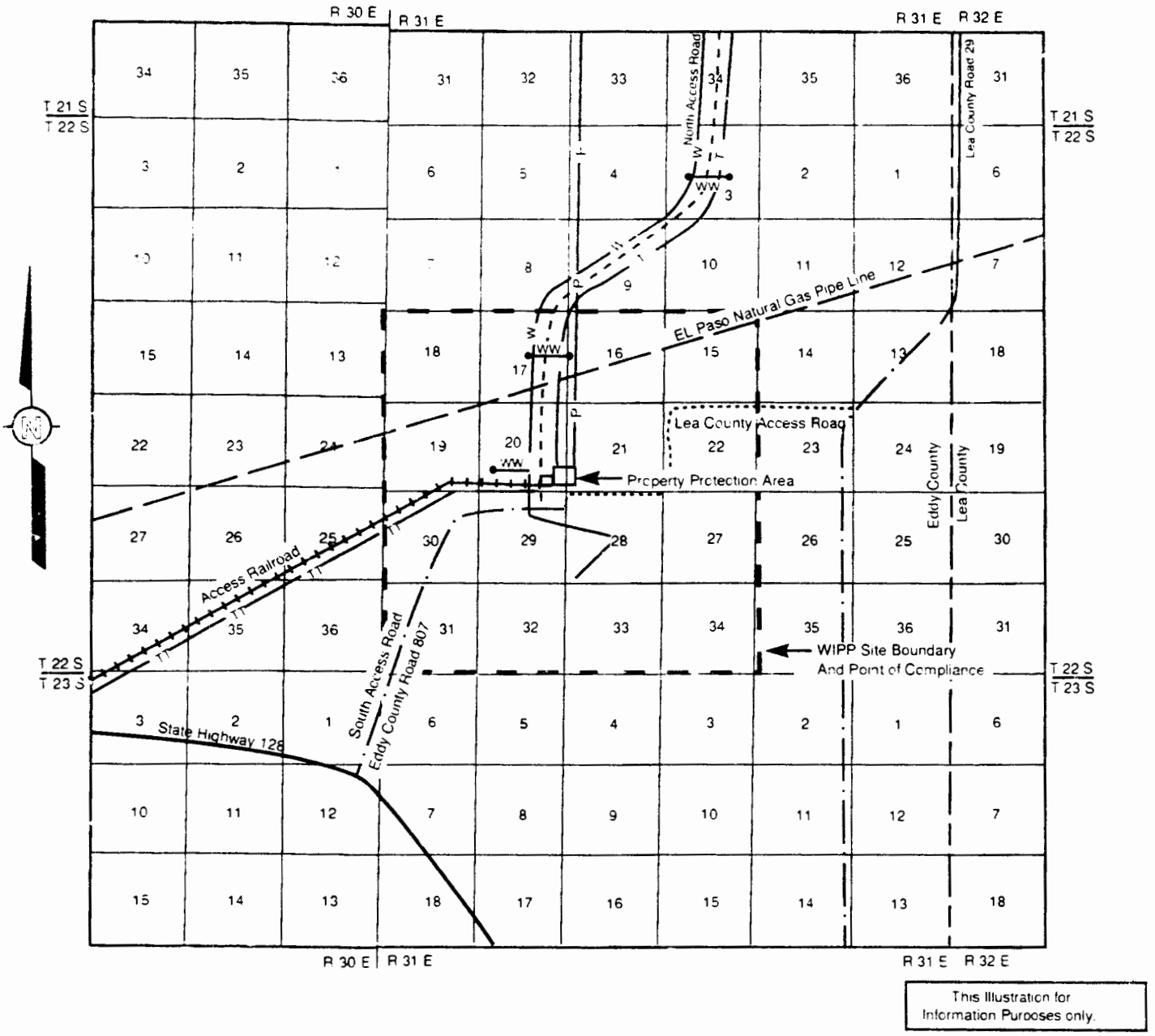


Figure D18-2

WIPP Facility Boundary Showing 16 Square-Mile Land Withdrawal Boundary and Point of Compliance

System	Series	Group	Formation	Member
Recent	Recent		Surficial Deposits	
Quaternary	Pliestocene		Mescalero Caliche	
			Gatuña	
Tertiary	Mid-Pliocene		Ogallala	
Triassic		Dockum	Santa Rosa	
Permian	Ochoan		Dewey Lake	
			Rustler	
			Salado	Upper Potash Lower
			Castile	
	Guadalupian	Delaware Mountain	Bell Canyon	
			Cherry Canyon	
			Brushy Canyon	

Figure D18-3
Site Geologic Column

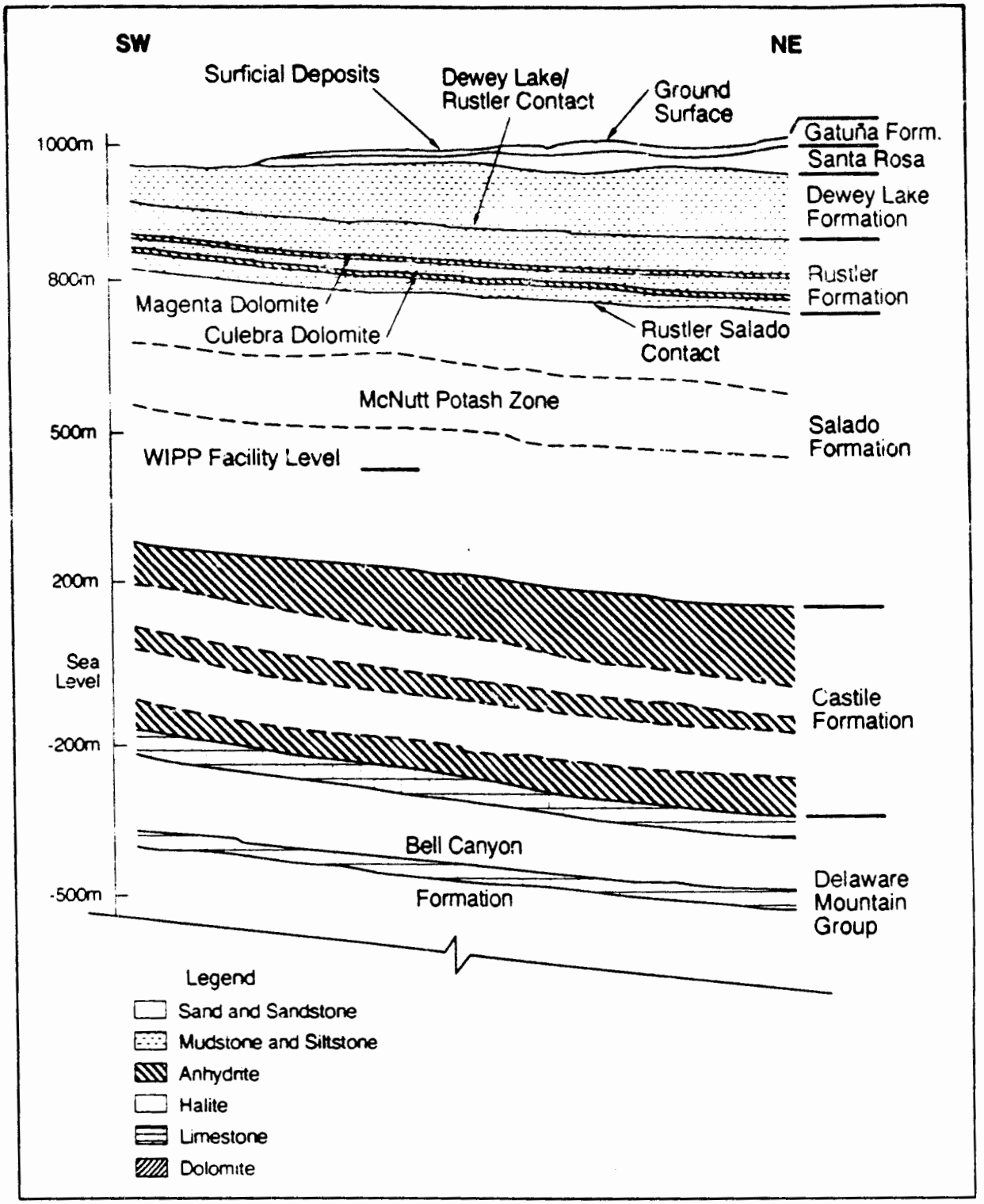


Figure D18-4
Generalized Stratigraphic Cross Section above Bell Canyon Formation at WIPP Site

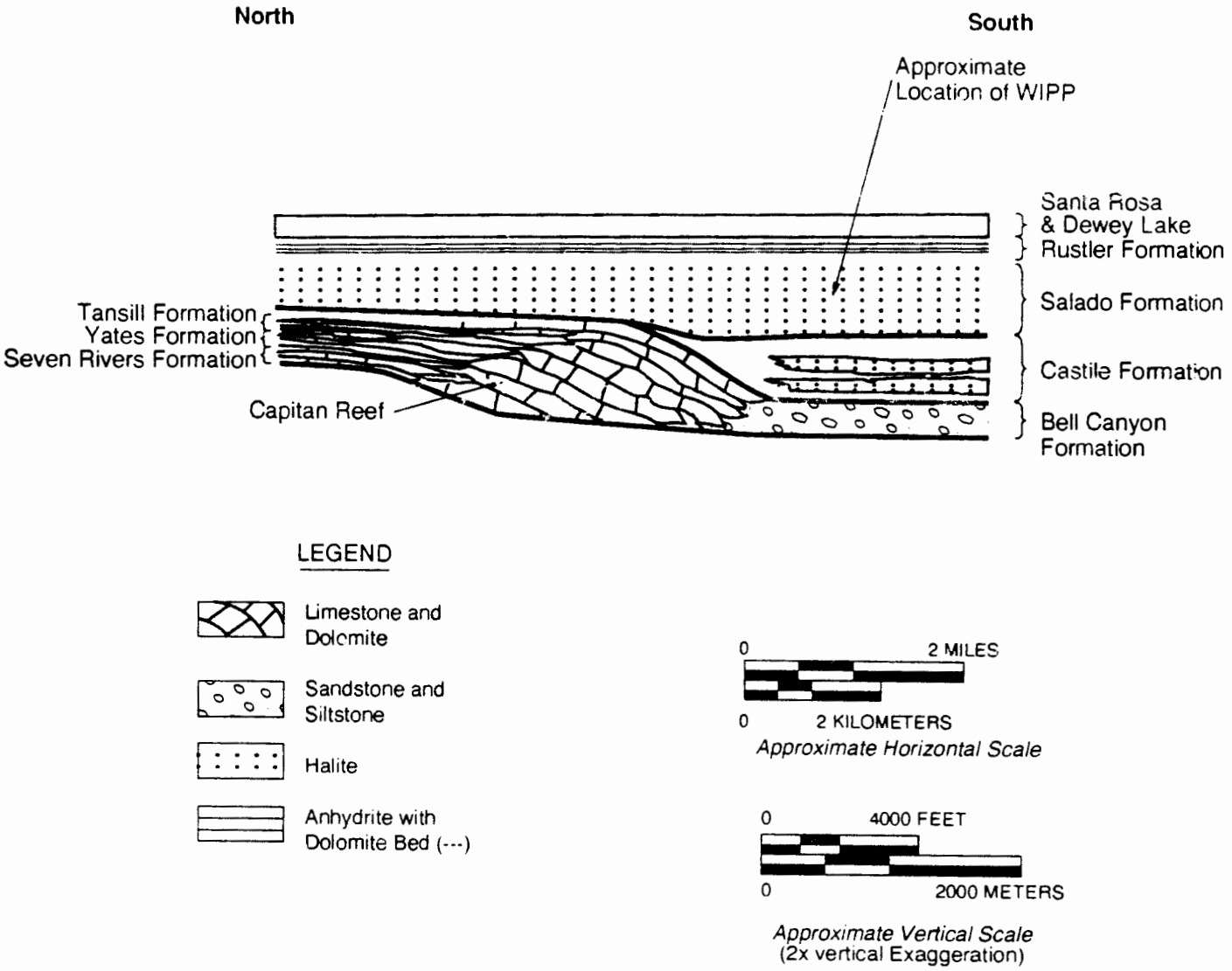
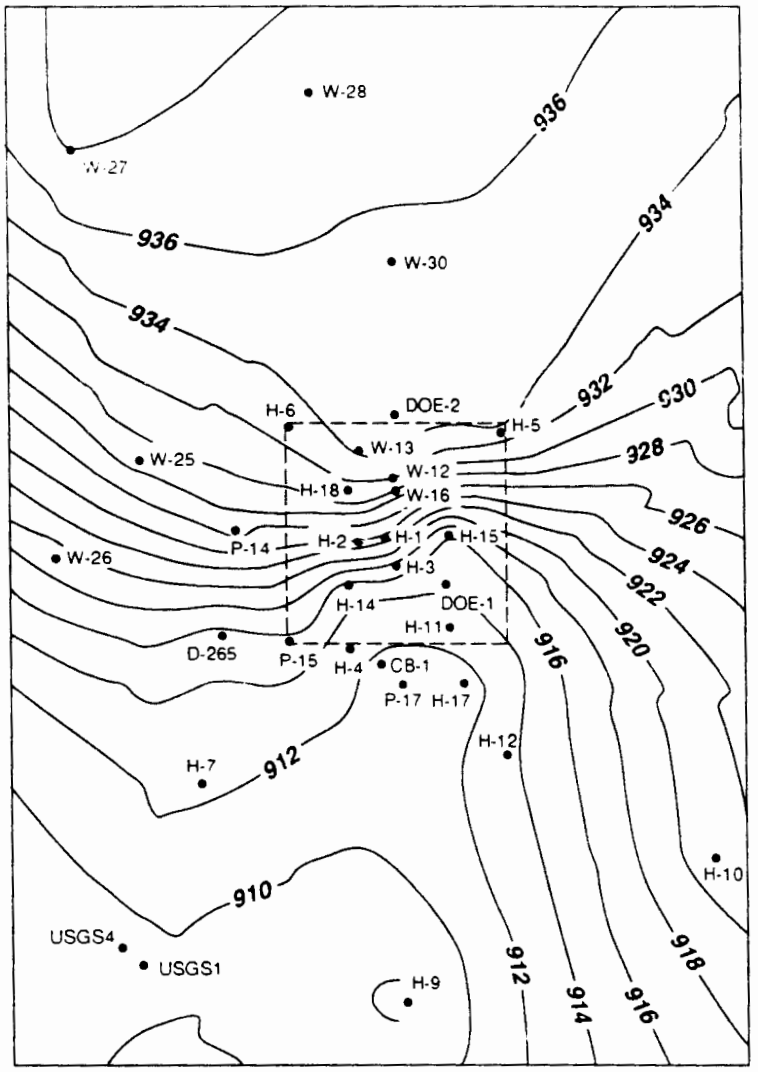


Figure D18-5
Schematic North-South Cross-Section through the North Delaware Basin



Source: Jones et al. 1992, Figure 2-5

• Observation Well

Freshwater Heads in m amsl

Contour Interval: 2m

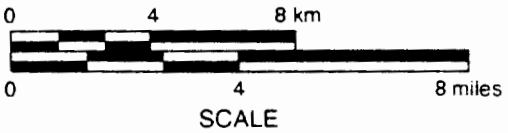


Figure D18-6
Culebra Freshwater-Head Contour Surface

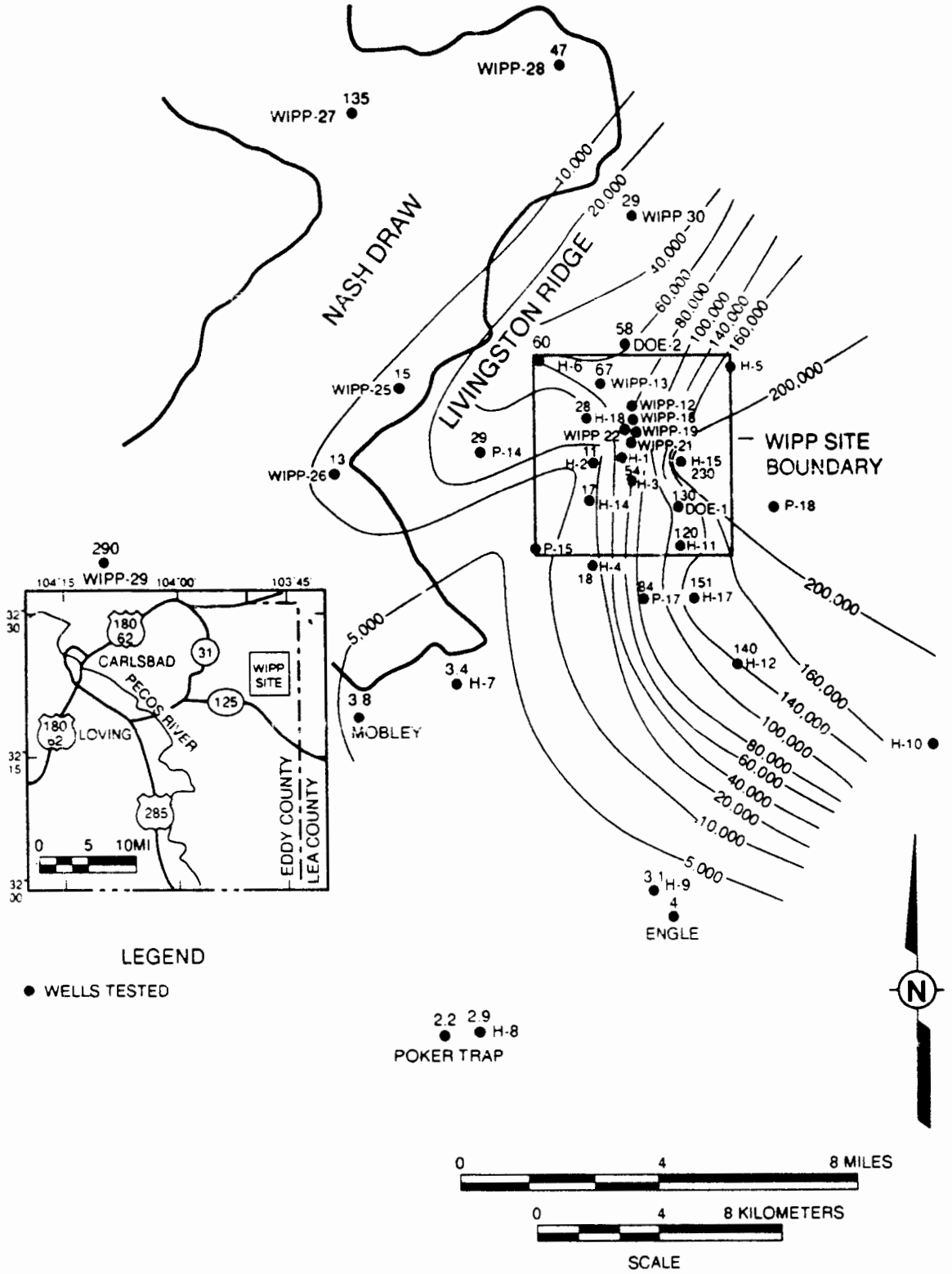


Figure 18-7
Total Dissolved Solids Distribution in the Culebra

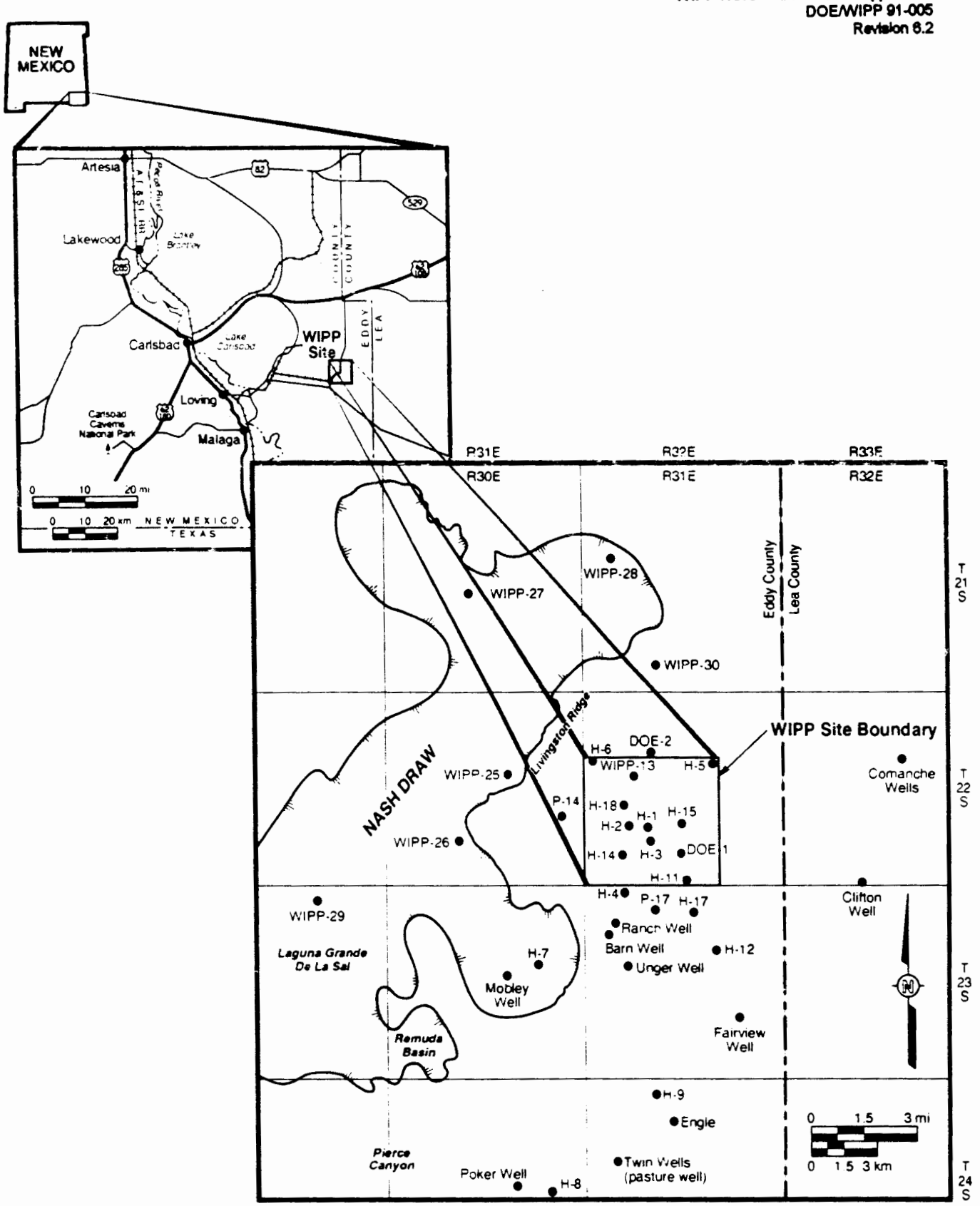
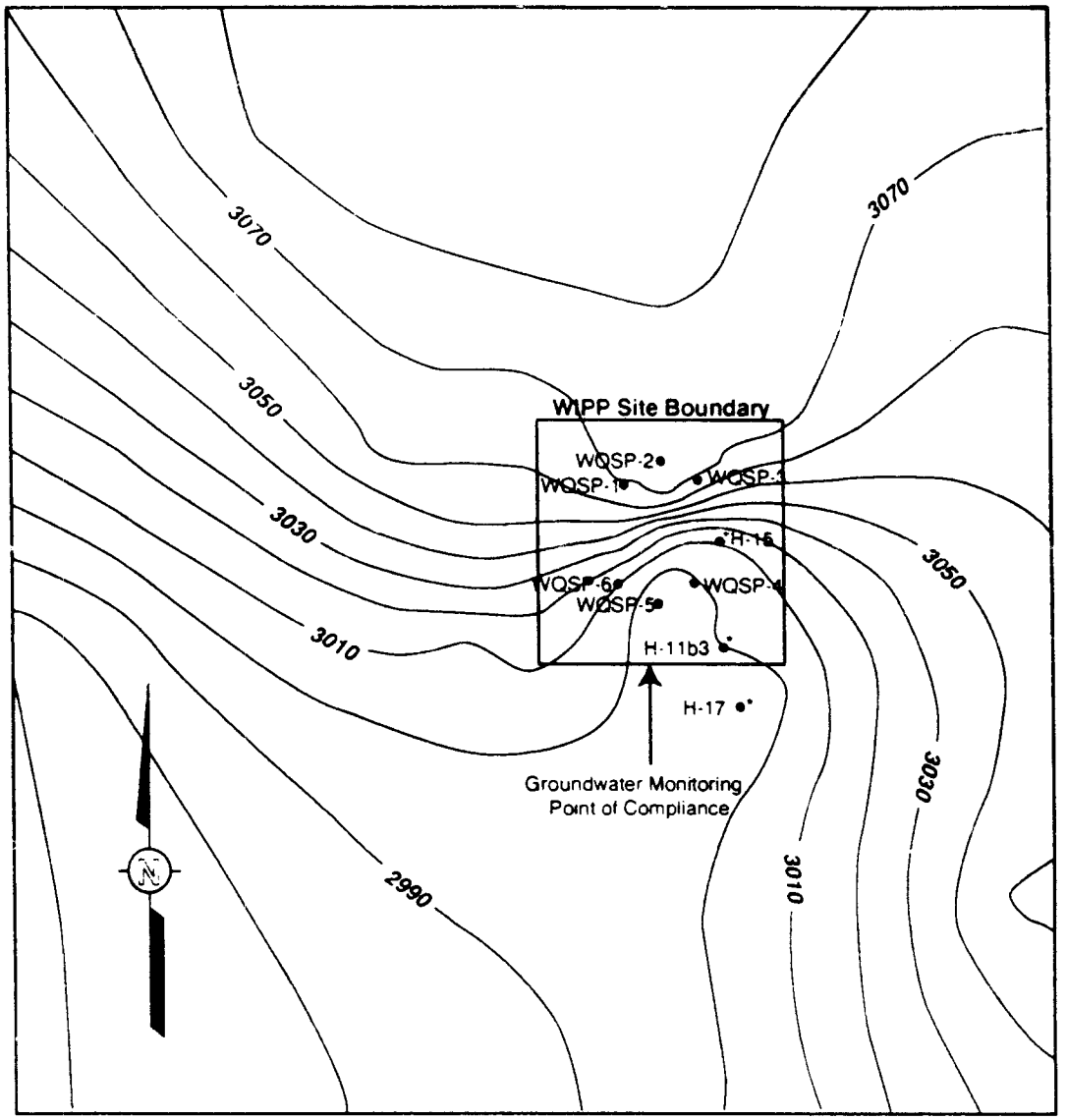


Figure D18-8
WQSP Monitor Well Locations



Note: Contour elevations are in feet above mean sea level

*The Wells are included for reference only–they are not part of GMP

Figure D18-9

WIPP GMP Monitor Well Locations and Potentiometric Surface of the Culebra Dolomite Member of the Rustler Formation Near the WIPP Site as of 12/96 (adjusted to equivalent freshwater head)

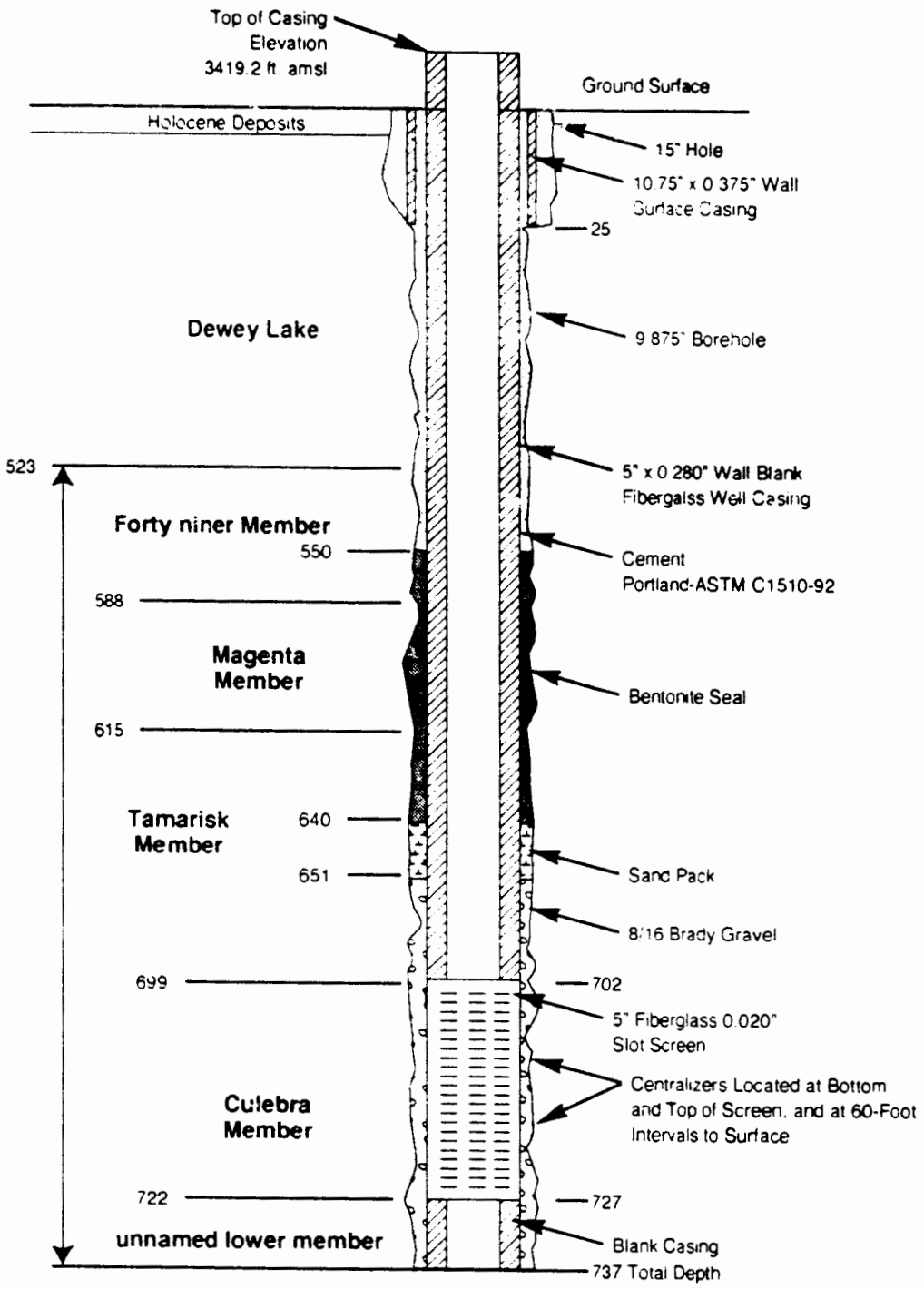
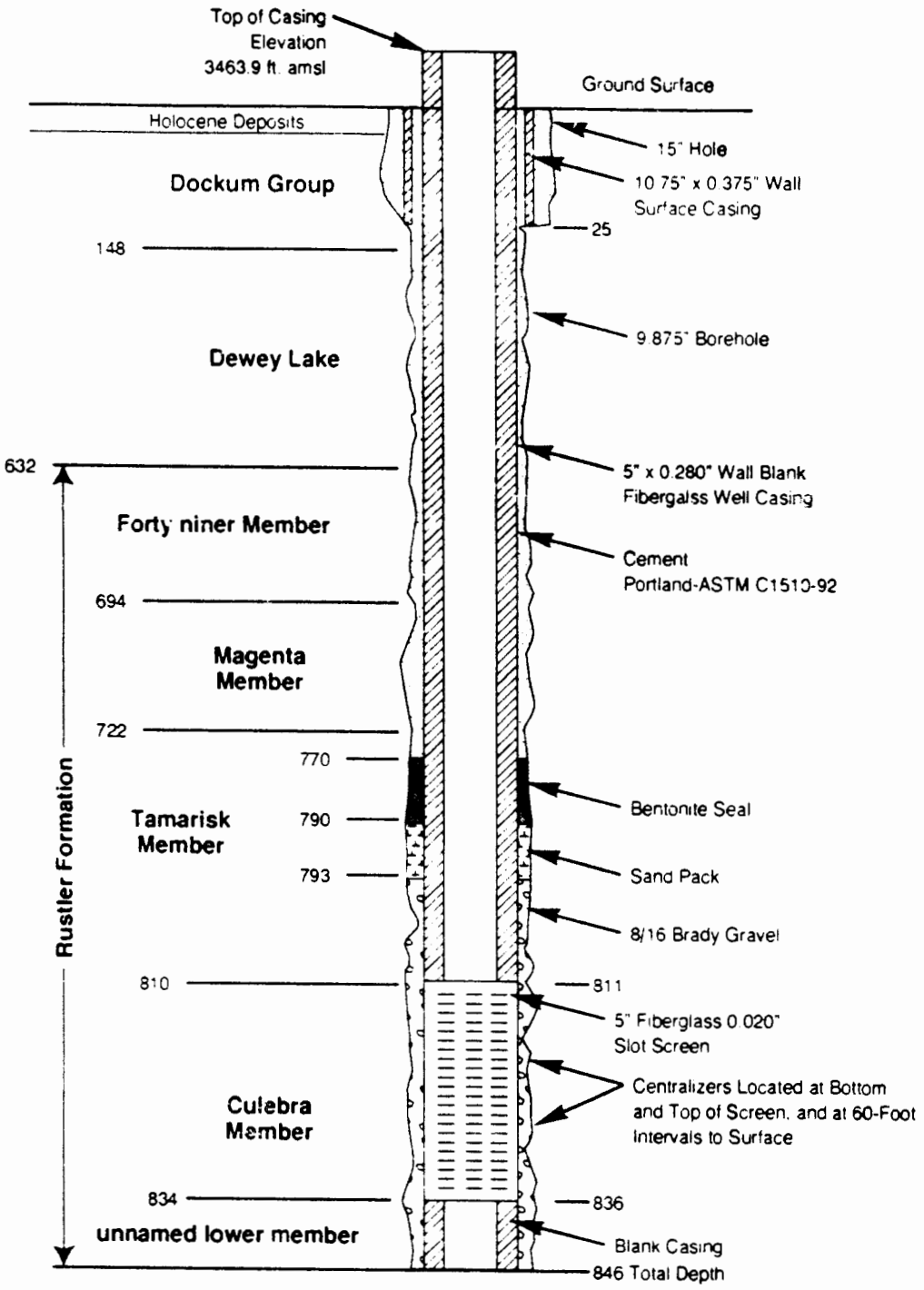
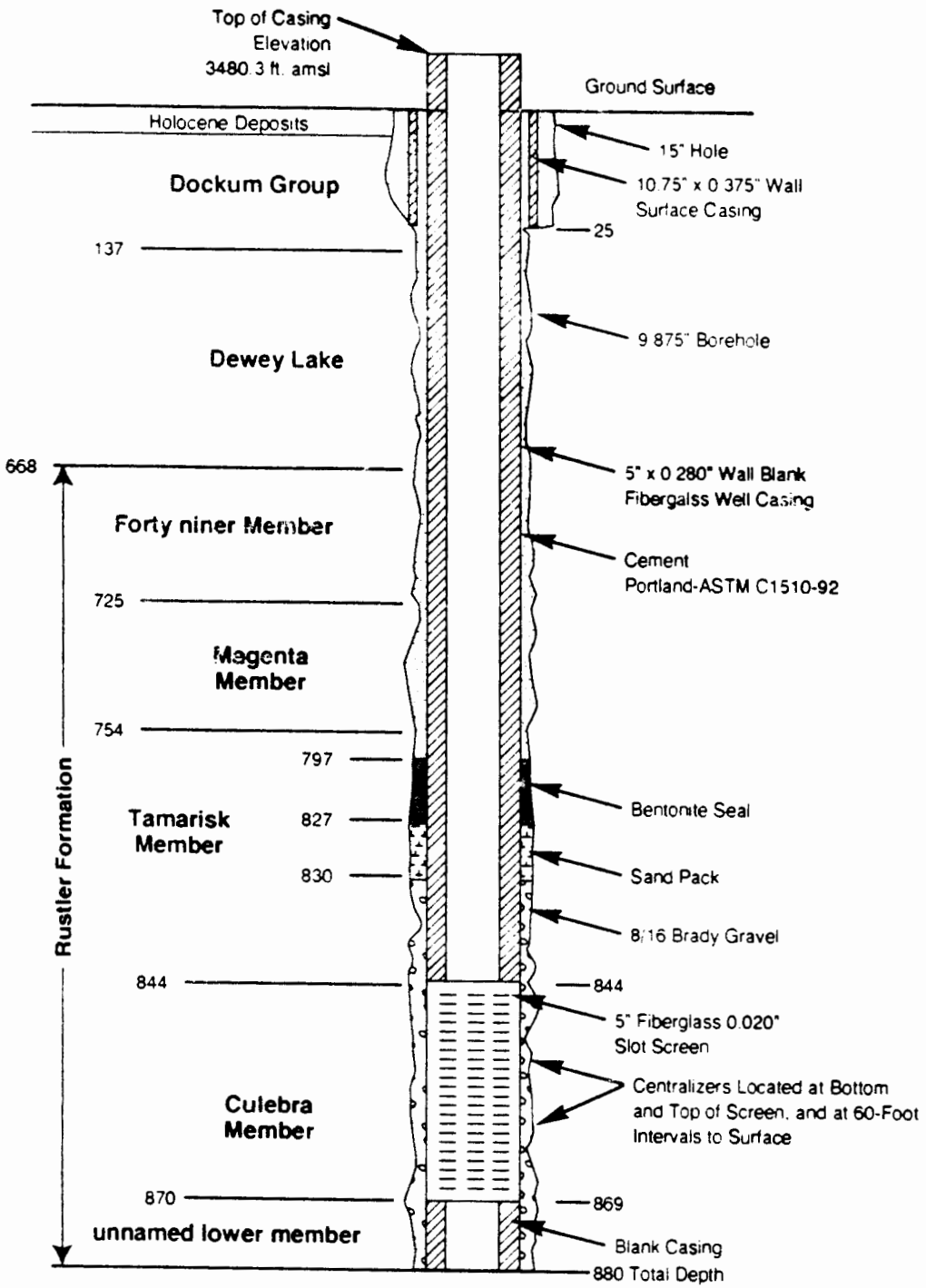


Figure D18-10
As-Built Configuration of Well WQSP-1



Note: Depths in feet bgs approximate
Not to Scale

Figure D18-11
As-Built Configuration of Well WQSP-2



Note: Depths in feet bgs approximate
Not to Scale

Figure D18-12
As-Built Configuration of Well WQSP-3

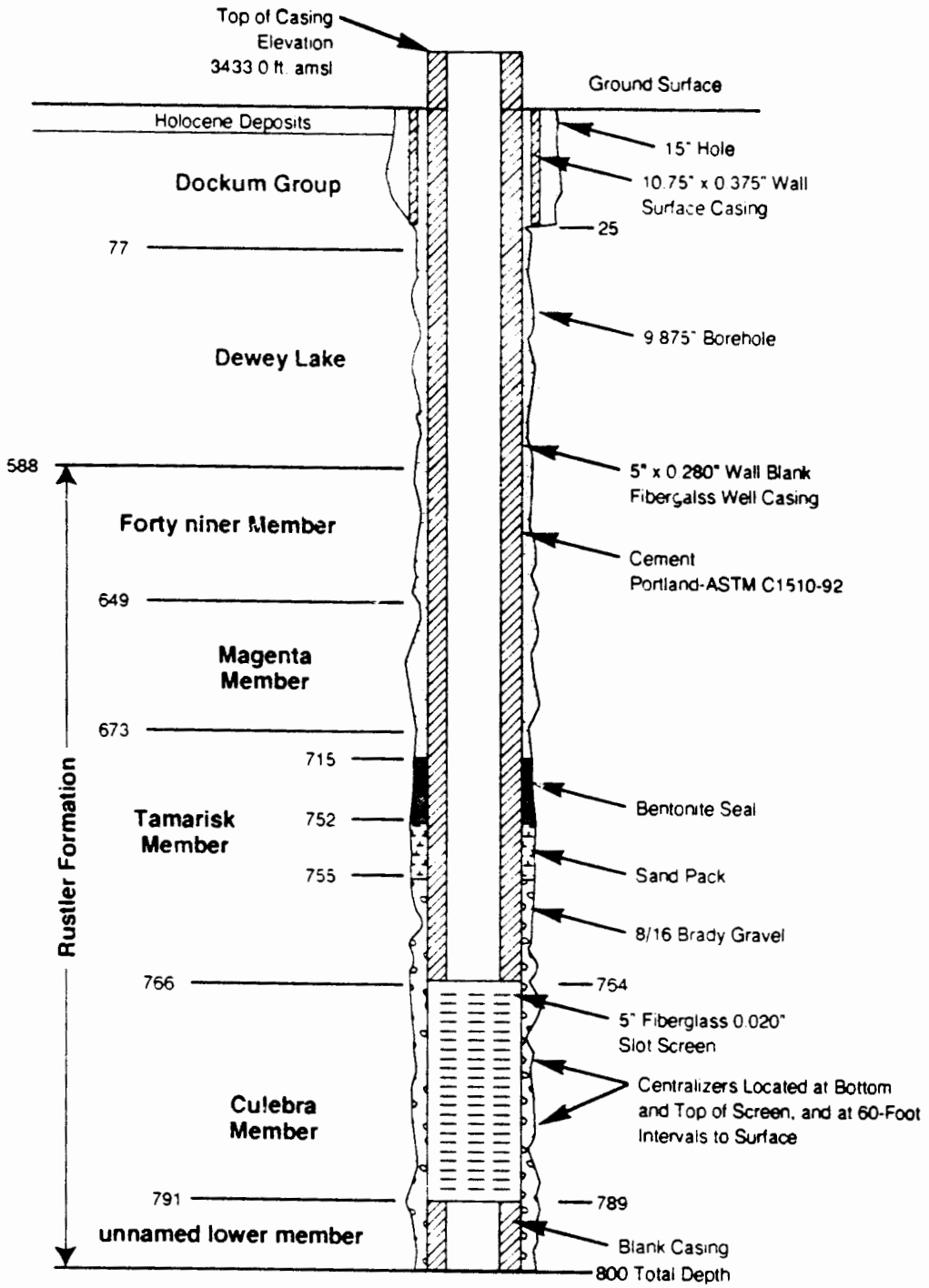


Figure D18-13
As-Built Configuration of Well WQSP-4

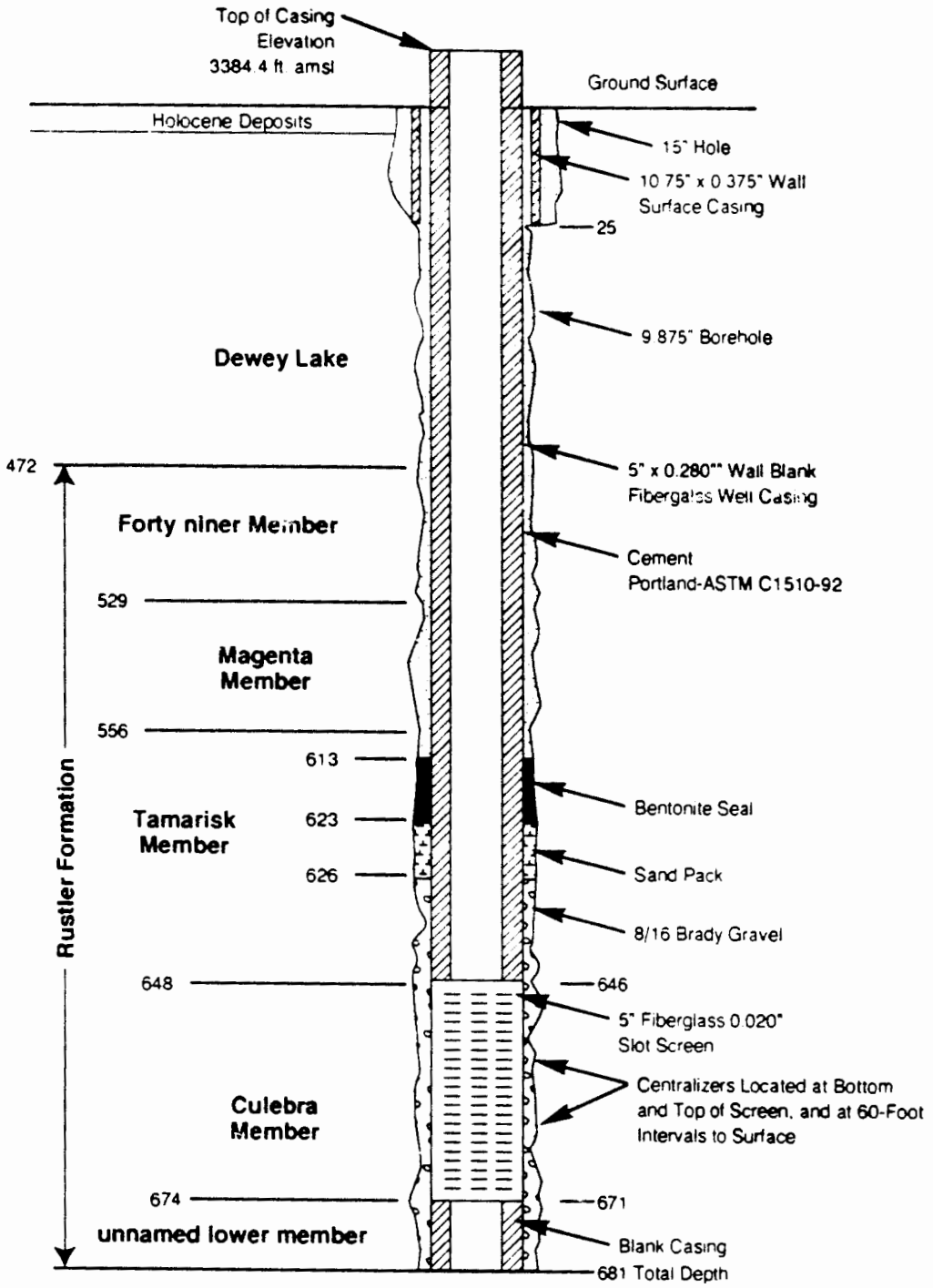
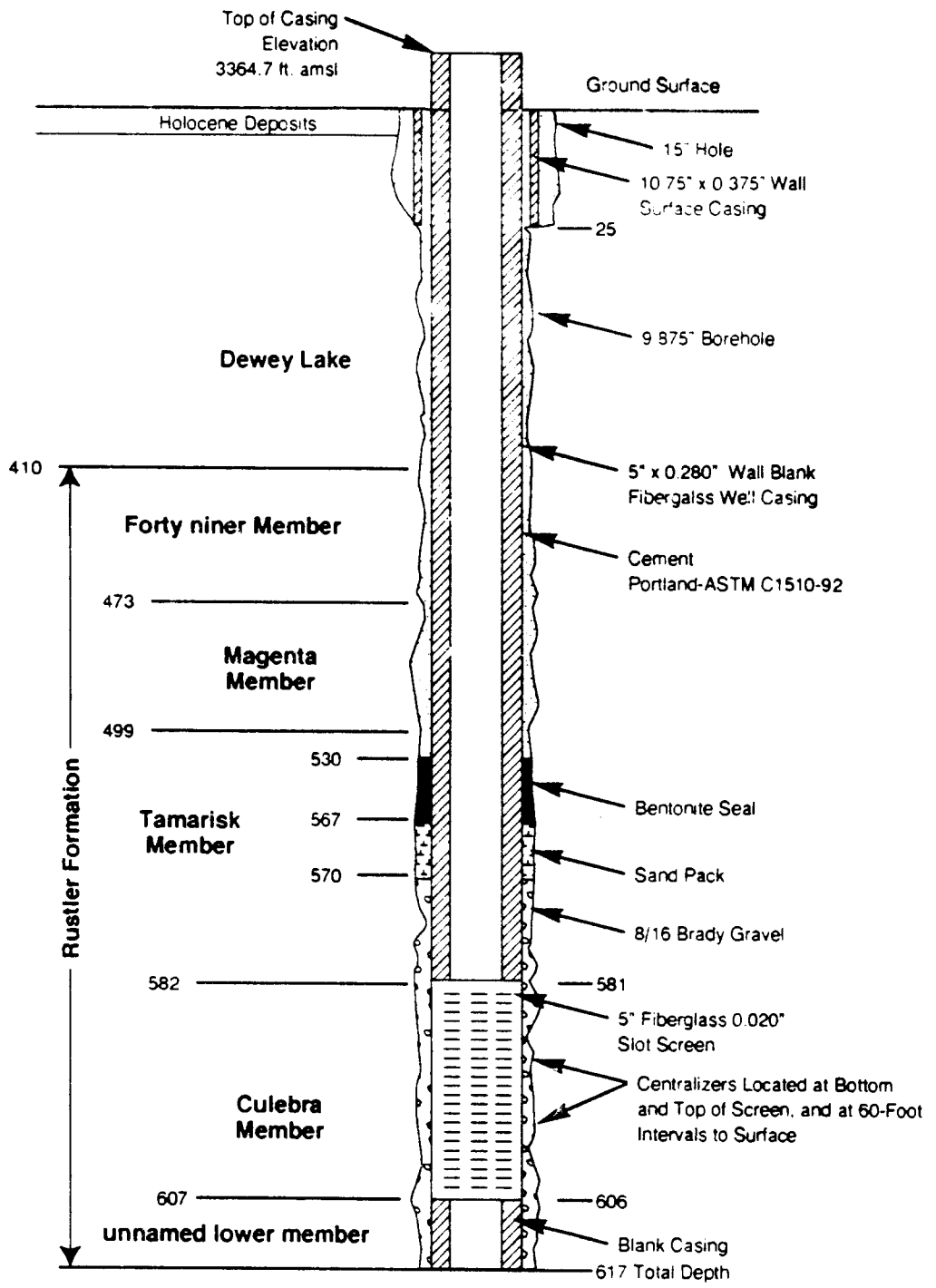


Figure D18-14
As-Built Configuration of Well WQSP-5



Note: Depths in feet bgs approximate
Not to Scale

Figure D18-15
As-Built Configuration of Well WQSP-6

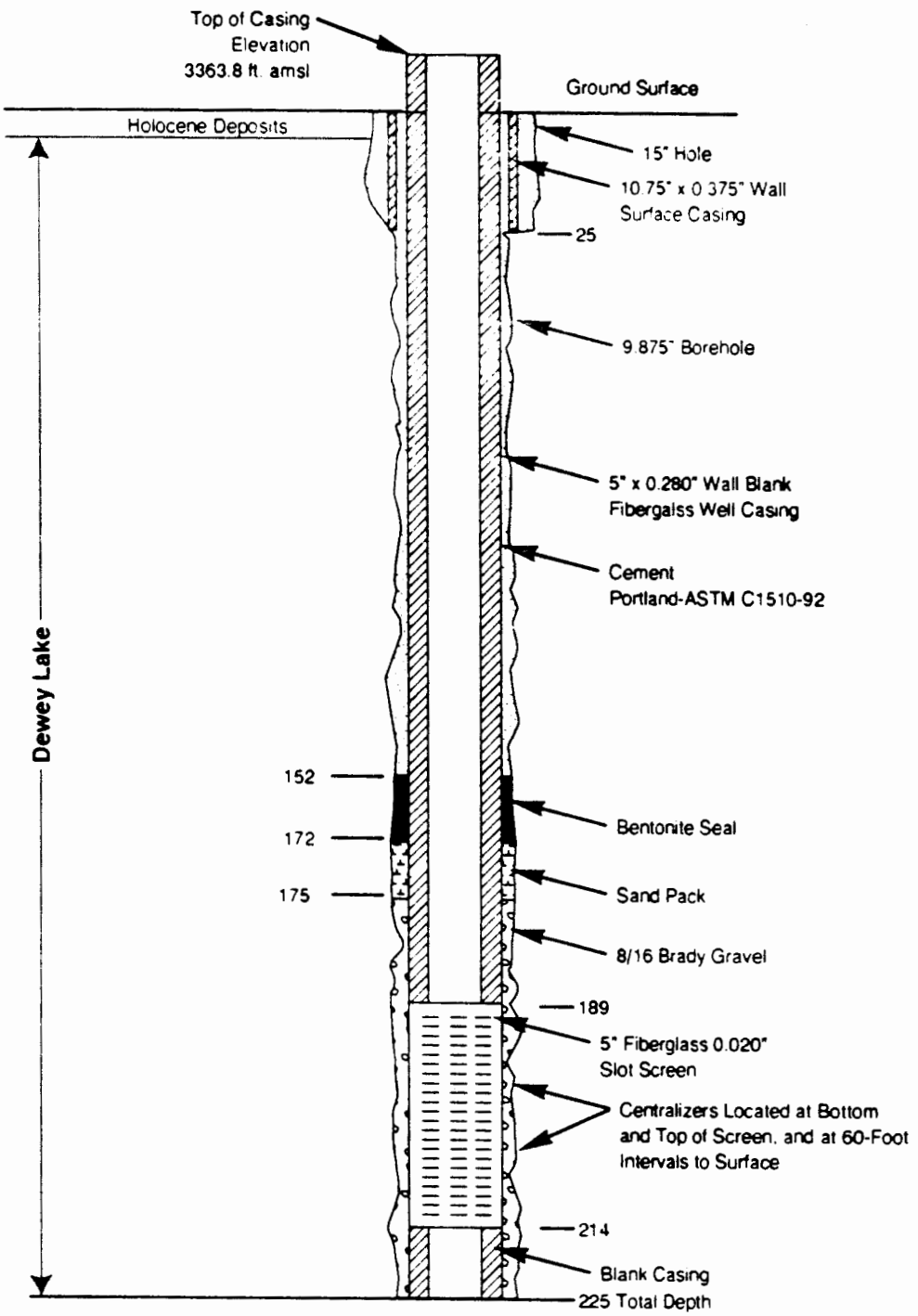


Figure D18-16
As-Built Configuration of Well WQSP-6A

Chain of Custody Record

WASTE ISOLATION PILOT PLANT
WESTINGHOUSE ELECTRIC CORP.
P.O. BOX 2078
CARLSBAD, NM 88221-2078

C of C Control No. _____

RFA Control No. _____

SAMPLING PROGRAM _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No.

Special Instructions _____

Possible Sample Hazards: _____

Signatures: (Name, Company, Date and Time: _____

1. Relinquished By: _____
   Received By: _____
2. Relinquished By: _____
   Received By: _____
3. Relinquished By: _____
   Received By: _____
4. Relinquished By: _____
   Received By: _____

WHITE-Original, to accompany samples YELLOW-Field Copy PINK-Other

Figure D18-17a
Chain of Custody Record

REQUEST FOR ANALYSIS

RFA Control No. _____

C of C Control No. _____

WASTE ISOLATION PILOT PLANT
WESTINGHOUSE ELECTRIC CORP.
P.O. BOX 2078
CARLSBAD, NM 88221-2078

DATE SAMPLES SHIPPED _____

LAB DESTINATION _____

LABORATORY CONTRACT _____

SEND LAB REPORT TO _____

_____

DATE REPORT REQUIRED _____

SAMPLING PROGRAM _____

PROJECT CONTACT _____

SAMPLE TEAM MEMBERS _____

PROJECT CONTACT PHONE NO. _____

Sample Number	Sample Type	Sample Quantity	Preservative	Req't Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by appropriate Manger) NORMAL ___ RUSH ___ (Subject to rush Surcharge)
POSSIBLE HAZARD IDENTIFICATION (Please indict if samples are hazardous materials and/or suspected to contain high levels of hazardous substance.)
NONHAZARD___ FLAMMABLE___ SKIN IRRITANT_____ HIGHLY TOXIC_____ BIOLOGICAL_____ OTHER_____
SAMPLE DISPOSAL: (Please indicate disposition of sample following analysis.) RETURN TO CLIENT_____ DISPOSAL BY LAB_____

FOR LAB USE ONLY_____
RECEIVED BY:_____ DATE/TIME_____

WHITE-Original, to accompany samples YELLOW-Field Copy PINK-Other

Figure D18-17b
Request for Analysis

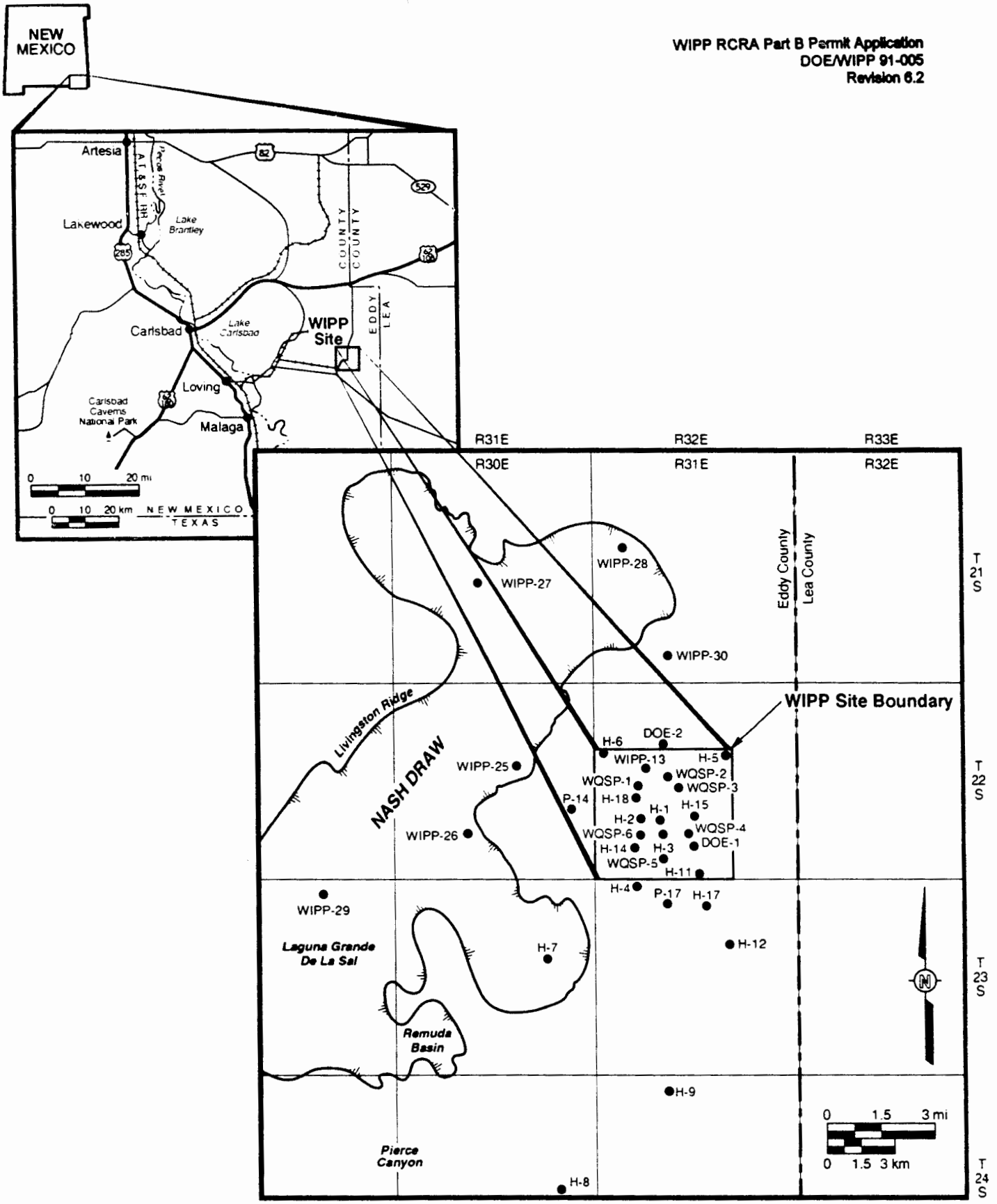


Figure D18-18
Water Level Monitoring Locations

ATTACHMENT A

WIPP PROCEDURES FOR SAMPLE COLLECTION, WATER-LEVEL MONITORING, QA/QC, AND RECORDS CONTROL

WP 02-EM3001

Revision 0

Administrative Processes for Environmental Monitoring Programs

Management Control Procedure

EFFECTIVE DATE: 14 MARCH 97

SIGNATURE	PRINTED NAME	DATE
	KEVIN S. DONOVAN	3/13/97

APPROVED FOR USE

REVIEW ORGANIZATIONS

Environmental Compliance & Support	Environmental Monitoring	Hazardous Waste Operations
Radiological Control	Quality & Regulatory Assurance	RCRA Permitting
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A

CONTROLLED COPY

TABLE OF CONTENTS

INTRODUCTION 3

REFERENCES 3

PRECAUTIONS AND LIMITATIONS 4

PERFORMANCE 4
1.0 QUALITY ASSURANCE/QUALITY CONTROL IMPLEMENTATION. 4
1.1 Data Sheet Verification. 4
1.2 Computer Input Verification 5
2.0 DATA SHEET OR SAMPLE TRACKING LOG BOOK (STLB) PREPARATION . . 5
3.0 SAMPLE TRACKING 6
3.1 Sample Holding 6
3.2 Samples Sent to Off-Site Analytical Laboratory 7
3.3 Samples Analyzed On-Site 7
3.4 Assigning Unique Sample Identity Codes 8
3.5 Assigning Unique Sample Identity Codes for Water Quality Sampling Program (WQSP) Final Samples 10
3.6 Assigning Unique Sample Identity Codes to Composite Samples 11
4.0 RESPONSE TO SAMPLE DATA RESULTS ABOVE REGULATORY LIMITS . . 12

Attachment 1 - Example Sample Tracking Log Book 13

Attachment 2 - Example Chain-of-Custody Record 14

Attachment 3 - Example Request for Analysis 15

INTRODUCTION

This procedure provides the administrative guidance Environmental Monitoring (EM) personnel use to maintain Quality Control (QC) associated with EM sampling activities, and to assure the validity of results.

Environmental monitoring programs that have been identified in the RCRA Part B permit application and procedures for these programs apply to RCRA-related activities and equipment.

EM Manager will assure that an Annual Environmental Sampling Schedule for all EM programs is developed and maintained. This schedule will be updated as necessary to assure that the overall objectives of the program are achieved.

One or more of the following records are generated by the use of this procedure:

- Attachment 2, Chain of Custody (C of C)
- Attachment 3, Request for Analysis (RFA)
- Acknowledgment of Receipt (AOR)
- Data sheets
- Log books
- Check-prints

REFERENCES

BASELINE

- DOE/WIPP 96-2194, Waste Isolation Pilot Plant Environmental Monitoring Plan

COMPLIANCE

- DOE/WIPP 91-005, RCRA Part B Permit (Rev. 6) Application
- DOE/EH-0173T, Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance
- WP 13-1, WID Quality Assurance Program Description

PRECAUTIONS AND LIMITATIONS

- Only personnel qualified in accordance with EM qualification cards or under the direct supervision of a qualified individual may perform technical procedures directly related to this procedure..
- Exercise extreme care in following sampling protocols to prevent contamination of samples.
- Sample hold times and perishable, packaging, or preservation shall be followed in accordance with analytical laboratories recommendations.

PERFORMANCE

1.0 QUALITY ASSURANCE/QUALITY CONTROL IMPLEMENTATION.

1.1 Data Sheet Verification.

NOTE

Data sheets will have a specific section, or the stamping "CHECK-PRINT" designated on them, for the reviewer to sign or initial, and date.

Unverified data used outside EM for information only must be marked "INFORMATION ONLY," "PRELIMINARY," or "DRAFT."

The reviewer will not be the person who recorded the original entries.

1.1.1 Reviewer, check-print verification for the following:

- Correct entries
- Complete entries
- Correct mathematical calculations

1.1.2 Team leader, **IF** the originator is not available to resolve discrepancies,
THEN assign a qualified designee.

1.1.3 Reviewer, **IF** discrepancies are found,
THEN resolve discrepancies with Originator or designee.

NOTE

All deletions shall be indicated by a single line strikeout, initials, and date.

Calculations must not be altered after final resolution of check-print corrections.

1.1.4 Originator, make corrections on original work found as a result of the check-print verification.

1.1.5 Reviewer, sign or initial, and date all check-printed pages.

1.2 Computer Input Verification

NOTE

The reviewer must not be the person who recorded the original entries.

1.2.1 Reviewer, check-print all items of input against the source document.

1.2.2 **IF** input data is incorrect,
THEN perform the following:

NOTE

A one-to-one agreement between printout and source document must exist.

[A] Reviewer, make corrections and return check-print to originator.

[B] Originator, **IF** in agreement with corrected check-print, **THEN** generate a corrected computer hard-copy for review

1.2.3 Reviewer, check-print all items of input against the source document.

NOTE

Check-print will have a specific section or the stamping "CHECK-PRINT" for the reviewer to sign or initial, and date.

1.2.4 **IF** input data is correct,
THEN sign or initial, and date the check-print.

2.0 DATA SHEET OR SAMPLE TRACKING LOG BOOK (STLB) PREPARATION

2.1 Obtain current specific data sheets for up-coming sampling events.

2.2 **GO TO** section 3.4 and develop unique sample identity code. **RETURN TO** step 2.3.

2.3 Record unique sample identity code for each sample to be collected.

2.4 Check equipment used to perform sampling is calibrated.

2.5 Record all available data and information before sampling proceeds.

NOTE

See Attachment 1, Example Sample Tracking Log Book.

2.6 Record the following information in the STLB as it becomes available:

- Chain of Custody Number
- Request for Analysis Number
- Sample ID or Quarterly Composite Sample ID Code
- Date (samples) Sent to Lab
- Lab Name
- AOR Information or Comments
- Well Name and Round Number

3.0 SAMPLE TRACKING

3.1 Sample Holding

3.1.1 **IF** samples are awaiting shipment,
THEN hold samples in a secured area.

3.1.2 Retain samples as needed for reserves until analysis from original samples have returned from laboratory.

3.1.3 Perform maintenance, or replace any markings or identification labels damaged during handling or due to aging.

3.1.4 Provide protection of identification markings subject to excessive deterioration from environmental exposure.

3.2 Samples Sent to Off-Site Analytical Laboratory

3.2.1 Verify STLB entries are current.

3.2.2 Request an AOR from the analytical laboratory in the transmittal letter.

3.2.3 Reference any previous C of C numbers for specific samples on the current C of C.

3.2.4 Retain at least one copy of the C of C, RFA, and transmittal letter in the appropriate section of the EM files.

3.2.5 Send samples, completed C of C, completed RFA, and transmittal letter to off-site lab for analysis.

3.2.6 **IF** an AOR is not received from the analytical lab in two weeks, **THEN** contact the lab.

3.2.7 **IF** the shipment cannot be found,
THEN note the information on the file copy of the C of C and log the status in the STLB.

3.2.8 Continue tracking the C of C until final sample disposition.

3.3 Samples Analyzed On-Site

3.3.1 Verify STLB entries are current.

3.3.2 Obtain signature on the original C of C and RFA by laboratory personnel who take custody of the sample.

3.3.3 Retain at least one copy of the C of C and RFA in EM files.

3.3.4 Keep C of C and RFA with samples.

3.3.5 Continue tracking C of C until final sample disposition.

3.4 Assigning Unique Sample Identity Codes

3.4.1 Assign unique identity codes to sample containers and data sheets using the following criteria in descending order:

NOTE

The subprogram code consists of two alphabetic characters which identify the sample type as follows:

- The first letter is an identification of the general subprogram (ex., A for Air, W for Water, B for Biotic)
- The second letter specifies type of sample within the general subprogram (ex., AL for Air Low volume (LO-VOL) sampling, WS for Water Surface sampling, BF for Biotic Fish tissue sampling).

[A] Subprogram (See Table 1 - Subprogram Codes)

Table 1 - Subprogram Codes

AH	Air Hi-vol continuous	EO	Effluent Oversight
AL	Air Lo-vol continuous	SB	Sediment Bottom
BB	Biotic Beef	SD	Soil Deep
BF	Biotic Fish	SI	Soil Intermediate
BD	Biotic Deer	SS	Soil Surface
BO	Biotic Other	WD	Water Deep
BQ	Biotic Quail	WI	Water Intermediate
BR	Biotic Rabbit	WR	Water Runoff
BV	Biotic Vegetation	WS	Water Surface
EC	Effluent Compliance		

NOTE

The location code consists of three alphanumeric characters which identify the site from where the sample was collected.

[B] Location (See Table 2 - Location Codes)

Table 2 - Location Codes

BHT	Bottom of the Hill Tank	RCP	Rainwater Catchment Pond
BRA	BRAntley lake	RED	RED tank
CBD	CarlsBaD	RNS	RiNSe aid blank
COW	COyote Well (deionized water blank)	SE1	SouthEast 1
COY	COYote (surface water duplicate)	SE2	SouthEast 2
CT1	ConTrol 1	SEC	SouthEast Control
CT2	ConTrol 2	SMR	SMith Ranch
FWT	Fresh Water Tank	SOO	Sample Of Opportunity
HIL	HILl tank	SWL	SeWage Lagoons
IDN	InDiaN tank	TUT	TUT tank
LST	LoSt Tank	UPR	Upper Pecos River
MLR	MiLls Ranch	WAB	WIPP Air Blank
NOY	NOYa tank	WE1	WIPP East 1
NW1	NorthWest 1	WEE	WIPP East
NW2	NorthWest 2	WIP	WIPP 16 sections
PCN	Pierce CanyoN	WFF	WIPP Far Field
PEC	PECos river	WSS	WIPP South
PKT	PoKer Trap		

NOTE

The date consists of eight numbers specifying the year, month, and day as follows:

- The year, expressed as four digits (ex., 1996)
- The month and day, expressed as two digits, with "0" proceeding any single-digit number
- No spaces are used between the numbers (ex., September 1, 1996 would be 19960901)

[C] Date

NOTE

The specification code is used to identify a particular sample from a possible group of samples of a particular type collected at a single location and scheduled for the same date.

The specification code is a numeric code consisting of two numbers separated by a decimal point as follows:

- The first number isolates the sample from all other samples in the group.
- The second number indicates the number of samples in the group.
- Examples: 2.10 - indicates sample number 2 of a group of 10, taken from the same location and time (i.e., 10 replicate samples)

  1.1 - indicates one sample was taken at that time and location

[D] Specification

NOTE

An example of a complete sample code is as follows:

AL-SMR-19960901-1.1

AL	Air Low volume
SMR	SMith Ranch
19960901	September 1, 1996
1.1	One sample collected

3.5 Assigning Unique Sample Identity Codes for Water Quality Sampling Program (WQSP) Final Samples

3.5.1 Assign unique identity codes to WQSP groundwater final samples using the following criteria in descending order:

NOTE

The well name consists of a designator specifying a well pad and, if applicable, a bore hole on that well pad (ex., H11b3 where H11 is the name of the well pad; b3 specifies the b3 bore hole).

[A] Well Name

NOTE

The water bearing formation is designated by a one or two letter abbreviation for the formation name. (Ex., C = Culebra Dolomite; M for the Magenta Dolomite; DL for the Dewey Lake Redbeds)

[B] Name of Formation (yielding sample water).

NOTE

The round number is designated with the letter R, followed by a number. A sampling round is one sampling event in which the well is purged and a final sample is taken for analysis.

[C] Round Number

NOTE

The sample number identifies samples which are designated for specific analysis. The sample number will be designated by the letter "N" followed by a number. There may be more than one sample bottle for each sample number requiring multiple labels with the same unique sample identity code.

[D] Sample Number

NOTE

An example of a sample identity code is as follows:

H11b3CR7N5

H11	H11 well pad
b3	b3 borehole
C	Culebra dolomite formation
R7	Sampling round 7
N5	Sample number 5

3.6 Assigning Unique Sample Identity Codes to Composite Samples

3.6.1 Assign a unique sample identity code to sample containers and data sheets using the following criteria in descending order:

[A] Subprogram

[B] Location

[C] Calender Quarter (followed by a dot)

[D] Specification (duplicate)

[E] Year

NOTE

An example of a complete Composite Sample Identity Code is as follows:

AL-SEC-3.2-1996

AL	Air Low volume
SEC	SouthEast Control
3	sample obtained in the third quarter
2	sample is a duplicate sample from this location
1996	sample obtained in 1996

4.0 RESPONSE TO SAMPLE DATA RESULTS ABOVE REGULATORY LIMITS

4.1 **IF** Analytical Lab indicates an analysis result is above regulatory limits, **THEN** perform the following:

4.1.1 **IF** result exceeds a radiological limit, **THEN** support Radiological Control (RC) personnel as directed by the RC Manager.

4.1.2 **IF** result exceeds a non-radiological hazardous material limit, **THEN** support Environmental Compliance and Support (EC&S) personnel as directed by EC&S Manager.

4.1.3 Notify Hazardous Waste Operations.

Attachment 1 - Example Sample Tracking Log Book

Well Name _____ Round Number _____

Sample ID	Chain of Custody Number	Request for Analysis Number	Date Sent to Lab	Lab Name	Data Sheet Number	Date Returned to EM	Abort	Quarterly Composite Sample ID Code	Chain of Custody Number	Request for Analysis Number	Date Sent to Lab	Lab Name	AOR Information or Comments

CONTROLLED COPY

Attachment 2 - Example Chain-of-Custody Record

CHAIN-OF-CUSTODY RECORD

WASTE ISOLATION PILOT PLANT
WESTINGHOUSE ELECTRIC CORP.
P.O. BOX 2078
CARLSBAD, NM 88221-2078

C of C Control No. _____

RFA Control No. _____

SAMPLING PROGRAM _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No.

Special Instructions: _____

Possible Sample Hazards: _____

SIGNATURES: (Name, Company, Date and Time)

1. Relinquished By: _____ 3. Relinquished By: _____

Received By: _____ Received By: _____

2. Relinquished By: _____ 4. Relinquished By: _____

Received By: _____ Received By: _____

WP 02-EM3001 WHITE - Original, to accompany samples YELLOW - Field Copy PINK - Other

CONTROLLED COPY

Attachment 3 - Example Request for Analysis

REQUEST FOR ANALYSIS

RFA Control No. _____
C of C Control No. _____

WASTE ISOLATION PILOT PLANT
WESTINGHOUSE ELECTRIC CORP.
P.O. BOX 2078
CARLSBAD, NM 88221-2078

DATE SAMPLES SHIPPED _____
LAB DESTINATION _____
LABORATORY CONTACT _____
SEND LAB REPORT TO _____

SAMPLING PROGRAM _____
PURCHASE ORDER NO. _____

DATE REPORT REQUIRED _____
PROJECT CONTACT _____
PROJECT CONTACT PHONE NO. _____

Sample Number	Sample Type	Sample Quantity	Preservative	Req'l. Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by appropriate Manager) NORMAL _____ RUSH _____ (Subject to rush surcharge)
POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances.)
NONHAZARD _____ FLAMMABLE _____ SKIN IRRITANT _____ HIGHLY TOXIC _____ BIOLOGICAL _____ OTHER _____
SAMPLE DISPOSAL: (Please indicate disposition of sample following analysis.) RETURN TO CLIENT _____ DISPOSAL BY LAB _____

FOR LAB USE ONLY
RECEIVED BY _____ DATE/TIME _____

WP 02-EM3001 WHITE - Original, to accompany samples YELLOW - Field Copy PINK - Other

CONTROLLED COPY

Attachment 1 - Procedure Change Notice Form

PROCEDURE CHANGE NOTICE

SECTION 1 - PROCEDURE & WRITER INFORMATION

1) PROCEDURE NUMBER	2) REVISION	3) COGNIZANT ORGANIZATION	4) CHANGE NUMBER
WP02-119	2	ES+H	2
5) WRITER'S PRINTED NAME	**6) ORGANIZATION**	**7) SIGNATURE**	**8) EXTENSION**
RON RICHARDSON	E M	Ron Richardson	8395

9) PROCEDURE TITLE

MANUAL WATER LEVEL MEASUREMENTS

10) JUSTIFICATION/REASON FOR CHANGE

ANNUAL REVIEW

15) RECORD THE LOCATIONS OF THE MANUALS WHERE THE CHANGES ARE IMMEDIATELY PLACED.	12) DURATION	13) CHANGED PAGES
1-19-96 2-13-96 CDL'S #2	PERMANENT ☑ OR TEMPORARY ☐	2, 8
		14) IF TEMPORARY, DATE OF EXPIRATION

04

11) CHANGE TYPE (✓ or X ONE)

MINOR CHANGE ☑ Meets the first criteria or all of the second criterion:

YES	NO	
☑	☐	1. Inconsequential editorial change only.
		OR
☑	☐	1. Changes that do not affect any other WID department
☑	☐	2. Changes that do not affect any external interface organization
☑	☐	3. Changes that do not alter the document's stated objectives, purpose, scope, intent, or methodology.

MAJOR CHANGE ☐ Does not meet the requirements of a Minor Change

RECORD ALL REVIEWS ON PAGE TWO OF THIS FORM

SECTION 2 - CHANGE APPROVAL SIGNATURE

APPROVED BY	PRINTED NAME	SIGNATURE	DATE
☑ COM ☐ FOSS ☐ PER TELCON	DAVE KUMP	D.R. Kump	2/13/96

SECTION 3 - CHANGE PROCESSING

PERFORMED BY	PRINTED NAME	SIGNATURE	DATE
RECEIVED BY: COG PROCEDURE GROUP			
CONCURRENCE REVIEW BY: DRC CHAIRPERSON			
RECEIVED BY: DOCUMENT DISTRIBUTION			

01 Attachment 1 - Procedure Change Notice Form

Procedure Number : WP02-119	PCN Number : 2

REVIEWS

TECHNICAL REVIEWS (other than PCN Writer)

ORGANIZATION	REVIEWER PRINTED NAME	SIGNATURE	DATE
ESH/EM	MEL BALDELLAMA	Mel Baldellama	02/09/96
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			

VALIDATION REVIEWS (other than PCN Writer)

ORGANIZATION	REVIEWER PRINTED NAME	SIGNATURE	DATE

01 Attachment 1 - Procedure Change Notice Form

PROCEDURE CHANGE NOTICE

SECTION 1 - PROCEDURE & WRITER INFORMATION

1] PROCEDURE NUMBER	2] REVISION	3] COGNIZANT ORGANIZATION	4] CHANGE NUMBER
WP02-119	~~03~~ 02 (TB 11/30/94)	Environmental Monitoring	~~02~~ 01
5] PCN WRITER'S PRINTED NAME	**6] ORGANIZATION**	**7] SIGNATURE**	**8] EXTENSION**
Mel Balderrama	ENV. MON	Mel Balderrama	8174

9] PROCEDURE TITLE

Manual Groundwater Level Measurements

10] JUSTIFICATION/REASON FOR CHANGE

Annual Review

11] CHANGE TYPE (✓ or X ONE)	12] DURATION (✓ or X ONE)	13] AFFECTED CHANGED PAGES
MAJOR CHANGE [ ] OR MINOR CHANGE [X]	PERMANENT [X] OR TEMPORARY [ ]	1,2,3,5,6,7,7A Attachment 2.
		14] IF TEMPORARY, DATE OF EXPIRATION

15] RECORD THE LOCATIONS OF THE MANUALS WHERE THE CHANGES ARE IMMEDIATELY PLACED.

ALL COLs

RECORD ALL REVIEWS ON PAGE TWO OF THIS FORM

SECTION 2 - CHANGE APPROVAL SIGNATURE

02 APPROVED BY: [X] COM [ ] FOSS [ ] PER TELCON

PRINTED NAME	SIGNATURE	ORGANIZATION	DATE
JJ Cotton	JJ Cotton	ENVIRONMENTAL MONITORING	11/30/94

SECTION 3 - CHANGE PROCESSING

PERFORMED BY	PRINTED NAME	SIGNATURE	DATE
RECEIVED BY: D&PS	Wendie Berry	Wendie Berry	12/2/94
CONCURRENCE REVIEW BY: DRC CHAIRPERSON	JJ Cotton	JJ Cotton	11/30/94
RECEIVED BY: DOCUMENT DISTRIBUTION	Shannon Quart	Shannon Quart	12/2/94

CONTROLLED COPY

01 Attachment 1 - Procedure Change Notice Form

Procedure Number :	PCN Number : 01

REVIEWS

TECHNICAL REVIEWS (other than PCN Writer)

ORGANIZATION	REVIEWER PRINTED NAME	SIGNATURE	DATE
ESHQ RC	RON RICHARDSON	Ron Richardson	11/30/94
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			
☐ Per Telcon			

VALIDATION REVIEWS (other than PCN Writer)

ORGANIZATION	REVIEWER PRINTED NAME	SIGNATURE	DATE

CONTROLLED COPY

NO.: 02-119 REV.: 2 EFFECTIVE DATE: 12/23/91

APPROVAL SIGNATURE: Leslie L. Rose

PROCEDURE AUTHOR: R. Richardson

TITLE: MANUAL GROUNDWATER LEVEL MEASUREMENTS

1.0 SCOPE

This document describes the procedure used by Environmental Monitoring personnel fo making manual groundwater level measurements. ~~Calibration of equipment used for takin manual groundwater level measurements and records pertaining to calibrations will b performed and maintained by the calibration department. This activity is conducted a part of the Waste Isolation Pilot Plant (WIPP) Groundwater Monitoring Program~~ Organizational responsibilities, personnel training and qualification, reporting records and document control, equipment calibration, and quality control requirement for the program are outlined in WP 02-1, WIPP Groundwater Monitoring Program Plan an Procedures Manual. Unless specific activity requirements are outlined in thi procedure, the user should refer to WP 02-1.

The following activities are detailed in Section 5.0 of this procedure:

~~5.1 Water Level Measurement Using Steel Tape~~
5.2 Water Level Measurement Using Electrical Conductance Probe
5.3 Data Management

2.0 DEFINITIONS

Analytical Task Leader - Person responsible for performing physical and chemica analyses of groundwater in accordance with applicable plans, procedures, and di rections. Qualifications include an educational background in chemistry and workir experience with wet lab procedures for water analysis.

Electrical Conductance Meter - Battery-powered water level measuring probe.

Length Standard - Length-measuring ruler traceable to the National Bureau of Standarc (NBS).

Principal Investigator - Person responsible for ensuring that the WIPP Groundwate Monitoring Program is being conducted in accordance with applicable plans ar procedures.

Solinst Probe - An electrical conductance probe used to measure water level in a well

~~Steel Tape - A measuring tape that is approximately 1/4 inch wide, and is graduate every 0.01 feet for the first 30 feet; and at every one foot increment for th remainder of the tape length.~~

3.0 REFERENCES

ASME NQA-1-1989, Quality Assurance Program Requirements for Nuclear Facilities
WP 02-1, WIPP Groundwater Monitoring Program Plan and Procedures Manual
~~WP 02-302, NES/RES Quality Assurance/Quality Control Implementation Procedures~~
~~WP 10-003, Instrument Control and Calibration~~ ~~10WC 3006~~ ~~10WC 3007~~
WP 13-1, Waste Isolation Division Quality Program Manual
~~WP 13-6, Quality Assurance Records Management Policy~~ WP 15-PR Record Management PL
~~WP 15-101, Preparing, Revising, Reviewing, and Cancelling Procedures~~
~~15-PS3001, 15-PS3002, & 15-PS3003~~
WP02-EM1010 QUALITY ASSURANCE, QUALITY CONTROL AND SAMPLE TRACKING

4.0 GENERAL

The primary objective of water level measurements is to monitor and document long-term water level fluctuations in the vicinity of the WIPP. The measurements will monitor changes in water level elevations due to seasonal fluctuations or hydrologic test activities.

Taking manual groundwater level measurements involves lowering a water level measuring device into a well, and recording the depth to water on WP Form 2288, Water Level Measurement Field Data Sheet (Attachment 1).

4.1 Prerequisites

Ensure the water level measuring device (~~steel tape or~~ solinst probe) calibration is current, per records maintained by the ~~calibration laboratory~~ EM SECTION.

Only qualified and authorized Environmental Monitoring personnel, per WP 02-1, may perform this procedure.

4.2 Safety Precautions

Radio and/or telephone communications are available at all times.

4.3 Equipment Calibration SECTION DELETED

Add text Below

~~The equipment used in taking water level measurements shall be calibrated by the WIPP Site Calibration Laboratory. The Calibration Laboratory is responsible for calibrating the following equipment on schedule, in accordance with WP 10-003, and for maintaining the calibration records.~~ 10WC 3006 10WC 3007

01 ~~Steel Tape - Calibrated against a standard length measuring device.~~
Solinst Probe - Calibrated with a standard length measuring device.

Equipment calibrations will be conducted as determined by the Calibration Laboratory, or when the operator suspects that the equipment may be out of calibration or is not functioning properly.

All equipment that is used in gathering data for input into reports and other documents is to be calibrated according to equipment calibration and maintenance records in accordance with ~~WP 10-003~~ 10WC 3006 10WC 3007

4.4 Performance Frequency

A water level measurement shall be taken prior to installing equipment into the well for any geohydrologic activities. Water level measurements will be taken either monthly or quarterly as directed by the Principal Investigator.

Water level measurements at each individual well will be repeated until the operators' confidence in the readings has been satisfied. ~~This may require additional measurements to be taken, using the two different types of measuring devices~~.

4.5 Quality Assurance

For the hydrologic activities performed in taking water level measurements, applicable portions of WP 13-1 shall be used to provide a means for control and review of the work. Applicable portions of the Quality Assurance (QA) program involve:

- Project procedures
- Field data verification and documentation
- Equipment calibration
- Nonconformance documentation
- Records administration
- Quality assurance auditing

Any additional QA requirements specified by Westinghouse Electric Corporation shall also be implemented.

4.6 Records

The following records generated in support of this procedure shall be retained as permanent quality assurance records:

WP Form 2288 (Attachment 1), Water Level Measurement Field Data Sheet

The following records generated in support of this procedure shall be retained as nonpermanent quality assurance records: None

Environmental Monitoring is responsible for validation of these records prior to transmittal to the ~~Master Records Center (MRC)~~. PROJECT RECORDS SERVICES (PRS)

Temporary storage of these records, when provided, shall be accomplished by storage in a fire-proof cabinet that complies with the requirements of ASME NQA-1-1989, Supplement 17S-1, paragraph 4.4.3 and ~~WP 13-6~~. WP-PR Record Management Plan

Corrections to these records, when needed, shall be made by drawing a single line through the erroneous text and inserting the correction. The corrected text shall not be obliterated. The person correcting the text shall initial and date the document adjacent to the corrected text using black ink.

These records shall be identified on the Environmental Monitoring Records Inventory and Disposition Schedule (RIDS). Permanent storage is the responsibility of the ~~MRC~~. PROJECT RECORDS SERVICES (PRS)

4.7 Change History

Page 4 and Att. 1 - 1/29/92; Page 4 and Attachment 1 - 11/06/92

5.0 PROCEDURE

Page Delete

5.1 Water Level Measurement Using Steel Tape

1. Check the calibration status of the tape, then coat the lower portion of the tape, which will come in contact with the water, with carpenter's chalk.

2. Lower the tape into the well.

NOTE: The depth to which the tape is lowered is estimated from previous water level measurements taken in the well.

3. Hold the steel tape at an even foot mark at the measuring reference point, usually the top of the casing. Subtract the length of the wetted chalk (correction length) from the measured length (footage held at the reference point) and record that figure.

4. Determine if the held depth is correct, too short, or too long. If measurement depth is correct, proceed to Step 9.

DEPTH CORRECT → GO TO STEP 8

DEPTH INCORRECT →

5. If the held depth is too short, the chalked portion will be dry when the tape is brought back to the surface and the measurement must be retaken at a new adjusted depth.

Page Delete

6. If the held length is too long, then the whole chalked portion will be wet and the measurement must be retaken at a new adjusted depth.

7. Retake measurements at adjusted depths, according to Steps 1 through 4.

8. If necessary, adjustments to the readings can be made for changes in the tape's length due to thermal change using parameters in Attachment 2.

NOTE: Because thermal change corrections are small (usually less than 0.10 feet for a 30°F temperature change) and the measurements are not cumulative, these adjustments are not incorporated in the final reading.

9. After completing the water level measurement at each well, clean and rinse the equipment with fresh water in preparation for the next well.

5.2 Water Level Measurement Using Electrical Conductance Probe

01

Insert steps OPerational checks

SEE PAGE 7A

1. Turn the probe power on and lower the probe into the well.

~~Insert~~ ~~Note 1~~

01

INSERT NOTE 1 (SEE TEXT BELOW)

~~NOTE: The battery can be checked on the surface by pressing a test button which short circuits the system and activates the alarm.~~

2. When the probe reaches the water level, an electrical circuit is completed, causing a buzzer to go off. After the buzzer sounds, measure the measuring reference point by reading the footage directly on the embossed electrical cable.

01

~~NOTE: The conductor cable has been marked at 0.05 foot increments.~~

3. After completing the water level measurement at each well, clean and rinse the equipment with fresh water in preparation for the next well.

01

NOTE 1

(NOTE:) The conductor cable TOP OF CASING is marked in increments of 0.01. Reference point on casing is at lock eye, or section of well head closest to pad lock. If well head is beveled or measurement cannot be taken accurately, then use straight edge. STRAIGHT

Lay ~~straight~~ edge across well head and use lowest edge of ~~stright~~ STRAIGHT edge to take measurement.

Operational checks of Solinst Water Level Meter.

1. Perform the following operational checks:

   Solinst Water Level Meter:

   A. Test button check: depress button to test the battery and circuitry (excluding the probe).

   B. Set toggle switch to "on" position, or turn rotary dial fully clockwise.

   C. Circuity check: submerse the electrode(probe)in tap water. This completes the circuit and activates the buzzer.

2. Sensitivity Adjustment:

   A. If on/off switch is a rotary dial, clockwise rotation of rotary dial turns meter on and increases sensitivty.

   B. Always set switch to Highest Sensitivity position, then decrease if necessary.

   C. If the alarm continues after removal from water the sensitivity should be reduced.

   D. If no alarm is heard (although the battery is in working order) then the sensitivity should be increased.

3. Battery Replacment:

   A. Battery type-alkaline, 9 volt.

   B. The battery is in the reel hub and is replaced by removing the front plate of the reel.

   C. Remove battery and install new one, making sure the polarity is correct and the wires are fully inside.

5.3 Data Management

1. Personnel verify the correct recording of data on WP Form 2288.

   NOTE: Time should be reported in a military format. All length measurements should be reported to the nearest hundredth.

2. Personnel set up a data file for WP Forms 2288, and submit the data file to the Principal Investigator. The data file should contain all field records, stamped "ORIGINAL."

3. Personnel input the data collected into computer data files where additional data reduction in both tabular and graphical methods are performed.

02

DELETE NOTE

~~NOTE: Data will be compiled and published annually in the Annual Site Environmental Report.~~

~~00224~~

WATER LEVEL MEASUREMENT FIELD DATA SHEET

Well Number	Zone	Date	Time	Measurement In Feet	Adjustment	Initial	Instrument I.D. No.	Calibration Due Date	Comments
AEC 07	CUL								
AEC 08	B/C								
CB 1	CUL								
D-268	CUL								
DOE-1	CUL								
DOE 2	CUL								
ERDA-9	CUL								
H-01	CUL								
H 01	MAG								
H 02a	CUL								
H 02b1	MAG								
H 02b2	CUL								
H 02c	CUL								
H 03b1	MAG								
H 03b2	CUL								
H 03b3	CUL								
H 03d	49er								
H 03d	DL								
H 04b	CUL								
H 04c	MAG								
H 05a	CUL								
H 05b	CUL								
H 05c	MAG								
H 06a	CUL								
H 06b	CUL								
H 06c	MAG								
H 07b1	CUL								
H 07b2	CUL								
H 08a	MAG								
H 08c	Rus/Sal								
H 09a	CUL								
H 09b	CUL								
H 09c	CUL								
H 10a	MAG								
H-10b	CUL								
H-11b1	CUL								
H-11b2	CUL								

CONTROLLED COPY

WATER LEVEL MEASUREMENT FIELD DATA SHEET

Well Number	Zone	Date	Time	Measurement In Feet	Adjustment	Initial	Instrument I.D. No.	Calibration Due Date	Comments
H-11b3	CUL								
H-12	CUL								
H-14	CUL								
H-15	CUL								
H-16	DL								
H-16	DHLM								
H-17	CUL								
H-18	CUL								
P-14	CUL								
P-15	CUL								
P-17	CUL								
P-18	CUL								
WIPP-12	CUL								
WIPP-15	CUL								
WIPP-18	CUL								
WIPP-19	CUL								
WIPP-21	CUL								
WIPP-22	CUL								
WIPP-25	CUL								
WIPP-25	MAG								
WIPP-26	CUL								
WIPP-27	CUL								
WIPP-27	MAG								
WIPP-28	Rus/Sal								
WIPP-29	CUL								
WIPP-30	CUL								
WIPP-30	MAG								

10828.0443a

CONTROLLED COPY

Delete Page

Steel tape elongation due to thermal expansion

$$C_t = k(T_1 - T) \times L$$

Where:

C_t = correction in length (feet)

k = coefficient of thermal expansion ($k = 6.5 \times 10^{-06}$ feet per degree Fahrenheit, for mild steel)

T_1 = temperature at the time of measurement (°F)

T = temperature at standard length (°F)

L = length of tape used for measurement

WP 02-EM1006

Revision 1

Final Sample and Serial Sample Collection

Technical Procedure

EFFECTIVE DATE: 8/29/96

SIGNATURE	PRINTED NAME	DATE
D. R. Kump	D. R. Kump	8/29/96

APPROVED FOR USE

REVIEW ORGANIZATIONS

Environmental Monitoring	Industrial Safety	Quality & Regulatory Assurance
Environmental Compliance & Support	Hazardous Waste Operations	RCRA Permitting
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A

CONTROLLED COPY

TABLE OF CONTENTS

INTRODUCTION 3

REFERENCES 3

EQUIPMENT 3

PRECAUTIONS AND LIMITATIONS 5

PREREQUISITE ACTIONS 5

PERFORMANCE 5
1.0 COLLECTION OF WATER SAMPLES FROM PRIVATE WELLS 5
2.0 COLLECTION OF SERIAL GROUNDWATER SAMPLES 10
3.0 COLLECTION OF FINAL SAMPLES 11
4.0 ANALYTICAL TASK LEADER REVIEW 14

Attachment 1 - Definitions 15

Attachment 2 - Final Sample Checklist 16

INTRODUCTION

This procedure describes the steps that Environmental Monitoring (EM) personnel perform to collect water samples from private wells, serial groundwater samples, and final samples in support of the Waste Isolation Pilot Plant (WIPP) Groundwater Surveillance Program.

RECORD GENERATED BY THIS PROCEDURE

- Final Sample Checklist (See Attachment 2 of this procedure).

REFERENCES

COMPLIANCE

- DOE/EH-0173T, Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance
- DOE/EP-0023, A Guide for Environmental Radiological Surveillance at U.S. Department of Energy Installations

BASELINE

- WP 13-1, WID Quality Assurance Program Description (Section 4).
- DOE/WIPP 96-2162 Groundwater Protection Management Program Plan.
- DOE/WIPP 94-024, Waste Isolation Pilot Plant Environmental Monitoring Plan

REFERENCED

- WP 15-PR, Records Management Plan

EQUIPMENT

CONSUMABLE MATERIALS

- 0.45-µm filters
- Deionized water
- Packing material
- Paper towels

TEST EQUIPMENT AND SUPPLIES

- 1 liter amber glass bottles
- 1 gallon plastic cubitainers
- 1 liter polyethylene bottles
- 250 ml amber glass bottles
- 40-ml glass vials with teflon-lined septums
- 47-mm filter holder
- 250-mL plastic beakers, with lids
- ½ gallon amber glass bottle
- Clear packing tape
- Request for analysis and chain-of-custody forms
- Electrical tape
- Indelible pens
- Labels or waterproof marker
- Portable water pump
- Ring stand and clamp
- Tempering beaker
- Timepiece (wristwatch, clock, etc.)
- Tweezers
- Waterproof coolers

REAGENTS

- Hydrochloric acid [HCl Ultrex II (36% by Wt.) or equivalent]
- Nitric acid [HNO_3 Ultrex II (70.6% by Wt.) or equivalent]
- Sulfuric acid [H_2SO_4 Ultrex II (89.4% by Wt.) or equivalent]

- Zinc acetate approximately (10%)
- Sodium hydroxide approximately (50%) (NaOH)

PRECAUTIONS AND LIMITATIONS

- The wearing of chemical goggles and rubber gloves is required when handling acids
- Verify EM personnel are qualified in accordance with applicable EM qualification cards **OR** are under direct supervision of qualified personnel
- The MSDS book must be up to date and readily available

PREREQUISITE ACTIONS

- Notify the well owner 10 days prior to the actual sampling activity.
- Verify EM personnel are qualified in accordance with applicable EM qualification cards **OR** are under direct supervision of qualified personnel before performing this procedure.

PERFORMANCE

1.0 COLLECTION OF WATER SAMPLES FROM PRIVATE WELLS

1.1 Filtered Sample Collection

1.1.1 Prepare the applicable Final Sample Checklist (Attachment 2) by recording the appropriate data for items 1 through 9.

1.1.2 Connect sample line from portable pump to well-pumping apparatus.

1.1.3 Rinse filter holder of portable pump with deionized water.

1.1.4 Use tweezers to place a clean 0.45-µm filter on bottom portion of filter holder.

1.1.5 Wet filter thoroughly with deionized water to prevent air locks.

1.1.6 Assemble filter holder

1.1.7 Connect filter holder to output line from pump.

1.1.8 Direct water through filtered sample line.

1.1.9 Rinse sample bottle(s) three times.

1.1.10 Collect one liter of filtered water for field laboratory analysis.

NOTE

Preserved samples should have bottle filled approximately ½ full.

1.1.11 Collect filtered water sample(s) to be sent off site for analysis and for WIPP archive and WIPP laboratories.

NOTE

Time should be recorded in a military format.

1.1.12 Record time each individual sample collection began and ended on the Final Sample Checklist (Attachment 2, Item 18).

1.1.13 **IF** sample requires preservative,
GO TO Section 1.3, perform water sample preservation steps,
RETURN TO Step 1.1.15.

NOTE

The Final Sample Checklist indicates which sample receives preservative and the type to be used.

1.1.14 **IF** sample does not require preservative,
THEN secure lid to bottle and seal with electrical tape.

1.1.15 Label filtered sample(s) with the following:

- Project name
- Sample number
- Sample location (zone sampled)
- Well number
- Collector's name
- Date
- Type of sample (deionized water or groundwater)
- Acid wash (yes or no)
- Parameter or destination
- Type of preservative

- Bottle number
- Method of collection (filter or unfiltered)

1.1.16 Affix label securely to bottle and cover with clear packing tape.

1.1.17 Disassemble filter holder.

1.1.18 Remove used 0.45-µm Filter

1.1.19 Rinse filter holder with deionized water.

1.2 Unfiltered Sample Collection

1.2.1 Direct water flow through an unfiltered sample line.

1.2.2 Rinse sample bottle(s) three times.

NOTE

Preserved samples should have bottle filled approximately ½ full.

1.2.3 Collect one liter of unfiltered water for field laboratory analysis.

1.2.4 Collect unfiltered water sample(s) to be sent off site for analysis.

NOTE

Record time in a military format.

1.2.5 Record time each individual sample collection began and ended on the Final Sample Checklist (Attachment 2, Item 18).

NOTE

The Final Sample Checklist indicates which sample receives preservative and the type to be used.

1.2.6 **IF** sample requires preservative,
GO TO Section 1.3, perform water sample preservation steps,
RETURN TO Step 1.2.8.

1.2.7 **IF** sample does not require preservative,
THEN secure lid to bottle and seal with electrical tape.

1.2.8 Label unfiltered sample(s) with the following data:

- Project name
- Sample number

- Sample location (zone sampled)
- Well number
- Collector's name
- Date
- Type of sample (deionized water or groundwater)
- Acid wash (yes or no)
- Parameter or destination
- Type of preservative
- Bottle number
- Method of collection (filter or unfiltered)

1.2.9 Affix label securely to sample bottle(s) **AND** cover with clear packing tape.

1.3 Water Sample Preservation

WARNING

To prevent injury to personnel, use of chemical goggles and gloves is required when using acids or strong bases.

NOTE

Some samples and blanks will require preservation immediately following collection.

1.3.1 Reference the Final Sample Checklist (Attachment 2, Item 17) to determine type of preservative to add to water sample(s).

1.3.2 For samples requiring acid preservation, add 2 ml per liter of selected high purity preservative (sulfuric acid [H_2SO_4], nitric acid [HNO_3]), or hydrochloric acid [HCl].

1.3.3 Fill bottle to shoulder.

1.3.4 Agitate preserved water sample.

1.3.5 Check pH factor.

1.3.6 **IF** pH is above 2.0 standard units,
THEN add one ml of selected preservative to lower pH factor.

1.3.7 Agitate preserved water sample again.

1.3.8 Repeat Steps 1.3.5 through Step 1.3.7 until pH factor drops below 2.0 standard units.

1.3.9 Add the same relative amount of preservative to each remaining sample requiring acid preservative as indicated by the Final Sample Checklist (Attachment 2, Item 17).

1.3.10 For samples requiring sodium hydroxide (NaOH) preservation, add 2 ml. of NaOH per liter of sample.

1.3.11 Agitate preserved sample.

1.3.12 Check pH factor.

1.3.13 **IF** pH is below 12 standard units,
THEN add one ml of NaOH to raise pH.

1.3.14 Agitate preserved sample again.

1.3.15 Repeat Steps 1.3.12 through Step 1.3.14 until pH is above 12 standard units.

1.3.16 Add the same relative amount of NaOH to remaining samples requiring this preservative.

1.3.17 Add 5 drops of zinc acetate to samples requiring this preservative.

1.3.18 Secure lid with electrical tape.

2.0 COLLECTION OF SERIAL GROUNDWATER SAMPLES

2.1 Serial Sample Collection of Filtered Groundwater

2.1.1 Prior to sampling, perform the following:

- Inspect filter holder for contaminants and damage.
- Rinse with deionized water.

2.1.2 Use tweezers to place one 0.45-µm filter on bottom portion of filter holder.

2.1.3 To prevent air locks, wet filter thoroughly with deionized water.

2.1.4 Assemble filter holder.

2.1.5 Place assembled filter holder on laboratory rack.

2.1.6 Divert water flow through sample line and filter.

2.1.7 Rinse three sample bottles, three times each, with filtered water.

2.1.8 Fill three bottles with filtered water.

2.1.9 Note time of final sample collection on Attachment 2, item 18.

2.1.10 Place one bottle in temperature bath at 25° C for measurement of specific conductance.

2.1.11 Analyze other two bottles according to Groundwater Serial Sample procedure, for filtered water sample, at a later time, but on same day.

2.1.12 Disassemble filter holder.

2.1.13 Remove used 0.45-µm filter.

2.1.14 Rinse filter holder thoroughly with deionized water.

2.2 Serial Sample Collection of Unfiltered Groundwater

2.2.1 Prior to sampling, inspect sample line and filter holder.

2.2.2 Rinse filter holder with deionized water.

2.2.3 Assemble filter holder without filter and secure to laboratory rack.

2.2.4 Divert water flow through filter holder.

2.2.5 Analyze unfiltered samples according to Groundwater Serial Samples Procedures for unfiltered water sample.

3.0 COLLECTION OF FINAL SAMPLES

3.1 Unfiltered Final Sample Collection

3.1.1 Direct water flow through an unfiltered sample line.

3.1.2 Rinse sample collection bottle three times.

3.1.3 **IF** unfiltered final samples collected are to be analyzed for VOC, Tox, **OR** Sulfide.
THEN unfiltered final samples should be filled in such a manner that no air remains in sample bottle.

NOTE

Preserved samples should have bottle filled approximately ½ full.

3.1.4 Fill bottle with final sample.

NOTE

Report time in a military format.

3.1.5 Record time sample collection began and ended on Final Sample Checklist (Attachment 2, Item 18).

NOTE

The Final Sample Checklist indicates which samples receive preservative and the type to be used.

3.1.6 **IF** sample requires a preservative,
GO TO Section 1.3, perform water sample preservation steps,
RETURN TO Step 3.1.8.

3.1.7 **IF** sample does not require a preservative,
THEN secure lid of sample bottle and seal with electrical tape.

3.1.8 Label sample with the following:

- Project name
- Sample number
- Sample location (zone sampled)
- Well number
- Collector's name
- Date
- Type of sample (deionized water or groundwater)
- Acid wash (yes or no)
- Parameter or destination
- Type of preservative
- Bottle number
- Method of collection (filter or unfiltered)

3.1.9 Affix label securely to sample bottle, and cover with clear packing tape for moisture protection.

3.2 Filtered Final Sample Collection

NOTE

Sample Numbers 21, 22, and 23 on the Final Sample Checklist are prepared and shipped only on selected wells at discretion of Team Leader (TL).

3.2.1 Rinse filter holder with deionized water.

3.2.2 Use tweezers to place one 0.45-µm filter on bottom portion of filter holder.

3.2.3 Wet 0.45-µm filter thoroughly with deionized water to prevent air locks.

3.2.4 Assemble and secure filter holder.

3.2.5 Divert water into filter assembly.

3.2.6 Use the filtered sample water to rinse out the sample bottle three times.

NOTE

Preserved samples are to be filled approximately ½ full.

3.2.7 Fill bottles with final sample.

NOTE

Report time in military format.

3.2.8 Record time final sample collection began and ended on the Final Sample Checklist (Attachment 2, Item 18).

NOTE

The Final Sample Checklist indicates which sample receives preservative and the type to be used.

3.2.9 **IF** sample requires a preservative,
GO TO Section 1.3, perform water sample preservation steps,
RETURN TO Step 3.2.11.

3.2.10 **IF** sample does not require a preservative,
THEN secure lid of sample bottle and seal with electrical tape.

3.2.11 Label final sample with the following:

- Project name
- Sample number
- Sample location (zone sampled)
- Well number
- Collector's name
- Date
- Type of sample (deionized water or groundwater)
- Acid wash (yes or no)
- Parameters or destination
- Type of preservative

- Bottle number
- Method of collection (filter or unfiltered)

3.2.12 Affix label securely to bottle and cover with clear packing tape for moisture protection.

3.2.13 Disassemble filter assembly.

3.2.14 Discard filter.

3.2.15 Rinse filter holder with deionized water.

4.0 ANALYTICAL TASK LEADER REVIEW

4.1 Verify that all responsible checklists are filled out properly.

4.2 Verify that all applicable checklists are archived in accordance with WP 15-PR, Records Management Plan.

Attachment 1 - Definitions

Analytical Task Leader (ATL) - Person responsible for performing physical and chemical analyses of groundwater in accordance with applicable plans, procedures, and directions.

Field Blank - A sample of deionized water that will be analyzed to monitor for possible contamination during shipment.

Final Sample - A sample of groundwater collected after it has been determined that the chemical and physical parameters of the water being sampled have stabilized.

Private Well - A well that provides water that is currently being used for human and/or livestock consumption.

Serial Sample - A serial sample is a sample of groundwater taken at periodic intervals to establish stabilization of chemical and physical parameters prior to final sampling, based on results of past analytical results.

Team Leader (TL) - Person responsible for assuring that the WIPP Groundwater Monitoring Program is being conducted in accordance with applicable plans and procedures.

CONTROLLED COPY

Attachment 2 - Final Sample Checklist

(1) Project Name: WIPP-WQSP, (2) Well Number: ___, (3) Filter Type: WHATMAN, (4) Collected by: _____

(5)Zone: _____, (6) Round: _____, (7) Lot #: _____, (8) Date Collected: _____

(9) SAMPLE NUMBER	(10) PARAMETERS	(11) DESTINATION	(12) No. of BOTTLES	(13) VOLUME	(14) TYPE	(15) ACID WASH	SAMPLE (16) FILTER	(17) PRESERVATIVE	(18) COLLECTION TIME
N 1	General Chemistry	RAS	3	1 Liter	Plastic	Yes	No	None	
N 2	General Chemistry	RAS	1	1 Liter	Plastic	Yes	No	H_2SO_4, pH<2	
N 3	General Chemistry	RAS	1	1 Liter	Plastic	Yes	No	HNO_3, pH<2	
N 4	TOX	RAS	3	250 ml	Glass	Yes	No	H_2SO_4, pH<2	
N 5	Phenolics	RAS	1	1 Liter	Amber Glass	Yes	No	H_2SO_4, pH<2	
N 6	Metals/Cations	RAS	2	1 Liter	Plastic	Yes	No	HNO_3, pH<2	
N 7	VOC	RAS	4	40 ml	Glass	No	No	HCL, pH<2	
N 8	VOC (Purgable)	RAS	2	40 ml	Glass	No	No	HCL, pH<2	
N 9	VOC (Non-Purgable)	RAS	2	40 ml	Glass	No	No	HCL, pH<2	
N 10	BN/A's	RAS	1	½ Gal.	Amber Glass	Yes	No	None	
N 11	Herbicides	RAS	2	1 Liter	Amber Glass	Yes	No	None	
N 12	Organo-Chlorine Pesticides/PCB	RAS	1	½ Gal.	Amber Glass	Yes	No	None	
N 13	Organo-Phosphorus Pesticides	RAS	1	½ Gal.	Amber Glass	Yes	No	None	
N 14	Dioxins/Furans	RAS	2	1 Liter	Amber Glass	Yes	No	None	
N 15	Appendix IX Metals	RAS	1	1 Liter	Plastic	Yes	No	HNO_3, pH<2	
N 16	Cyanide (Total)	RAS	1	1 Liter	Plastic	Yes	No	NaOH, pH>12	
N 17	Sulfide (Total)	RAS	1	250 ml	Amber Glass	Yes	No	NaOH + ZnAcetate	
N 18	Radionuclides	DCL	1	1 Gal.	Plastic Cube	Yes	Yes	HNO_3, pH<2	
N 19	Archival Sample	Archive	1	1 Gal.	Plastic Cube	Yes	Yes	HNO_3, pH<2	

CONTROLLED COPY

Attachment 2 - Final Sample Checklist

FINAL SAMPLE CHECKLIST
ACID/TRIP/REAGENT BLANKS

(1) Project Name: WIPP-WQSP, (2) Well Number: ___, (3) Filter Type: BARNSTEAD, (4) Collected by: _____
(5) Zone: CULEBRA, (6) Round: _____, (7) Lot #: _____, (8) Date Collected: _____

(9) SAMPLE NUMBER	(10) PARAMETERS	(11) DESTINATION	(12) No. of BOTTLES	(13) VOLUME	(14) TYPE	(15) ACID WASH	SAMPLE (16) FILTER	(17) PRESERVATIVE	(18) COLLECTION TIME
N20	VOC	RAS	4	40 mL	Glass	NO	YES	HCl, pH < 2	
N21	Metals/Acid	RAS	2	1 Liter	Plastic	YES	YES	HNO_3, pH < 2	
N22	Metals/D.I. H_2O	RAS	2	1 Liter	Plastic	YES	YES	NONE	

PRESERVATIVES

(19) ULTREX NITRIC ACID: MANUFACTURER: _____; LOT # _____; DATE OPENED: _____;

(20) ULTREX SULFURIC ACID: MANUFACTURER: _____; LOT # _____; DATE OPENED: _____;

(21) HYDROCHLORIC ACID: MANUFACTURER: _____; LOT # _____; DATE OPENED: _____.

CONTROLLED COPY

NON-OSR

IMPACT LEVEL

	1	2	3	4
A				
B				
C				
D				
E				
F				
G				
H				
I				
J			X	

Management Control Procedure

WP 15-PR3001

Revision 0

Generation, Storage and Control of Active WIPP Records

EFFECTIVE DATE: 1-28-94

Project Document Services

Andrew Gromko
WRITER

Phil Porter — 1-26-94
COGNIZANT INDIVIDUAL (PRINTED NAME) (SIGNATURE) DATE

Jeff J. Cotton — 1/26/94
DRC CHAIRPERSON (PRINTED NAME) (SIGNATURE) DATE

Valerie Geisler — 1-27-94
DEPARTMENT MANAGER (PRINTED NAME) (SIGNATURE) DATE

REVIEW ORGANIZATIONS

Program Management	Operations	N/A
External and Governmental Affairs	Controller	N/A
Quality and Regulatory Assurance	Office of the General Manager	N/A
Engineering	N/A	N/A
Environment, Safety and Health	N/A	N/A
Human Resources	N/A	N/A

CONTROLLED COP

TABLE OF CONTENTS

1.0 INTRODUCTION 3

2.0 REFERENCES 3
2.1 Baseline Documents 3
2.2 Referenced Documents 3

3.0 PERFORMANCE 3
3.1 General Requirements for the Generation, Storage and Control of WIPP Records 3
3.2 Generation of WIPP Records 3
3.3 Storage and Control of Active Records 7

4.0 REVIEW 8

CONTROLLED COPY

1.0 INTRODUCTION

The purpose of this procedure is to provide direction and instruction to WID employees on generating, storing, and controlling active project records.

2.0 REFERENCES

2.1 Baseline Documents

- WP 15-PR, Records Management Plan

2.2 Referenced Documents

NONE

3.0 PERFORMANCE

3.1 General Requirements for the Generation, Storage and Control of WIPP Records

3.1.1 Generate WIPP records in a manner to provide records that are legible and complete.

3.1.2 Store and control records kept in the work area in such a way as to protect them against unauthorized access, damage or loss.

NOTE Records should be filed by record series, in the order in which they will later be transferred or otherwise dispositioned.

3.1.3 Routinely add completed records to the records index for the applicable record series.

3.1.4 Do <u>NOT</u> ship active records to Project Records Services (PRS) for interim storage.

NOTE Ensuring that these steps are done on a daily routine basis will help guarantee that completed records are in compliance with requirements, and that a minimum amount of effort will be expended during the preparation of records for transfer.

3.2 Generation of WIPP Records

NOTE All WIPP records shall be generated in accordance with the requirements defined in paragraphs 3.2.1 through 3.2.6. An additional requirement for Quality Records is defined in paragraph 3.2.7.

CONTROLLED COPY

3.2.1 Legibility

NOTE A record must be legible and reproducible.

A) Determine whether the record is legible and reproducible by copying the record and then recopy the copied record (copy a copy test).

B) Inspect both copies of the record for legibility. The reason for this "copy a copy test" is to ensure that a microfilmed copy of the record can be easily read.

C) Contact PRS for directions on how to handle records that are not legible.

3.2.2 Corrections

NOTE The use of correction fluid, correction tape or write-overs is not allowed. The original, incorrect information in a record must remain legible.

A) Inspect records for errors.

B) Correct errors by drawing a single line through the incorrect information, enter the correct information, and date and initial the correction.

C) Send corrected or changed quality records which have been validated, back to the validating authority for review and revalidation.

D) Make corrections to records which have been transferred to PRS and microfilmed by issuing a supplement or revision to that record.

3.2.3 Pagination

NOTE The total number of pages of a record must be indicated.

A) Count the total number of pages in a record.

B) Number the pages using one of the following:

- The preferred method is to number each page as "1 of 10; 2 of 10", etc.
- The acceptable alternate method is to number the first page only as "1 of 10".

NOTE Paginating a quality record after it has been validated is not considered a change requiring revalidation of the record.

CONTROLLED COPY

3.2.4 Ink

NOTE Although the use of black ink is encouraged when generating records, it is not required.

A) Use black ink whenever possible to ensure maximum contrast on printed records. Records created in black ink typically produce better quality microfilm copies than do records printed in other color inks.

B) Perform the "copy a copy test" discussed above in the requirements for legibility on records generated using lighter colored inks.

3.2.5 Highlighter

NOTE The use of highlighter marking pens on records is discouraged. Some highlighters obliterate the text when it is copied or microfilmed and some highlighters cause the paper to deteriorate over time. Clouding around the text or underlining text are preferred alternatives to highlighting.

A) If a record has highlighter used on it, copy the record and check the copy for obliterated text.

B) A copy in which text is not obliterated indicates the record is legible.

C) Obtain a clean copy of the record if the copied text is obliterated.

D) Write a statement to the effect that; "the highlighted text of line -- reads '----------'. This text is obliterated when copied", when a clean copy of an obliterated record is not available. Make that statement directly on the affected page if ample room is available. If necessary, use a separate piece of paper and ensure the statement clearly defines exactly which record it applies to.

E) Sign and date the statement in either case.

3.2.6 Blank Spaces

NOTE Complete records appropriate to the work accomplished, including the completion of all blank spaces where information is required to be entered in order to make the document complete. A blank space in a record does not, in itself, make the record incomplete.

CONTROLLED COP

A) Test for document completeness as follows:

- Read the document and determine if its intent is clear and unambiguous even with some blanks not filled in. If so, it meets the requirements for a complete record

- The person who originally generated the record will review it and enter "NA" where necessary if it is not clear whether the blanks spaces represent missing information

- Mark "Best Available Copy" on any record that lacks information if the originator of the record can not be contacted.

3.2.7 Validation

NOTE In addition to the requirements above for the generation of all WIPP records, quality records have an additional requirement known as validation (authentication).

A) Indicate validation of quality records through one of the following methods:

- Signature and date by personnel authorized by the cognizant organization manager (COM)

- Other means of validation approved by the COM and the manager of PRS.

NOTE Validation of a quality record may take any reasonable form that clearly indicates that an authorized individual believes the record to be complete and accurate.

B) Validated quality records may be originals or copies.

C) Revalidate any quality record that is changed after it has been validated.

CONTROLLED COPY

3.3 Storage and Control of Active Records

3.3.1 Completed or in-process records maintained in the work area are considered active records. Handle active records in accordance with the following storage and control requirements:

A) Minimize damage, loss, or access by unauthorized personnel to records by storing the records in file cabinets or other suitable filing arrangements, when not in use.

B) Do NOT intermingle record types in the same filing space.

- Where filing space is limited, use a space divider (such as a drawer divider) to clearly separate the active records, inactive records, nonrecords, and personal papers.

C) File like records together in file series, and by the filing order shown on the RIDS covering the records, to ensure efficient transfer or disposition at the appropriate time.

D) Provide a means to retrieve active records while stored in the work area such as an index, a log, file labeling, or other mechanism.

E) Provide adequate protection of the records within the storage location to minimize the risk of loss from natural disasters, adverse weather conditions, or infestations of insects, rodents, and mold.

F) Complete a sign-out log, record-out card, or other tracking system, e.g., formal transfer letter, providing the name of the individual temporarily responsible for the record to prevent loss and ensure retrievability of records removed from active storage.

3.3.2 Including the requirements for the storage and control of all active records while maintained in the work area, quality records have the following additional requirements:

A) Store uncopied quality records in the work area in a one-hour fire rated container. The one-hour fire rated container shall bear a UL label (or equivalent) certifying one-hour fire protection, or be certified by a person competent in the technical field of fire protection.

CONTROLLED COPY

B) If a one-hour fire rated container is not available, store a copy of the record in a location sufficiently remote from the original to preclude destruction of both copies as a result of a single event such as fire or natural disaster.

C) Prevent unauthorized access to quality records in active storage by one or more of the following means:

- Locking the storage container
- Controlling personnel access to the storage location
- Other methods deemed appropriate by the responsible manager.

4.0 REVIEW

NONE

CONTROLLED COPY

ATTACHMENT B
CONTRACT LABORATORY OPERATING PROCEDURES

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Nonconformances and Corrective Action.			
Prepared by:	Approved by:	Date: 1/23/97	QA Approval:

1.0 PURPOSE

The purpose of this SOP is to establish procedures for the identification, documentation and correction of nonconformances.

2.0 APPLICATION

A nonconformance is an uncorrected deviation from the requirements of a standard operating procedure or Quality Assurance Project Plan (QAPjP). SOP QC-001, then, applies to any occurrence or practice which does not follow an SOP or QAPjP.

Nonconformances always have the potential to affect data quality. Nonconformances must be communicated to the appropriate supervisor and the Quality Assurance Manager and corrective actions approved by them. Many nonconformances require that the client be notified so that they may provide direction. A noncompliance memo is not permission to accept the error but an effort to prevent its reoccurrence.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 The nonconformance must first be identified. Remember that a nonconformance is an <u>uncorrected</u> deviation from an SOP. An out-of-control event which is immediately corrected before samples are analyzed is not a nonconformance.

5.1.1 Examples of nonconformances include:

- A sample received in an improper container, with improper preservation, or past any holding time.
- An analysis holding time not met on a sample received within the holding time. This includes a late repreparation or reanalysis which does not confirm matrix interferences suspected from an initial analysis.
- A sample lost during preparation or analysis when reanalysis within holding is not possible or when insufficient sample remains for reanalysis.

- An out-of-control blank, surrogate, LCS, internal standard, etc., whose standard corrective action (usually reanalysis) is not performed within an timely manner (within holding time).

- An out-of-control calibration whose standard corrective action is not performed.

- EQLs raised due to interferences or other factors not addressed in the SOP. This includes extracts which cannot be concentrated to their normal final volume.

- Analysis of a matrix for which the SOP does not provide standard conditions.

- An entire data set miscalculated which is discovered at any level of review.

5.1.2 Examples of occurrences which are <u>not</u> nonconformances are listed below. Many of these occurrences need to be recorded in a narrative form for inclusion in report comments.

- A sample received with missing or incomplete paperwork which is corrected in time to allow analysis within holding times.

- Use of a non-standard aliquot of a sample, even if adjustments are made to retain standard EQLs.

- An out-of-control blank whose associated samples are reanalyzed within holding time.

- An out-of-control surrogate which is repeated in a reanalysis and therefore confirms a matrix interference.

- A mechanical failure which is repaired before samples are analyzed or for which affected samples are reanalyzed within holding times.

- Random calculation errors found during data review.

5.2 The person identifying a nonconformance must immediately document it in a Nonconformance Memo (See Section 6.0, Figure 1). Describe the problem briefly but completely. Describe the actions taken to deal with the immediate occurrence. Describe the actions taken or which need to be taken to prevent a reoccurrence of the problem. Sign and date the memo on the "Corrective action initiated/performed by" line.

If you cannot perform the corrective action yourself (e.g. if a client needs to be notified and provide direction), pass the memo to the appropriate person for completion.

5.3 The person performing corrective actions must obtain the agreement of his or her supervisor that the actions are sufficient. The supervisor signs and dates the memo on the "Operations concurrence" line. In the absence of the supervisor, the Operations Manager or QA Manager may substitute.

5.4 The person performing corrective actions must also obtain the agreement of the Quality Assurance Manager that the actions are sufficient. The QA Manager will sign and date the memo on the "QA approval" line.

5.5 Corrective action must be designed to correct the problem and to minimize the probability of its recurrence. It is the ultimate responsibility of the supervisor of the affected department to ensure that corrective actions

(short and long term) are taken. A copy of the nonconformance memo should be provided to the Project Manager.

5.6 The Quality Assurance Manager maintains a log of all nonconformances and corrective actions. The QA Manager periodically reviews the log to determine if long-term corrective actions have been taken.

5.7 The Nonconformance Memo used in the RASI Laboratory is shown in Section 6.0, Figure 1. Copies of the form, which are printed on pink paper, are available from the Quality Assurance Manager.

6.0 FIGURES

Figure 1 - Nonconformance Memo

NONCONFORMANCE MEMO

Client: _____ Work Order No.: _____ Filed by: _____ Date: _____

Sample(s) Affected: _____ Test(s) _____

Area: ___ SC ___ OP ___ IP ___ II ___ GC ___ GC/MS ___ MT ___ CW ___ RP ___ Other _____

Nonconformance

A nonconformance is an uncorrected deviation from an SOP or QAPjP. Examples include (1) holding times exceeded; (2) samples which are lost during extraction or analysis when no re-prep. is possible; (3) quality control (QC) data is outside of defined limits and standard corrective action was not taken; (4) when reported detection limits are higher than specified in contract, method, and/or project plan; and (5) sample received in a broken condition, without proper preservation, or in an improper container.

Describe all nonconformances briefly but completely in the space below. Describe the action taken to deal with the immediate occurrence. Describe the root cause of the nonconformance. Describe the actions taken or which need to be taken to prevent a reoccurrence of the problem.

_____

_____

_____

_____

_____

_____

_____

CORRECTIVE ACTION

__ Client informed orally by _____ Comments: _____ Date: _____

__ Client informed in writing by (means) _____ By whom: _____ Date: _____

__ Sample(s) processed "as is". Comments: _____

__ Sample(s) on hold until _____ If released, notify: _____

Corrective action initiated/performed by: _____ Date: _____

Operations concurrence: _____ Date: _____

QA Concurrence: _____ Date: _____

CORRECTIVE ACTION VERIFICATION

__ Verified __ Cannot verify. Reason: _____

Verified by: _____ Date _____

ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title:	Water Quality Check						
Prepared by:	BES	Approved by:		Date:	2/10/97	QA Approval:	

1.0 PURPOSE

To describe the procedure for monitoring and documenting the quality of the water used in the RASI Laboratory.

2.0 SCOPE/APPLICATION

The conductivity of the water is checked daily for conductivity and documented.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Reagents/Equipment

5.1.1 Conductivity bridge, YSI Model 35

5.1.2 Conductivity cell, 0.1 cell constant, flow-through type or equivalent

5.1.3 KCl solution, purchased at approximately 1000 μmhos/cm.

5.2 Check the calibration of the conductivity meter using the KCl solution. If results are not within 10% of the calculated result for a given temperature, clean the electrode. If this does not work, calculate a factor to adjust the results.

NOTE: The correction factor used is obtained from the Correction Factor Graph. (Section 6.0, Figure 2).

5.3 Conductivity Analysis

5.3.1 The conductivity of the deionized water and LabConco water in the Metals Preparation/Wet Chemistry, Organic Preparation and Metals Laboratories are determined daily.

5.3.2 Turn on water and allow to run for about 30 seconds.

5.3.3 Collect ~ 400ml of water in a clean beaker and allow water sample to reach room before measuring.

NOTE: Conductivity is dependent on temperature at which it is measured. Temperature of measurement should be between 20° to 23° C to maintain consistency of records.

5.3.4 Set the read-out control to lowest setting on conductance meter. Take reading, calculate reading in μmhos (reciprocal ohms) per cm using following formula:

(Initial reading) **X** (Cell constant) **X** (Correction factor) = Conductivity in μmhos

5.3.5 Record results on the Daily Water Quality Check Form (Section 6.0, Figure 1).

5.3.5 The conductivity of the deionized water should be below 1.0 μmhos/cm. The conductivity of the LabConco water should be below 0.2 μmhos/cm.

5.3.6 If the conductivity reading is at or above the acceptable value, notify the QA Manager immediately.

6.0 FIGURES/TABLES

Figure 1
Water Quality Check Sheet

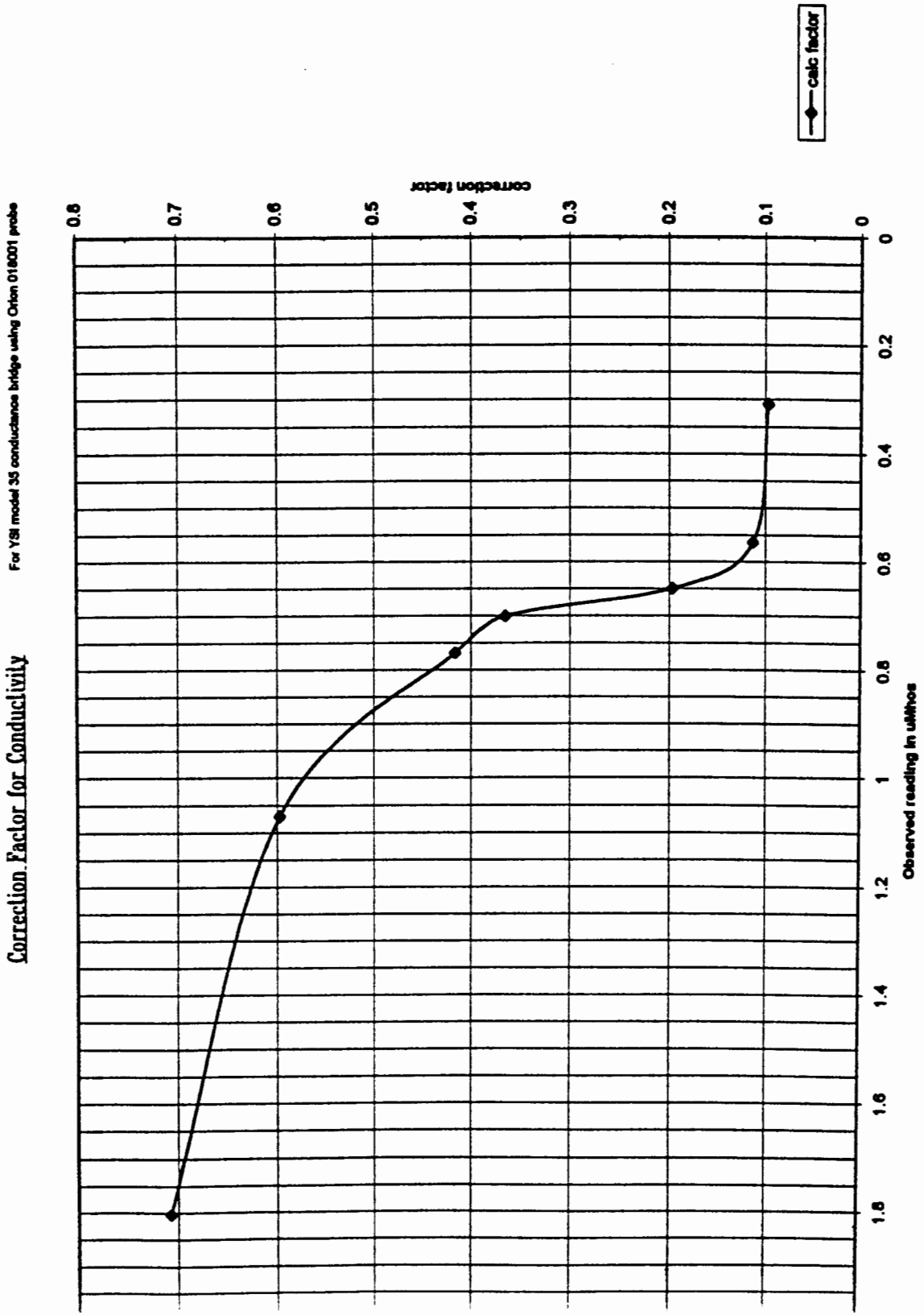


Figure 2
Correction Factor for Conductivity Sheet

Daily Water Quality Check

Reading at Room Temp	X Cell Constant	X Correction factor	=*Conductivity*	Acceptable Criteria: Deionized<1.0 Labconco<0.2 umhos/cm	
Example: 5.130	X 0.106	X 0.1	=0.0544 umhos		
Date	DI Water System	labconco unit IP lab	labconco unit MT lab	labconco unit OP lab	Analyst

Ross Analytical Services, Inc.

Table 1
Conductivity of 0.01 M KCl determined at each °C listed below.

Temperature °C	Conductivity µmhos/cm
21	1305
22	1332
23	1359
24	1386
25	1413
26	1441
27	1468
28	1496

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Preparation and Control of QA Documents.			
Prepared by:	Approved by:	Date: 11/16/94	QA Approval:

1.0 PURPOSE

To describe the procedure for the preparation and control of quality assurance (QA) documentation.

2.0 APPLICATION

This procedure applies to the Ross Analytical Services, Inc., (RASI), Laboratory Quality Assurance Manual and all Standard Operating Procedures (SOPs) prepared in and for RASI.

3.0 REFERENCES

Ross Analytical Services, Inc., Quality Assurance Manual

4.0 ASSOCIATED SOPs

AD-001, Writing Standard Operating Procedures

5.0 PROCEDURE

5.1 Preparation

5.1.1 QA Manual - The QA Manager and the Technical and Quality Director are responsible for the format and content of the RASI QA Manual. Before issuance, it must be approved by the President of RASI. The QA Manager is then responsible for its distribution.

5.1.2 SOPs - SOP preparation is performed by the supervisor or his/her designee utilizing SOP AD-001, Writing Standard Operating Procedures.

5.2 Control

5.2.1 QA Manual

5.2.1.1 Each controlled copy shall be assigned a unique control copy number.

5.2.1.2 The QA Manager shall maintain a numbered distribution list.

5.2.1.3 Uncontrolled copies of the manual will be current at the time of issue and will not be updated after distribution.

5.2.2 Standard Operating Procedures

5.2.2.1 SOPs are approved by the responsible supervisors or if written by the supervisor will be sent directly to the QA Manager. The QA Manager will review each SOP comparing it to the method(s) from which it was derived. Any deviations will be noted and discussed with both/either the Technical and Quality Director and/or the supervisor. Results of the discussion(s) will be incorporated into the given SOP. Once complete, the SOP will be sent to the Technical and Quality Director for review. The document will then be sent to typing. Following typing, the SOP will be reviewed by the QA Manager and sent to the supervisor for final comment. After final comment has been made, the SOP will be finalized.

5.2.2.2 Control copies will be made; numbered according to departmental logbooks; logged into a distribution logbook containing the SOP number, date issued, date returned, and book into which the controlled copy will be placed; and, distributed to the individual departments.

5.2.2.3 The original copy of the finalized SOP will be maintained in the office of the QA Manager.

5.3 Document Review and Updating

5.3.1 The QA Manual is reviewed and updated once every two years. Review of the QA Manual is the responsibility of the QA Manager and the Technical and Quality Director.

5.3.2 Standard operating procedures are reviewed and updated once per year. Review of SOPs are the responsibility of the appropriate supervisors in conjunction with the Quality Assurance Department.

5.3.3 Changes in procedure often occur more frequently than once a year and it is too time-consuming and costly to completely revise a document each time. An SOP Modification Form (Figure 1) does exist and should be used for interim additions or changes to controlled documents.

5.3.4 SOP Modification Forms are subject to the same review and approval process as the original document, and are then considered controlled documents.

Figure 1

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and re-issue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: _____

SOP Number: _____

Date change implemented: _____

Change initiated by: _____

Operational approval: _____

QA Approval: _____

Section to be changed: _____

Change(s): _____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: GC/MS Data Review and Verification			
Prepared by:	Approved by:	Date: 6/8/95	QA Approval:

1.0 PURPOSE

To describe the procedure for the review and quality control checking of GC/MS data and verification of the analytical reports generated from the data at the RASI laboratory.

2.0 APPLICATION

These procedures are used to verify that volatile and semivolatile data, calculations, and analytical reports are free from errors. These procedures are used for all projects.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Data Generation

The instrument operator is responsible for verifying that his/her instrument is properly calibrated and is operating within the specifications outlined in the specified method before any analyses are begun. The operator should check the work order associated with the samples to verify the proper method before any analyses are begun. If any questions regarding method or quality control remain, he should see the laboratory supervisor or team leader before beginning.

As samples are analyzed, specially designed computer software is used to generate an initial report of quantitation of the compounds of interest in the sample. Software also generates lists of surrogate recoveries, internal standard areas, and retention times for the compounds which were spiked into the samples and checks for sample concentrations exceeding the range of the curve. The operator reviews these reports for problems such as false identifications; improperly quantitated peaks; targets which have been omitted; surrogate recoveries, or internal standard areas, or retention times outside acceptance windows, or target compound amounts which are above the calibration range.

As samples are analyzed, information pertaining to those samples must be recorded on the instrument run log summary sheet. That information is as follows: Ross Sample ID, Client Sample ID, Instrument Run Number, Volume/Amount of Sample Analyzed, Dilution Factor, Time of Analysis, Autosampler Position, and any special notes about the sample. This run log page will also contain in the header the following information: Instrument ID, Analyst Name or Initials, Tape Storage ID, Column Type, GC Temperature

Program, Date of Analysis, and Date of Initial Calibration. Any unused portion of this summary page must be Z'ed out and signed by the analyst. (See Section 6.0, Figure 1 for an example run log sheet).

5.2 Data Package Assembly

After the operator has determined that the analytical results are acceptable, he then prints spectra of all target compounds which were found by the computer software. These spectra are visually compared to spectra of standards with the same compounds which were generated on the same instrument. Spectral matches are checked and noted as good when criteria are met, while compounds are removed from the quantitation report to be searched. If tentative identification of non-target peaks is requested, library search results (spectra of 3 best matches) are printed for peaks with areas which are greater than 10 percent of the nearest internal standard (up to 20 for BNA, 10 for VOA; 30 for BNAs and VOAs for CLP). The completed data package is then forwarded to the laboratory supervisor or team leader for checking.

5.3 Data Review

The laboratory supervisor (or designate) reviews all tune and calibration data, reviews all confirmations, checks all dilution calculations, and reviews a minimum of 20 percent of non-target compound tentative identifications. The first page of each sample data package is initialed. The data package summary (Section 6.0, Figures 2, 3 are example data package summary sheets for BNAs and VOAs) is completed by the final reviewer. The reviewer then prepares a draft final report. CLP data are then taken into Envision for packaging. At any stage of the review, any corrections, alterations, additions or deletions are initialed and dated.

6.0 FIGURES

Figure 1

Example Run Log Sheet

000125

ROSS ANALITICAL SERVICES, INC.

CLIENT: WORK ORDER #: METHOD:

INSTRUMENT ID:_____ | TAPE STORAGE ID#:_____ | TODAY'S DATE:_____
ANALYST:_____ | COLUMN TYPE:_____ | INIT. CAL. DATE:_____

** GC PROGRAM **

INITIAL TEMP.:_____ | INITIAL HOLD:_____ | TEMP. RATE:_____
FINAL TEMP.:_____ | FINAL HOLD:_____ | INJECTOR TEMP.:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

RUN #:_____ POSITION #:_____ | RASI SAMPLE ID:_____ | QA/QC|__|
CLIENT SAMPLE ID:_____
VOLUME/AMOUNT ANALYZED:_____ | DILUTION FACTOR:_____
NOTES:_____

ANALYST SIGNATURE:_____ APPROVAL:_____

Figure 2

Example BNA Data Package Summary Sheet

BNA's **7/93**

___ All necessary samples and MS/MSD's present and in alphanumeric order. Correct Case and SDG numbers are on the first sample (which will carry throughout batch).

___ Match Form V's with Form VI's (Initial calibrations) and Form VII's (Continuing calibrations). DFTPP must precede all other runs that day. All present and in order by date for each instrument.

___ Initial and continuing calibrations meet specifications (allowed 4 out > 25%). RIC's and quant lists have EPA names and instrument ID's. All standards have unique names. Raw data date/times match the Forms VI and VII.

___ Match DFTPP tunes with Form V's. Need one tune for each day standards or samples were run. Bars, quant lists, and RIC's labeled properly (page B-15, § b(3)). All present and in order by date for each instrument.

___ Match Blanks with Form IV's and Form V's. Need one Blank per batch of extractions. All present, have unique names, and are in order by date.

___ Each Blank Form I has proper matrix, weight or volume (30 g or 1000 mL for low level), level, GPC Y/N, pH, date extracted, and units.

___ Blanks' RIC's and spectra labeled properly.

___ Each Blank has the right number of triple plot and TIC spectra.

___ Form II present for each matrix and level, with all samples listed. Any samples with more than one acid and one base/neutral "*" must have a reanalysis with a "*" on at least some of the same surrogates. Record RE's and DL's for narrative.

___ Form III MS/MSD for each matrix and level present. Results on Form III and MS/MSD Form I's are the same.

___ Form IV's, with all samples, in order by date of analysis for each instrument.

___ Form V's DFTPP, with all samples, in order by date of analysis for each instrument.

___ Form VIII's, with all samples, present for each matrix and level. Any samples with "*" must have a reanalysis with a "*" on at least some of the same internal standards. Record RE's for narrative.

___ Each Sample Form I has proper matrix, weight or volume (~30 g or 1000 mL for low level, ~1 g for medium), level, moisture (soils only), decanted Y/N, GPC Y/N, pH, date received, date extracted, and units.

___ Each Sample's RIC and spectra labeled properly.

___ Each Sample has the right number of triple plot and TIC spectra. TIC's should not include hits from VOA fraction.

___ Each MS/MSD Form I has proper matrix, weight or volume, level, moisture, decanted Y/N, date received, date extracted, and units.

___ Section in proper order (Forms II, III, IV, V, VIII, Samples (Form 1 + raw data), Forms VI + raw initial cal data, Forms VII + raw continuing cal data, GPC calibration traces (soils only/copy from pesticides), DFTPP, Blanks, and MS/MSD's.

Figure 3

Example VOA Data Package Summary Sheet

CLP DATA PACKAGE REVIEW AND ASSEMBLY CHECKLIST

VOA'S **7/93**

___ All necessary Samples and MS/MSD's present and in alphanumeric order. Correct Case and SDG numbers are on the first sample (which will carry throughout batch).

___ pH report for water samples.

___ Match Form V's BFB with Form VI's (Initial calibrations) and Form VII's (Continuing calibrations). BFB must precede all other runs that day. All present and in order by date for each instrument.

___ Initial and continuing calibrations meet specifications (allowed 2 >25%). RIC's and quant lists have EPA names and instrument ID's. All standards have unique names. Raw data date/times match the Forms VI and VII.

___ Match BFB tunes with Form V's. Need one tune for each day standards or samples were run. Bar, quant, and RIC labeled properly (page B-11, § b(3)). All present and in order by date for each instrument.

___ Match Blanks with Form IV's and Form V's. Need one blank for each day samples were run. All present, have unique names, and are in order by date.

___ Each Blank Form I has proper matrix, weight or volume (5 g or mL for low level), level, and units. (Look for hits).

___ Blanks' RIC's and spectra labeled properly. Should be <5 x PQL.

___ Each Blank has the right number of triple plot and TIC spectra.

___ Form II present for each matrix and level, with all samples listed. Any samples with "*" must have a reanalysis with a "*" on at least some of the same surrogates. Record RE's and DL's for Narrative.

___ Form III MS/MSD for each matrix and level present. Results on Form III and MS/MSD Form I's are the same.

___ Form IV's, with all samples, in order by date of analysis for each instrument.

___ Form V's, with all samples, in order by date of analysis for each instrument.

___ Form VIII's, with all samples, present for every day on which analyes performed. Any samples with "*" must have a reanalysis with a "*" on at least some of the same internal standards. Record RE's for narrative.

___ Each Sample Form I has proper matrix, weight or volume (almost always 5 g or mL), level, moisture (soils only), date received, and units.

___ Each Sample's RIC and spectra labeled properly (especially EPA name and instrument ID).

___ Each Sample has the right number of triple plot and TIC spectra.

___ Each MS/MSD Form I has proper matrix, weight or volume, level, moisture, date received, and units.

___ VOA section in proper order (Forms II, III, IV, V, VIII, samples (Form I + raw data, Forms VI + raw initial cal data, Forms VII + raw continuing cal data, BFB, Blanks, and MS/MSD's.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Organic Preparation Data Verification							
Prepared by: BES	Approved by: CAK	Date: 6/8/95	QA Approval:				

1.0 PURPOSE

To describe the procedure for reviewing data generated in the organic prep laboratory.

2.0 APPLICATION

This procedure will be followed for all samples processed through the organic prep laboratory.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Extraction Summaries

5.1.1 Extraction summaries are reviewed for the following parameters:

Transcription errors from the laboratory notebooks or workorders to the extraction summary,

Correct surrogate entry (name, identification, volume, concentration),

Correct matrix spike entry (name, identification, volume, concentration),

Transfer of notes from notebook to extraction summary,

Correct mathematical calculations,

Completeness (presence of all necessary information).

5.1.2 Extraction summaries are reviewed on a 100% basis by a senior technician. The reviewer will initial the Extraction Summary and date it when determined that it is filled out correctly.

5.2 All paperwork associated with a group of samples (nonconformances, GC screening chromatograms) are sent along to the instrument lab with the extraction summary.

5.3 Labels on extracts are checked for transcription errors by an independent analyst prior to release of extracts from the laboratory.

5.4 Extract volumes are checked by an independent analyst prior to release of extracts from the laboratory.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Determining Method Detection Limits (MDLs), Estimated Quantitation Limits (EQLs), and Instrument Detection Limits (IDLs)			
Prepared by:	Approved by:	Date: 10/21/94	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to ensure that a systematic method for performing detection limit studies is being used throughout the laboratory.

2.0 APPLICATION

This procedure is to be used when performing detection limit studies on water, soil, or sand matrices for both inorganic and organic analyses in the laboratory.

3.0 REFERENCES

3.1 Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020

3.2 Test Methods for Evaluating Solid Waste, SW-846

3.3 Methods for Organic Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-57

3.4 U.S. EPA CLP Statement of Work for Inorganics Analysis, ILM02.0

3.5 U.S. EPA CLP Statement of Work for Organics Analysis, OLM03.0

3.6 Definition and Procedure for the Determination of the Method Detection Limit, 40 CFR Part 136, Appendix B

4.0 ASSOCIATED SOP's

None

5.0 PROCEDURE

Definitions

MDL The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

EQL The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it

may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent.

With few exceptions, reports to clients will use EQLs rather than MDLs or IDLs. Likewise, results below the EQL, even if above the MDL or IDL, are normally expressed as <EQL.

IDL Instrument detection limits can be defined as a three nonconsecutive day MDL study. Its purpose is the same as the MDL study; however the criteria and necessity for performing an IDL study comes from the inorganic Contract Lab Program statement of work.

Instrument detection limit studies shall be determined for each instrument used for inorganic CLP work at least once each calendar quarter.

The quarterly determined IDL for an instrument shall always be used as the IDL for that instrument during that quarter. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument shall be re-determined and the results used as the established IDL for that instrument for the remainder of the quarter.

5.1 <u>MDL Procedure</u>

5.1.1 Make an estimate of the MDL for the given matrix and particular analysis using one of the following criteria:

(a) Previous experience.

(b) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(c) The region of the standard curve where there is a significant change in sensitivity, e.g., a break in the slope of the standard curve.

or, (d) Instrumental limitation.

5.1.2 If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated detection limit. (It is recommended to spike at a level of between 1 and 5 times the estimated detection limit or at the EQL.)

5.1.3 If the MDL is to be determined in another matrix which does not already contain the analyte to be spiked, spike at a level that is between one to five times the estimated detection limit of that particular analyte in *reagent water*. If the matrix does contain the analyte to be spiked at a level which exceeds 10 times the MDL of that particular analyte in reagent water, obtain another blank sample with a lower level of analyte.

If an MDL study has previously been conducted on this matrix, spike according to the previous study.

5.1.4 Prepare a minimum of seven aliquots of the sample and process each through the entire analytical method.

500 Series Methods require that these replicates be analyzed over a period of several days.

5.1.5 Calculate the standard deviation of the replicate measurements. Compute the MDL as follows:

MDL = (t) * (standard deviation)

t = (Student t value appropriate for a 99% confidence level and a standard deviation estimate with **n-1** degrees for freedom.)

Student *t*-Distribution

Number of Samples	*t*-Statistic
6	3.36
7	3.14
8	3.00
9	2.90

5.1.6 If the calculated MDL is greater than the concentration used to determine it, repeat the MDL study using a higher concentration of analyte. If the calculated MDL is less than 10% of the concentration used to determine it, repeat the MDL study using a lower concentration of analyte.

500 Series Methods contain specific MDL acceptance criteria such as average percent recoveries, relative percent differences, and concentration levels which the MDL study must meet prior to being deemed ok by the SDWA.

5.2 IDL Procedure

5.2.1 Prepare a standard solution in acidified reagent water containing the analytes of interest at a concentration of 3x - 5x the instrument manufacturer's suggested IDL's or IDL's determined previously.

5.2.2 Analyze the standard solution 7 times on three nonconsecutive days according to the method. (Non consecutive means at least one day separates each pair of days on which analyses are done.)

5.2.3 Calculate the (n-1) standard deviation of0 each of the 7 replicates, then average the 3 days

5.2.4 Multiply by 3 the average of the standard deviations obtained. These will be your new IDL values.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Calibration of Fixed and Adjustable Volume Micropipets			
Prepared by:	Approved by:	Date: 1/23/97	QA Approval:

1.0 PURPOSE

To describe the procedures used to calibrate the fixed and adjustable volume micro-pipettes used at RASI.

2.0 APPLICATION

This SOP applies to all Eppendorf pipettes that dispense fixed or variable volumes of reagents or standards.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Procedure for calibrating pipettes for all volumes.

5.1.1 Tare a dried 50 mL beaker on an analytical balance.

5.1.2 Depending on the volume which is being tested, fill the pipettes with deionized water. For small volume readings, dispense at least 5 times into the weighed beaker the volume being measured. The larger volume readings may require fewer dispensations.

5.1.3 Weigh the beaker with the water and record the weight.

5.1.4 Repeat steps 5.1.1 through 5.1.3 for 3 replicates.

5.2 Calculation

Theoretical amount = Pipette setting (mL)*(# of times this volume was dispensed)

Actual amount = Total weight = [(Beaker + water wt. (g)) X 1 mL/1.0 g]

% Recovery = Actual/Theoretical X 100%

6.0 Quality Control

6.1 All pipettes should be calibrated quarterly unless some of the internal parts have been changed, in which case, the pipettes should be calibrated at that time.

6.2 The acceptable range for pipette volume is ± 5% of stated volume. If this is not obtained, repair the pipette. **Do not use until corrected**.

6.3 For adjustable volume pipettes, calibration should be done at evenly spaced intervals over the entire dispensing range of the pipettes.

6.4 All pipettes must be marked with a unique identifier and a record must be kept for each pipette and the volume for which it corresponds. This record is maintained by the individual group supervisor.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Internal Quality Assurance Audit Procedure			
Prepared by:	Approved by:	Date: 1/12/96	QA Approval:

1.0 PURPOSE

The primary reason for this procedure is to describe the timing and content of the internal quality assurance audit function.

2.0 APPLICATION

This procedure applies to the quality assurance audits conducted within each of the individual group labs.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Each year an audit schedule will be set up. The individual laboratories include Classical Wet, Inorganic Preparation, Organic Preparation, Inorganic Instrumentation/Metals, Semivolatiles, Volatiles - Gas Chromatography, Semivolatiles, Volatiles - Gas Chromatography/Mass Spectroscopy, Gas Chromatography/Mass Spectroscopy - Air Department, and Sample Control. Each group will be audited at least once a year.

5.2 During the month in which an audit is to be conducted, the QA Manager will contact the area leader of the laboratory and set up a mutually convenient time in which it can be performed.

5.3 The content of the audit is subject to change during a given year in order to remain current with the types of activities which are being conducted within each group. The current audit sheets are attached at the end of this operating procedure. These are subject to change throughout the course of a year.

ORGANIC PREPARATION AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES

Material Control

Are all materials logged through reagent control before use? (Check solvent storage cabinets under the hoods for proper labeling.)
☐Yes ☐No

Are containers with reagents/solvents in them labeled properly? (Check for reagent name, concentration, date of preparation, expiration date, ID number, and initials of preparer.)
☐Yes ☐No

Are all spike solutions prepared by either the GC or GC/MS labs verified and documented in a file in the OP lab?
☐Yes ☐No

Check the verified spike solution file to see that all verified spikes have recoveries within (85-115)% range. Are all ok?
☐Yes ☐No

Are solvent lots checked for contamination prior to use? (Check the hard copy of the solvent analysis, then check the lot number of the solvents in use to see that they have been analyzed.)
☐Yes ☐No

What procedure is being used to test for contamination and adsorption of Florisil media?

Is Florisil being tested using a 2,3,4,5-trichlorophenol solution and a pesticide/PCB standard mix in hexane?
☐Yes ☐No

Are all pesticide/PCB samples, blanks, matrix spikes put through Florisil before being sent to GC?
☐Yes ☐No

What determines when samples are put through GPC cleanup?

Are standards/solvents stored properly? (Check refrigerator, hoods, and flammable cabinets.)
☐Yes ☐No

Logbooks

Are maintenance logs available, current and complete for the sonicators, GPCs, and IR?
☐Yes ☐No

Sonicators - Is the removing of residue from probes and the polishing of tips after every 5 hours of use or as needed with an emery cloth practiced and noted?
☐Yes ☐No

GPC - Is the reseating of accessible cable connectors performed semiannually? Is this practiced and noted?
☐Yes ☐No

Is the GPC sample run log current and complete? (Check logbook.)
☐Yes ☐No

Are periodic calibrations current and complete for the sonicators? (Check logbook.)
☐Yes ☐No

Are spare parts available for the instruments above?
☐Yes ☐No

Is the refrigerator log current and complete?
☐Yes ☐No

Are the daily balance logs current and complete?
☐Yes ☐No

Is the oven log current and complete?
☐Yes ☐No

Laboratory Analysis

Are any major SOPs needed? ☐Yes ☐No

If yes, list below.

_____

_____

_____

_____

_____

_____

Have SOPs been modified in the laboratory SOP books?
☐Yes ☐No

If so, have the modifications been documented on the SOP Modification Form and submitted to the Manager of QA?
☐Yes ☐No

Have SOPs been reviewed/revised within the past year? (Check annually.)
☐Yes ☐No

Are the GPCs calibrations verified with two check mixtures (one pesticide mixture and one Aroclor mixture) once every 7 days as required by SOP # OP-004?
☐Yes ☐No

Are QC samples analyzed at the proper frequency (1 MS/MSD pair every 20 samples or 1 pair every time 5 samples are extracted in a batch; 1 LCS per batch; 1 blank per batch)? (Check the sample preparation logbooks particularly the MS/MSD and LCS logbooks.)
☐Yes ☐No

Are MDLs determined on a yearly basis for the analyses performed?
☐Yes ☐No

Are there any MDLs which are needed and have not been scheduled?
☐Yes ☐No

If yes, what are they?

_____

_____

_____

Has an analyst proficiency study been conducted for all of the staff currently analyzing samples in the lab?
☐Yes ☐No

If no, who has not performed an analyst proficiency study for what analysis?

Who	Analysis

Data Recording and Validation

Are extraction summary sheets and notebooks complete with test, method, analyst, date, client, solvent and Florisil (if applicable) lot numbers? (Check extraction summary logbook.)
☐Yes ☐No

Are extraction summary sheets and notebooks legible and complete?
☐Yes ☐No

Are data recorded in black ink?
☐Yes ☐No

Are errors corrected by drawing a line through the error, writing in the correct entry, and initialing and dating the correction? (Check data sheets and logbooks.)
☐Yes ☐No

Are data and procedural technique recorded consistently?
☐Yes ☐No

Are extraction summaries reviewed on a 100 % basis, checked off, then initials placed after the entry?
☐Yes ☐No

Other

Does the glassware and preparation apparatus appear to be in good shape?
☐Yes ☐No

When was the last date that glassware was fire polished?

INORGANIC INSTRUMENT/METALS AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES

Material Control

Are standard and reagent preparation documented properly? (Check logbooks.)
☐Yes ☐No

Are standard/reagent labels complete with standard/reagent number, concentration, dates of preparation and expiration, and name of analysts?
☐Yes ☐No

Are standards/reagents stored properly?
☐Yes ☐No

Are expired standards/reagents disposed of promptly?
☐Yes ☐No

Logbooks

Before an analyst begins to run an instrument, is the routine preventive maintenance check sheet filled out?
☐Yes ☐No

Are spare parts inventories available for the instruments in the lab?
☐Yes ☐No

Does the supervisor review the maintenance log on a monthly basis?
☐Yes ☐No

Are there IDL schedules present and posted for both ICP and GFAA instruments?

Laboratory Analysis

Are any major SOPs needed? If yes, list below.

_____

_____

_____

_____

_____

_____

Are nonconformances/deficiencies filed when necessary?
☐Yes ☐No

Are MDLs determined on a yearly basis for the analyses performed?
☐Yes ☐No

Are there any MDLs which are needed and have not been scheduled?
☐Yes ☐No

If yes, what are they?

_____

_____

_____

Has an analyst proficiency study been conducted for all of the staff currently analyzing samples in the lab?
☐Yes ☐No

If no, who has not performed an analyst proficiency study for what analysis?

Who	Analysis

Data Recording and Validation

Are data sheets complete with test, method, analyst, date, client? (Check current data sheets.)
☐Yes ☐No

Are data recorded in black ink?
☐Yes ☐No

Are errors corrected by drawing a line through the error, writing in the correct entry, and initialing and dating the correction?
☐Yes ☐No

Are data recorded consistently?
☐Yes ☐No

Are out-of-control QC samples documented along with the corrective action?
☐Yes ☐No

Are data validated 100% for correctness and accuracy prior to leaving the department.
☐Yes ☐No

GC AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES

Material Control

Are COAs sent to the Quality Assurance Manager?
☐Yes ☐No

Is standard preparation documented properly?
☐Yes ☐No

Are all standards used in the GC department verified and approved before use?
☐Yes ☐No

Does GC have sufficient notice to produce and verify standards produced for OP prior to use?
☐Yes ☐No

Is documentation present in the GC lab showing that standards are verified for accuracy prior to being used?
☐Yes ☐No

Do all standards fall within (85 - 115)%?

When new volatile standards are prepared, are responses checked against previous curve to see if they fall within +/- 50% of curve responses?

Are 2nd party standards being run whenever new *internal standard* stock solutions are prepared?
☐Yes ☐No

Are standards identified uniquely and traceable to their preparation? Trace 3 standards back to storage locations.
☐Yes ☐No

ID	Location	Found

Are standard/reagent labeled so that they are traceable to the preparation logbooks?
☐Yes ☐No

Are standards/reagents stored properly?
☐Yes ☐No

Are expired standards disposed of promptly?
☐Yes ☐No

Logbooks

Are maintenance logs current and complete for each individual GC instrument?
☐Yes ☐No

Instrument	Issue

Are refrigerator logs current and complete?
☐Yes ☐No

Is the desorption efficiency logbook current and complete?
☐Yes ☐No

Are standard preparation logs current and complete?
☐Yes ☐No

Are GC run logs current and complete?
☐Yes ☐No

Instrument	Issue

Does the supervisor or another analyst review and sign logbooks on a monthly basis?
☐Yes ☐No

Laboratory Analysis

Are any major SOPs needed? If yes, list below.
☐Yes ☐No

_____

_____

_____

Are QC samples analyzed at the proper frequency?
☐Yes ☐No

Are method blanks run for every analysis?
☐Yes ☐No

Is a LCS being analyzed for both volatiles and semivolatiles?
☐Yes ☐No

One MS/MSD pair for every 20 samples? Check volatile QC log.
☐Yes ☐No

Are MDLs determined on a yearly basis for the analyses performed?
☐Yes ☐No

Are there any MDLs which are needed and have not been scheduled?
☐Yes ☐No

If yes, what are they?

_____

_____

_____

Has an analyst proficiency study been conducted for all of the staff currently analyzing samples in the lab?
☐Yes ☐No

If no, who has not performed an analyst proficiency study for what analysis?

Who	Analysis

Are nonconformances/deficiencies filed when necessary?
☐Yes ☐No

Data Recording and Validation

Are data packages complete with supporting information and problems documented?
☐Yes ☐No

Is data recorded in black ink?
☐Yes ☐No

Are errors corrected by drawing a line through the error, writing in the correct entry, and initialing and dating the correction?
☐Yes ☐No

Does supervisor review and sign data sheets prior to transmittal?
☐Yes ☐No

Is data recorded consistently?
☐Yes ☐No

Does the analyst report data in a final form?
☐Yes ☐No

Are out-of-control QC samples documented along with the corrective action?
☐Yes ☐No

Other

Are spare parts inventories available?
☐Yes ☐No

GC/MS AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES

Material Control

Are COAs sent to the QAM?
☐Yes ☐No

Are standards identified uniquely and traceable to their preparation?
☐Yes ☐No

Is standard preparation documented properly?
☐Yes ☐No

Are all standards verified and approved before use?
☐Yes ☐No

Are standards labeled appropriately complete with standard, concentration, dates of preparation and expiration, and name of preparer or is there sufficient information present on each standard which allows others to find this information by using a given number to refer back to a notebook?
☐Yes ☐No

Are standards stored properly?
☐Yes ☐No

Are expired standards disposed of promptly?
☐Yes ☐No

Logbooks

Are maintenance logs current and complete for the instruments in the lab, as described in SOP MV-003? (Check to see that the preventive maintenance check sheets are in use.)
☐Yes ☐No

Instrument	Issue

Are spare parts inventories available?
☐Yes ☐No

Are refrigerator logs current and complete?
☐Yes ☐No

Are standard preparation logs current and complete?
☐Yes ☐No

Is standard traceability/comparison documentation current and complete?
☐Yes ☐No

Are run logs current and complete?
☐Yes ☐No Which logs are not up to date?

Instrument	Issue

Does the supervisor review and sign logbooks on a monthly basis?
☐Yes ☐No

Laboratory Analysis

Are any major SOPs needed? If yes, list below.
☐Yes ☐No

_____

_____

_____

_____

_____

Are QC samples analyzed at the proper frequency?

One MS/MSD pair for every 20 samples?
☐Yes ☐No

Are method blanks run for every analysis?
☐Yes ☐No

Are LCSs analyzed at the proper frequency?
☐Yes ☐No

Are MDLs determined on a yearly basis for the analyses performed?
☐Yes ☐No

Are there any MDLs which are needed and have not been scheduled?
☐Yes ☐No

If yes, what are they?

_____

_____

_____

Has an analyst proficiency study been conducted for all of the staff currently analyzing samples in the lab?
☐Yes ☐No

If no, who has not performed an analyst proficiency study for what analysis?

Who	Analysis

Are nonconformances/deficiencies filed when necessary?
☐Yes ☐No

Data Recording and Validation

Are data packages complete with supporting information and problems documented?
☐Yes ☐No

Are data recorded in black ink?
☐Yes ☐No

Are errors corrected by drawing a line through the error, writing in the correct entry, and initialing and dating the correction?
☐Yes ☐No If not, state the location and page number of an example

Is data recorded consistently?
☐Yes ☐No

Does the analyst report data in a final form?
☐Yes ☐No

Are out-of-control QC samples documented along with the corrective action?
☐Yes ☐No

SAMPLE CONTROL AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES

Sample Receipt and Inspection

Are sample containers checked for damage?
☐Yes ☐No If damaged, is customer notified?

Are samples checked for proper identification against the chain-of-custody?
☐Yes ☐No

Are samples checked for proper preservation?
☐Yes ☐No

Is at least one sample checked for temperature upon receipt of a cooler?
☐Yes ☐No

Are sample volumes checked for adequacy?
☐Yes ☐No

If any of the above occur, are the details documented on a Nonconformance Form?
☐Yes ☐No

Refrigerators

Are logs for the six refrigerators current and complete?
☐Yes ☐No

Are Nonconformance Forms filed when an abnormality exist in a given refrigerator?
☐Yes ☐No

Internal Logs and Chain-of-Custody

Are internal chain-of-custody forms complete?
☐Yes ☐No If not, what is missing from each log?

Are internal logs legible?
☐Yes ☐No

Have staff and their supervisors been notified by letter if samples which they had in their possession have not been logged back into Sample Control?
☐Yes ☐No

Sample Storage and Discards

Is adequate space available for sample storage?
☐Yes ☐No

Choose three random samples and locate them according to where their work order says they will be. Can all three of them be located?
☐Yes ☐No

ID	Location	Found

Are discards up-to-date?
☐Yes ☐No

Is documentation available regarding when discard samples were transferred to the SHE department?
☐Yes ☐No

Other
Is the PC File up-to-date and complete?
☐Yes ☐No

Are bottle certifications for cleanliness kept in Sample Control?
☐Yes ☐No

Are there items needed in this department which are not covered above?
☐Yes ☐No List.

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

WET CHEMISTRY AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES, INC.

Material Control

Are all purchased materials logged through reagent control before use? (Check the chemical storage cabinet for proper labeling.)
☐Yes ☐No

Are reagent number, date received, date opened, and expiration dates placed onto the labels for the purchased chemicals?
☐Yes ☐No

Are prepared standard and reagent labels complete with standard/reagent number, concentration, dates of preparation and expiration, and name of analyst?
☐Yes ☐No

Are all standards and reagents stored properly? (Check the chemicals in the chemical cabinet for correct storage location according to the different hazard ratings.)
☐Yes ☐No

Are all expired standards and reagents disposed of promptly? (Check dates on standard and reagent labels.)
☐Yes ☐No

Are acidic distillation wastes neutralized prior to disposal?
☐Yes ☐No

Logbooks

Are maintenance logs current and complete for the following instruments?
☐Yes ☐No

UV/Vis spectrophotometer

A. Is the response of the UV/Vis using commercial solutions checked quarterly? (Review log.)
☐Yes ☐No

B. Is the instrument wavelength checked with the internal wavelength scan quarterly? (Review log.)
☐Yes ☐No

Lachat spectrophotometer
☐Yes ☐No

IC instrument
☐Yes ☐No

Does the lab have a spare parts inventories list for the various instruments? Are they complete and up to date?
☐Yes ☐No

Are reagent/standard preparations documented properly? (Review log.)
☐Yes ☐No

Does the supervisor review and initial the logbooks?
☐Yes ☐No

Check QA/QC Files and Current Working Data Sheets:

Are balance logs current and complete?
☐Yes ☐No

Are oven/refrigerator logs current and complete?
☐Yes ☐No

Are the quarterly calibrations for the autopipets current and complete?
☐Yes ☐No

Are the balances checked to 95% of their capacity quarterly? Have they been certified yearly by an outside contractor?
☐Yes ☐No

Are thermometers checked annually?
☐Yes ☐No

Laboratory Analysis

Are QC samples analyzed at the proper frequency? (Review current data sheets.)
☐Yes ☐No

Are holding times posted in the lab?
☐Yes ☐No

Is oven temperature recorded at the beginning and end of each cycle?
☐Yes ☐No

Is a scheduling calendar maintained for BOD analysis?
☐Yes ☐No

Is the BOD incubator temperature recorded twice daily when in use?
☐Yes ☐No

Are MDLs determined on a yearly basis for the analyses performed?
☐Yes ☐No

Are there any MDLs which are needed and have not been scheduled?
☐Yes ☐No

If yes, what are they?

_____

_____

_____

Has an analyst proficiency study been conducted for all of the staff currently analyzing samples in the lab?
☐Yes ☐No

If no, who has not performed an analyst proficiency study for what analysis?

Who	Analysis

Data Recording and Validation

Are data sheets complete with test, method, analyst, date, and client? (Check files.)
☐Yes ☐No

Are data sheets checked? (Check files.)
☐Yes ☐No

Does the supervisor review and sign data sheets? (Check files.)
☐Yes ☐No

Are data recorded in black ink? (Check files.)
☐Yes ☐No

Are errors corrected by drawing a line through the error, writing in the correct entry, and initialing and dating the correction? (Check files.)
☐Yes ☐No

Are data recorded in the same format consistently? (Check files.)
☐Yes ☐No

Does the analyst report data in a final form with all calculations performed?
☐Yes ☐No

Are out-of-control QC samples documented along with the corrective action or are all reworks included in the data packages?
☐Yes ☐No

Are data and calculations checked on a 100% basis before being accepted?
☐Yes ☐No

Other

Are any major SOPs needed?
☐Yes ☐No If yes, list below.

_____

_____

_____

_____

_____

Are SOPs available for use in the lab?
☐Yes ☐No

Has the WC lab updated or modified any SOPs internally to the WC lab without using a modification form?
☐Yes ☐No

Which SOPs have been modified?

_____

_____

_____

Is documentation for the training of analysts in current and new methods complete?
☐Yes ☐No
Are the laboratory area and balance room weighing area clean?
☐Yes ☐No
Comments _____

_____

_____

_____

INORGANIC PREPARATION AUDIT CHECKLIST

ROSS ANALYTICAL SERVICES, INC.

Material Control

Are all materials logged through reagent control before use, excluding high volume acids?
☐Yes ☐No

Are containers containing reagents/standards/solutions labeled properly? (Check for reagent name, concentration, date of preparation, expiration date, ID number, and initials of preparer.)
☐Yes ☐No

Are reagents/solutions traceable from label to preparation procedure to chemical stock used for preparation?
☐Yes ☐No

Are all reagents/standards/solutions stored properly?
☐Yes ☐No

Are expired reagents/standards/solutions discarded?
☐Yes ☐No

Logbooks

Are reagents/solutions preparation documented properly?
☐Yes ☐No

Laboratory Analysis

Are QC samples (blanks, LCSs, MS/MSDs) analyzed at the proper frequency?
☐Yes ☐No

Are MDLs determined on a yearly basis for the analyses performed?
☐Yes ☐No

Are there any MDLs which are needed and have not been scheduled?
☐Yes ☐No

If yes, what are they?

_____

_____

_____

Has an analyst proficiency study been conducted for all of the staff currently analyzing samples in the lab?
☐Yes ☐No

If no, who has not performed an analyst proficiency study for what analysis?

Who	Analysis

Data Recording and Validation

Are data sheets complete with test, method, analyst, date, and client? (Check files.)
☐Yes ☐No

Are data recorded in black ink? (Check files.)
☐Yes ☐No

Does supervisor review and sign data sheets? (Check files.)
☐Yes ☐No

Are data sheets checked? (Check files.)
☐Yes ☐No

Are errors corrected by drawing a line through the error, writing in the correct entry, and initialing and dating the correction? (Check files.)
☐Yes ☐No

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Requirements for Proof of Analyst Proficiency			
Prepared by:	Approved by:	Date: 9/12/94	QC Approval:

1.0 PURPOSE

The purpose of this SOP is to define the minimum criteria for an analyst to prove proficiency in a given analytical procedure.

2.0 APPLICATION

These criteria apply to all analyses performed at Ross Analytical. An analyst must prove proficiency in a method, as described below, before performing the method on client samples without the direct supervision of the department's Group Leader.

3.0 REFERENCES

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", EPA 600/4-82-057
"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846

4.0 ASSOCIATED SOP'S

Each operating department has individual training SOP's which must include this SOP by reference.

5.0 PROCEDURE

Chromatography analyses have different criteria for proof of analyst proficiency than metals and wet test analyses, because most chromatography reference methods include specific proficiency criteria. A section is devoted to each of those two major categories. The Group Leader must notify the Manger of Quality Assurance when he/she determines that an analyst has sufficient training to be tested for proficiency.

5.1 Chromatography (GC and GC/MS)

Prepare stock solutions of each method's targets at the concentrations specified below. Commercial solutions which approximate these concentrations or are multiples of them are acceptable. Prepare samples for analysis from the stocks as described. In each case, use the normal RASI target list for the method (i.e., the "HSL", not Appendix IX).

The analyst must set up (if appropriate), tune, and calibrate the instrument before running the samples, as those steps are part of the analysis. Spike and analyze the four working solutions described for each method. Calculate the mean recovery (Xbar) and the standard deviation of the recoveries (s) in µg/L for each target.

For 8000 series methods (SW-846), compare Xbar and s for each analyte with the corresponding acceptance criteria for accuracy and precision, respectively, given in the QC Acceptance Criteria Table at the end of each method. If Xbar and s for all targets are within the limits, the analyst is considered proficient. If the method includes an extraction, the person who did the extraction is considered proficient as well.

For 600 series methods (EPA 600/4-82-057) compare Xbar and s obtained for each analyte (Xbar-obt and s-obt) with the corresponding expected Xbar and s (Xbar-exp and s-exp) given in the Accuracy and Precision Table at the end of each method. If $|\text{Xbar-obt} - \text{Xbar-exp}| < \text{s-exp}$ or if $\text{s-obt} < 2\text{s-exp}$ the analyst is considered proficient. If the method includes an extraction, the person who did the extraction is considered proficient as well.

For TO-14, calculate the mean recovery (Xbar) and the standard deviation of the recoveries (s) of each analyte. If 80% <= Xbar <= 120% and s <= 20%, the analyst is considered proficient.

If any analytes fail either criterion, locate and correct the source of the problem. Repeat the test for the analytes which failed.

5.1.1 Volatile Organics (601, 602, 624, 8010, 8020, 8021, 8240, & 8260)

Prepare a stock QC Check Sample (QCCS) containing each target at 10 mg/L. Dilute 200 µL of the stock to 100 mL. If a stock has a different concentration, dilute so the working QCCS concentration is 20 µg/L. Analyze four 5 mL aliquots of the working QCCS.

5.1.2 Pesticides/PCB's (608 & 8080)

Prepare stock QCCS's containing 10 mg/L each of DDD, DDT, endosulfan II, endosulfan sulfate, and endrin and 2 mg/L of each other single-component target. Prepare QCCS's for technical chlordane, toxaphene, and PCB's at 50 mg/L. Add 1 mL to each of four 1 L aliquots of reagent water to make the working QCCS. If a stock has different concentrations, dilute so the working QCCS concentrations are 10 µg/L, 2 µg/L, and 50 µg/L for the subsets described above. Extract and analyze.

5.1.3 PAH's by GC (610 & 8100 for BUSTR)

Prepare a stock QCCS containing each PAH at 100 mg/L. Add 1 mL to each of four 1 L aliquots of reagent water. If a stock has a different concentration, dilute so the working QCCS concentration is 100 µg/L. Extract and analyze.

5.1.4 Semivolatile Organics (625 & 8270)

Prepare a stock QCCS containing each target at 100 mg/L. Add 1 mL to each of four 1 L aliquots of reagent water to make the working QCCS. If a stock has a different concentration, dilute so the working QCCS concentration is 100 µg/L. Extract and analyze.

5.1.5 Organophosphate Pesticides and Phenoxy Acid Herbicides (8140 & 8150)

Prepare a stock QCCS containing each target at 10 mg/L. Add 1 mL to each of four 1 L aliquots of reagent water to make the working QCCS. If a stock has a different concentration, dilute so the working QCCS concentration is 10 µg/L. Extract and analyze.

5.1.6 Other Extractable Organics (e.g. 8040 & 8120)

Prepare stock QCCS's as described in Section 8.5 of EPA Method 3500. Add 1 mL to each of four 1 L aliquots of reagent water. Extract and analyze.

5.1.7 TO-14

Analyze four aliquots of a standard whose analyte concentrations are approximately 10 times their EQL's.

5.1.8 Other Air ("TO") Methods

For those "TO" methods requiring an extraction, prepare stock QCCS solutions as described for the most nearly similar SW-846 method. Spike four sets of collection media, extract and analyze. Use the acceptance criteria from the SW-846 method.

5.2 Metals and Wet Chemistry

Prepare or purchase a standard solution whose concentration is approximately 10 times the analyte's EQL. Analyze four aliquots of the standard. If the method requires or normally includes digestion, distillation, or other preparation, include those steps. If the analysis uses an instrument, the analyst must set it up (if appropriate) and calibrate it before running the samples, as those steps are part of the analysis.

Calculate the mean recovery (Xbar) and the standard deviation of the recoveries (s) of the analyte (or of each analyte if the test has multiple targets). If 80% <= Xbar <= 120% and s <= 20%, the analyst is considered proficient. If the method includes a preparation step, the person who did the preparation is considered proficient as well.

If any analyte fails either criterion, locate and correct the source of the problem. Repeat the test for the analytes which failed.

6.0 RECORDKEEPING

The Quality Assurance Manager will keep the summary records of analyst proficiency. The summaries will include the method number, analyte (or target list), and date of the analysis which met acceptance criteria. The QAM will also retain the raw data and statistical calculations for proof of proficiency analyses.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Subcontractor Approval Process				
Prepared by:	Approved by:	Date: 9/29/94	QC Approval:	

1.0 PURPOSE

The purpose of this procedure is to define the requirement which must be met before a laboratory can be used by Ross Analytical Services, Inc., (RASI) to perform analyses on a subcontracting basis.

2.0 APPLICATION

This procedure is to apply to all laboratories to which RASI intends to send subcontracting work.

3.0 REFERENCES

Ross Analytical Quality Evaluation, F.S. Ordway

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

The subcontractor approval process includes, at a minimum, an examination of the Quality Assurance Program Plan (QAPP) for proposed subcontractor and related documents such as SOPs. Results of third-party performance evaluation (PE) samples for the analytes in question will also be examined, if available. The approval process also includes the submission and evaluation of single- or double-blind PE samples.

For subcontractors under consideration as participants in large projects, multiple analyses, complex analyses, or as general "back-up" labs, an on-site audit is required.

Subcontractors specified by clients are exempt from the approval process.

5.1 Evaluation of Minimum Requirements

This section describes the supporting documentation and the criteria therein which must be reviewed by the Quality Assurance (QA) Department. This evaluation must take place before performing an on-site audit. All questions and findings generated by the review process will be summarized and asked of the subcontractor.

5.1.1 Laboratory Quality Assurance Manual

The QA Manual should contain the following information whether in the below described areas c in a different form altogether. If an area is missing, this must be documented by the reviewer.

5.1.1.1 Introduction

5.1.1.1.1 Approach of Management Toward Quality

In general, this section should describe the commitment of management to quality through a continuous improvement process.

5.1.1.1.2 Objectives of the QA Program

This section should address the systems which control documents, laboratory organization, standard lab practices, material procurement and control, sample receipt and testing, calibration procedures, preventive maintenance, quality control (QC) samples, analytical procedures, data validation and verification, analysis of statistical data, data reports, records management, nonconformances and corrective action, internal audits, quality reports to management, and software QC.

5.1.1.1.3 QA Documents and Files

The various QA documents generated and where they are stored should be described in this section. These include balance calibration sheets, internal audit reports, control charting and control limit documentation.

5.1.1.2 Laboratory Organization

An organizational chart, description of facilities, equipment, and facility security should be discussed in this section.

5.1.1.3 QA Objectives for Measurement of Data in Terms of Precision, Accuracy, and Completeness

5.1.1.3.1 Data Precision and Accuracy

The purpose of each type of quality control sample used in the lab and how it generates information about the measurement of precision and accuracy should be present in this section. Method-specific information should be present which describes the acceptance criteria and actions to take when criteria are not met.

5.1.1.3.2 Data Completeness

Completeness should be generally discussed and define how completeness is determined for any given project.

5.1.1.4 Sample Containers, Preservation and Holding Times

This section should include a discussion of how the lab meets the DOT shipping requirements, the procedure for cleaning and preparing shipping containers, and

informative tables documenting sample containers, sample volumes required for specific analyses, matrix-specific sample preservation, and holding time requirements.

5.1.1.5 Sample Custody

The sample custody process should be described including inspection of samples, verification of sample receipt against the chain-of-custody form, sample storage, and the logging and tracking of samples throughout the analytical sequence.

5.1.1.6 Calibration Procedures and Frequency

Check this section to see if that it contains information describing what the calibration frequency and procedures are for given methods and instruments.

5.1.1.7 Analytical Methods

A listing of methods with their sources and detection or reporting limits should be included in this section.

5.1.1.8 Data Reduction, Validation, Reporting and Records Management

Requirements for data reduction and validation should include the actions taken at each level of data processing. This section should describe the procedures for data acquisition, the storage and security of computer readable data, auditing computer readable data, the system used to prevent errors and the tracking of them, making changes to data already submitted to clients, and the chain-of-command and the responsibilities regarding the resubmission of corrected data.

This section should conclude with a description of what records are generated, who generates and manages them, and documentation of the system for filing and long term storage of them.

5.1.1.9 Project Files

This section should address information which is contained in each project file.

5.1.1.10 Internal Quality Control

Check the materials procurement information, look for type of water used, glassware preparation procedure, and compressed gas purity.

5.1.1.11 Performance and System Audits

This section should include definitions of the types of audits performed in the lab, the frequency of audits, and any performance evaluation samples which the lab regularly performs.

5.1.1.12 Preventative Maintenance

Tables defining the preventative maintenance for each type of instrument present in the lab and an inventory of the parts maintained on-hand should be documented in this section.

5.1.1.13 Procedures Used to Assess Data Precision, Accuracy, and Completeness

Specific practices used to evaluate precision and accuracy should be included in this section. This section should detail the calculations to be used to generate each of the categories and describe what steps are taken to document and utilize the information generated.

5.1.1.14 Nonconformance and Corrective Action

A nonconformance must be defined and the procedure used when such an event occurs should be described. Description of the documentation flow and the steps which occur when both deciding upon a corrective action and follow-up actions should be detailed.

5.1.1.15 Quality Assurance Reports to Management

The frequency and types of information included in reports to management should be described in this section.

5.1.1.16 Training

This section should include the types of training which are offered to each individual in the laboratory. It should be detailed enough to specify training courses or procedures which are to be provided to the different departmental heads, and bench level personnel each year and at the beginning of employment.

5.1.1.17 Software Quality Control

This section should document all the steps taken whenever either new software is installed or when a software package is being edited.

5.1.2 Methods Being Used

Documentation of what methods the subcontractor is using must be present; this information may be included in the QA Manual. Check to ensure that the methods employed are consistent with methods which our clients and RASI agree upon. Document any points of contention.

5.1.3 Standard Operating Procedures (SOPs) and Content

SOPs must be present for each analytical method which RASI will require the subcontractor to perform. If not available, the subcontractor must write a SOP, which is adequate for our client's needs, prior to being placed onto our approval list. EPA methods are not SOPs.

SOPs must address all of the major areas described in RASI SOP AD-001, Writing Standard Operating Procedures. If a SOP does not contain an important element or step which the RASI QA Department deems necessary, then RASI may request a change in the SOP be made or that written clarification be provided to RASI in order to satisfy the conditions which we will accept.

5.1.4 Method Validation Studies

RASI must receive a copy of the below listed studies and review them to ensure that they meet both the criteria of the methods, including performing a study on a given frequency, and the

requirements of our customers. If it is found that the studies are out of date or that they do not meet our needs, RASI will request that a new study be completed prior to deciding upon accepting or denying approval of the subcontractor.

5.1.4.1 Method Detection Limits

RASI must request that MDL studies be provided for any analysis which RASI is to subcontract out. These studies must have been performed according to either 40 CFR, Part 136, Appendix B or SW-846, Chapter One.

If a MDL study has not been performed, RASI must request that it be performed and provided us along with the MDL data prior to continuing the approval process.

5.1.4.2 Linear Range Studies, Interelement Correction Factors, Instrument Detection Limits (IDLs) for Metals

In order for RASI to permit a subcontractor to perform analyses for metals work, RASI must receive documentation for the above three studies which are current and meets the needs of RASI and its client(s). These studies are not performed by non-CLP laboratories on a regular basis; however, this documentation must be present prior to subcontractor approval.

5.1.4.3 Analyst Proficiency Studies

An analyst proficiency study is a one-time demonstration that an analyst is capable of performing an analysis within the guidelines of a given method. This is generally accomplished by the analysis of quadruplicate samples where sample results must conform to either method criteria or laboratory criteria for precision and accuracy. Analyst proficiency documentation must be available for all analyst who will be performing work for RASI. The QA Department of RASI must check to make sure the proficiency data pass the given method criteria, if any, and to ensure that documentation is available for each analyst contained on the organizational chart. If this documentation is not available, RASI must request and receive it prior to continuing the process of reviewing the subcontractor.

5.1.5 Third-Party Performance Evaluation (PE) Results

The QA Department of RASI must request and review all third-party PE results for the previous one year. All problem areas must be documented by the QA staff.

5.1.6 Independent Audit Summary and Response

As with the third-party PE results, the QA Department of RASI will review all independent audit summaries and responses provided by the subcontractor. All problem areas must be documented by the QA staff.

5.1.7 Submittal and Review of QC Samples

RASI will submit a QC sample to the potential subcontractor prior to conducting an on-site audit. If results for the desired analytes are acceptable, proceed to Section 5.2. If results are not correct, then the QA Department must decide whether to pursue this lab further.

5.2 On-site Audit

5.2.1 Purpose

The on-site audit serves many purposes, the main one is to verify that the quality systems described in the QA Manual and SOPs are being implemented. Second, this audit will verify whether or not the systems are adequate for the needs of RASI.

5.2.2 Audit Preparation

5.2.2.1 Prior to visiting a potential subcontractor, the QA Department will provide the lab with a copy of the checklist which will be used during the audit. Any information which the RASI team will require at time of visit such as a data packet and its accompanying raw data must be documented in a letter to the potential subcontractor and sent to them at least one week in advance of the on-site audit.

5.2.2.2 Once 5.2.2.1 has occurred, the potential subcontractor must reply in writing that the above information is available prior to the visit.

5.2.2.3 In order for RASI to perform a method audit, RASI must provide the potential subcontractor with a list of the particular method(s) which will be audited. All method supporting documentation listed in Section 5.1 must be on hand at time of audit. If this data/information is not available, the laboratory must inform RASI in advance.

5.2.3 Audit Format

5.2.3.1 Introductory Meeting

This introductory meeting should take place between RASI personnel and the potential subcontracting personnel. This may include direct supervisors, bench-level analyst, quality personnel, someone from sales, and others.

The meeting should address the focus of potential project(s) at hand and the agenda for the audit.

5.2.3.2 Tour of Facility

Like most audits a logical approach for a tour is to start at the point at which a sample enters the laboratory and follow it through to sample disposal.

During the tour, the RASI team will assess general laboratory practices, e.g., chain-of-custody documentation and up-keep, logbooks, balance calibration, refrigerator and oven temperature records, LIMS documentation, and other items.

5.2.3.3 Method Audits

RASI will conduct method audits for individual analyses which the QA Department deems necessary. The RASI team will discuss the method with bench-level analysts to get an understanding of the skill and knowledge which they have acquired and utilize in their positions. Specifically, RASI will be looking for method know-how and at what decision-making expectations are being made of the analysts.

5.2.3.4 Data Audit

A final report will be reviewed for the quality control elements (LCSs, MS/MSD pairs, Duplicates) provided to each client; the raw data of this report will likewise be reviewed. Extraction and preparation sheets will be reviewed for completeness, e.g., check to see that the correct solvents or reagents are being used for particular analyses and that the solvent or acid lot numbers are indicated on the sheet. Documentation for the verification of "cleanliness" will be reviewed for any solvent lots used in this report. Spikes and standards traceability will be reviewed and followed through all the documentation steps. Calibrations and tuning data will be reviewed for method compliance. Run logs will be reviewed. SOP conformance will be checked.

5.2.3.5 Concluding Meeting

The RASI team will present recommendations and requirements to the assembled group of individuals representing the potential subcontractor. These recommendations and requirements will be included in the final report and must be met prior to the lab becoming an approved subcontractor.

5.2.4 Follow-up

RASI is not required to follow-up any further than an initial written notification; however, if deemed necessary, RASI may conduct a follow-up audit.

When a subcontractor passes an audit, a letter confirming that they meet our requirements will be sent by the RASI QA Department. This letter will indicate the analyses for which the laboratory is approved to perform on RASI samples. It should be noted that passing an audit does not guarantee work.

5.2.5 Frequency

On-site audits must be repeated every other year in order to continue to be an approved RASI subcontractor.

5.2.6 Confidentially

RASI personnel will honor the confidentiality of the subcontractor and any of its clients. If asked, RASI personnel may sign a statement to this effect.

RASI requires non-RASI employees to honor the confidentiality of this document and our auditing procedure. All RASI samples and results are considered confidential.

Audit Checklist

Requirements	Auditor Comments
1.0 General Lab Review	
Does managerial staff have authority and resources needed to perform their functions? ☐Y ☐N ☐NA	
Is the lab organized in such a way that confidence in its independence of judgement and integrity is maintained at all times? ☐Y ☐N ☐NA	
Are the responsibility, authority, and interrelations of personnel who manage, perform or verify work coordinated in such a manner that the quality of work is not jeopardized? Note and document. ☐Y ☐N ☐NA	
Are the supervisors familiar with the test methods, procedures used in the lab, the objective of the analysis, and how to assess the results? ☐Y ☐N ☐NA	
Is the ratio of supervisory to non-supervisory personnel adequate to provide comprehensive supervision. ☐Y ☐N ☐NA	
Is there a technical manager or person working in that capacity? ☐Y ☐N ☐NA	
Is there a quality manager responsible for the quality system and its implementation? Does the quality manager have direct access to the highest level of management where decisions of lab policy are made? ☐Y ☐N ☐NA	
Where appropriate, does the lab participate in interlaboratory proficiency testing programs? Attach a copy of the last two PE studies performed. ☐Y ☐N ☐NA	

Requirements	Auditor Comments
2.0 Quality System	
Does the lab maintain a quality system appropriate to the type, range, and volume of testing activities it undertakes? ☐Y ☐N ☐NA	
Are all elements of the quality system documented? ☐Y ☐N ☐NA	
Is the quality documentation available for use by all laboratory personnel? ☐Y ☐N ☐NA	
Do the staff members have a basic understanding of the QA manual? ☐Y ☐N ☐NA	
Is the QA manual current and the responsibility of the quality manager? ☐Y ☐N ☐NA	
Does the lab conduct internal audits to verify that its operations continue to comply with the requirements of the quality system? ☐Y ☐N ☐NA	
Where the audit findings cast doubt on the correctness or validity of the lab test results or procedures, does the lab immediately take corrective action? ☐Y ☐N ☐NA	
Are audit findings and the corrective actions taken documented? ☐Y ☐N ☐NA	
Does the quality manager ensure that these actions are taken within the agreed time-frame? ☐Y ☐N ☐NA	
When a nonconformance is found, how is it handled? Document.	

Requirements	Auditor Comments
Is the quality system reviewed annually to ensure that it remains effective? How are changes documented? Document.	
Are standard curves prepared to cover the expected concentration ranges of RASI samples? ☐Y ☐N ☐NA	
Are standard curves prepared as specified by the method? ☐Y ☐N ☐NA	
Are new curves prepared whenever an out-of-control condition occurs and after new reagents are prepared? ☐Y ☐N ☐NA	
Are control charting data or control limits generated for routine analyses? ☐Y ☐N ☐NA	
Is there a corrective action plan which is implemented when analytical results fail to meet QC criteria? ☐Y ☐N ☐NA	
Are records maintained which indicate what corrective actions took place whenever 2.6.1 occurs? ☐Y ☐N ☐NA	
Do supervisors review a percentage of data, calculations, and QC results? ☐Y ☐N ☐NA	
Are deviations or deficiencies in QC reported to the quality manager? How are these issues documented to client? ☐Y ☐N ☐NA Document.	
Are QC data (batch) retrievable for all analytical results? ☐Y ☐N ☐NA	

Requirements	Auditor Comments
Are MDLs determined and documented for analyses required by RASI? ☐Y ☐N ☐NA	
Are solvents routinely analyzed to check for purity? ☐Y ☐N ☐NA	
Do all SOPs contain a QC section describing blanks, when to perform duplicate analyses and spikes, what surrogates and internal standards are used, frequency of LCSs, and control limits. ☐Y ☐N ☐NA	
3.0 **Metals Analyses**	
Does the lab analyze a LCS with each batch of samples? ☐Y ☐N ☐NA	
What is the frequency of performing a LCS, MS/MSD? ☐Y ☐N ☐NA	
Are the metals acceptance criteria %R = 80-120 and %RPD < 20? ☐Y ☐N ☐NA	
Are calibrations verified with and independently prepared check standard? ☐Y ☐N ☐NA	
What is the frequency of performing a continuing calibration and are the acceptance criteria %R = 90-110? ☐Y ☐N ☐NA	
Is the highest standard reanalyzed after calibration and are the accept criteria +/- 5% of true? ☐Y ☐N ☐NA	

Requirements	Auditor Comments
How frequently are the interferent check samples analyzed? Are they analyzed at the beginning and end of each run or twice during and 8 hour shift, whichever is more frequent? Are results +/- 20% of true? ☐Y ☐N ☐NA	
Are post-spikes performed for 7000 methods when matrix interferences are present? What recovery ranges are used? 85-115%R? ☐Y ☐N ☐NA	
Are MSAs performed when post-spike fails? ☐Y ☐N ☐NA	
Are all working standards verified against primary (reference) standards? ☐Y ☐N ☐NA	
Are standards NIST traceable? ☐Y ☐N ☐NA	
Are preparation of standards/reagents documented thoroughly? Are they traceable back to vendor and lot number? ☐Y ☐N ☐NA	
4.0 Metals Preparation	
Is glassware acid washed prior to preparation? If so, for how long (time)? ☐Y ☐N ☐NA	
Is there sufficient room and equipment available to handle a normal sample volume? Are hot plates the sole means of metals preparation or does the lab have a microwave? ☐Y ☐N ☐NA	
What grade of acid is being used for preparation?	
What grade of water is being used?	
Are standards/reagents preparation information documented? ☐Y ☐N ☐NA	

Requirements	Auditor Comments
Is Class A glassware being used? Are pipettes being used? ☐Y ☐N ☐NA	
If pipettes are being used, at what frequency and how are pipettes check for calibration? ☐Y ☐N ☐NA	
Are reagent blanks and LCS prepared with each batch? ☐Y ☐N ☐NA	
What type of system is being used to track the frequency of MS/MSDs? Document.	
Are acids stored in a corrosive-safe container and are acids transported in oversized safety containers? ☐Y ☐N ☐NA	
Are balances calibrated on a daily basis with Class S weights? Is there sufficient documentation showing daily calibration checks? ☐Y ☐N ☐NA	
Are analytical balances capable of weighing to 0.1 mg? ☐Y ☐N ☐NA	
Are balances calibrated annually? ☐Y ☐N ☐NA	
Is water conductivity measured on a daily basis and is there appropriate documentation? ☐Y ☐N ☐NA	
Are reagents/standards labeled with concentrations, date of preparation or date opened, expiration date and identity of person preparing the reagent? ☐Y ☐N ☐NA	
Are standards and reagents current not expired? ☐Y ☐N ☐NA	

Requirements	Auditor Comments
Do preparation sheets contain sufficient information to be able to track lots of acids, personnel backward if questions are raised? ☐Y ☐N ☐NA	
Does the lab track percent of re-preparations? ☐Y ☐N ☐NA	
5.0 Organic Analyses	
What are the frequency MS/MSDs performed? What acceptance criteria are used for each method? Document per method.	
What is the frequency of blanks?	
What are the acceptance criteria for acceptance of a curve and what levels are analyzed?	
How frequently are continuing calibration checks run? What are the acceptance criteria?	
What surrogate limits are used per method? Are they laboratory established or method defined limits? ☐Y ☐N ☐NA	
Are surrogate control limits generated using laboratory data? ☐Y ☐N ☐NA	
In what methods are internal standards used? What are the criteria used to prove acceptance.	

Requirements	Auditor Comments
For what methods is a second column confirmation being used in GC?	
How frequently are tunes performed?	
6.0 Organic Preparation	
Is sufficient information contained on the preparation sheet in order to track back any problems to personnel, solvent lot, Florisil lot, and matrix spike? ☐Y ☐N ☐NA	
Are both the date extracted and a place for the check placed on the preparation sheet? ☐Y ☐N ☐NA	
Are there sufficient continuous extractors and hot plates available for a normal work load? How many to be exact?	
Is there adequate ventilation present and are there separate air exchanges between the preparation group and instrument groups? ☐Y ☐N ☐NA	
Does the lab contain a GPC? ☐Y ☐N ☐NA	
Is the GPC checked with 2,4,5-trichlorophenol and a pesticide solution prior to use or every 7 days? ☐Y ☐N ☐NA	
Are there adequate personnel? ☐Y ☐N ☐NA	
Are standards stored at 4 C or colder? ☐Y ☐N ☐NA	
Are refrigerator logs current and complete? ☐Y ☐N ☐NA	

Requirements	Auditor Comments
Are surrogate and matrix spikes prepared by OP? If so, is there documentation of verifications by GC or GC/MS? ☐Y ☐N ☐NA	
Are reagent blanks and LCSs prepared with each batch? ☐Y ☐N ☐NA	
Are balances calibrated on a daily basis with Class S weights? Is there sufficient documentation showing daily calibration checks? ☐Y ☐N ☐NA	
Are analytical balances capable of weighing to 0.1 mg? ☐Y ☐N ☐NA	
Are balances calibrated annually? ☐Y ☐N ☐NA	
Are solvent containers and standards labeled with date opened, person who opened or prepared it, and concentration, expiration date, if applicable? ☐Y ☐N ☐NA	
Are solvents, standards stored properly? ☐Y ☐N ☐NA	

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Writing Standard Operating Procedures				
Prepared by:	Approved by:	Date: 8/11/94	QA Approval:	

1.0 PURPOSE

The purpose of this procedure is to assist in the development of standard operating procedures (SOPs) both for instrumental and non-instrumental areas. It is the intent of this procedure to provide guidance and show examples of items to include in SOPs so that the analyst will have the most available information at his or her disposal.

2.0 APPLICATION

This procedure is to be used as a tool and explanation for laboratory staff to document procedures in a uniform and complete fashion. All methods and practices used in the Ross Analytical Services, Inc., facility shall be documented in the form an SOP consisting of items listed herein.

3.0 REFERENCES

U.S.E.P.A. Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM03.0, Exhibit E.

4.0 ASSOCIATED SOPs

None.

5.0 PROCEDURE

5.1 Header Documentation

5.1.1 SOP numbers are generated by the Manager of Quality Assurance. These are unique numbers which are assigned sequentially according to department. Below is the basis of the numbering system which is used to generate an SOP number.

Department Code	SOP No.	Revision No.
XX	001	00

For most groups, the first two letters represent the department in which the SOP will be used. For the instrumentation laboratories, the department code also contains information which indicates what type of analysis will be performed. For example, GP indicates that the SOP belongs to the Gas Chromatography Laboratory and that the type of analysis to be performed is for pesticides and/or PCBs. Other departmental codes are listed below:

QC Quality Control
AD Administrative or general practices which are not specific to any one department
CW Classical Wet chemistry analyses

GA	GC/Air analyses
GP	GC/Pesticides or PCBs
GV	GC/Volatiles
GX	GC/Extractables
II	Inorganic Instrument analyses
IP	Inorganic Preparation for metals analyses
MA	GC/MS Air work
MT	Metals by ICP or AA
MV	GC/MS Volatile analyses
MX	GC/MS Extractable analyses
OP	Organic Preparation for GC and GC/MS extractable samples
SC	Sample Control

SOP No. is a three-digit number which starts at 001 and ends at 999. Basically SOP No. indicates what SOP is being described in department XX.

The initial Revision No. is listed as 00 for all SOPs which have just been started. For each subsequent revision, the Revision No. will be incremented by 01.

5.1.2 Additional header information which must be included on the front page of each SOP is as follows:

1. Name of company - Ross Analytical Services, Inc.

2. Standard Operating Procedure.

3. Title of SOP.

4. Prepared by: Approved by: Date: QA Approval:

5. Page ____ of ____.

5.1.3 Header documentation for the top corner of each page of the subsequent SOP pages.

SOP No.:
Date Initiated:
Page ___ of
Date Revised:

5.1.4 Obtain a SOP No. and header template from the Manager of Quality Assurance before starting to write.

5.2 Purpose

Define instrumental or preparatory analysis to be performed. This is a general statement about the basis for the existence of this SOP. If instrumental, detail the instrument analysis and type of instrument including specific detectors, if applicable, to be used in performing the method. Examples of both instrumental and preparatory SOPs are as follows:

"The purpose of this procedure is to describe the analysis of organophosphorus pesticides by Gas Chromatography using a thermionic specific detector (TSD)."

"This procedure describes the method used to clean up extracts using a Florisil column prior to analysis b GC or GC/MS."

5.3 Application

Describe what specific method is being employed and the document where it originated, such as SW-846, Method 8080.

Document what the analysis is trying to accomplish, such as, "this procedure is to be used for the quantitative analysis of chlorinated pesticides and PCBs".

Note the type(s) of matrix(ces) for which this procedure is to be used.

Reference the section in the SOP where information regarding interferences are located.

Denote the section in the SOP (see Section 5.8.8 of this SOP) which contains tables of targets and Estimated Quantitation Limits (EQLs) or Reporting Limits (RLs).

5.4 References

The intent of this section is to both document all information from which this SOP is derived and provide any new employee with various sources of information to become more informed about the procedure at hand.

Note all references used to derive this method and others which will impact on the application of this SOP. For instance, include instrumentation manuals, specific helpful documents from the EPA, informative articles about theoretical occurrences which may be taking place in a given system, practical laboratory application articles, and all EPA methods which may be pertinent to the SOP, accounting for sample preparatory methods used for the samples to be analyzed in a particular instrumental SOP.

5.5 Associated SOPs

Document all SOPs which have bearing on the outcome of the method herein. This means, for a metals or chromatography instrumentation SOP, list all SOPs for preparation of the samples for this method.

5.6 Equipment

List all equipment which is pertinent to the analysis to be performed. This list should be detailed enough that a new employee will be able to gather the items listed and perform the analysis.

5.7 Standards and Solutions

5.7.1 Solutions

Describe in detail how a solution is prepared from a chemical and at what concentration the solution is prepared. State how the solution is to be stored, e.g., this solution must be stored in a plastic container at room temperature. State the length of time the solution is good for use.

Note how and in what logbook the solution preparation must be documented.

Name the vendors which are used as sources of chemicals for the method being described. Detail the grade which is purchased, such as "reagent grade".

Transfer all Certificate of Analysis sheets to the office of the Manager of Quality Assurance.

5.7.2 Stock Standards

State where standards from vendors are purchased and at what concentration level they are purchased. A requirement is that all standards purchased must meet or exceed U.S.E.P.A. QC criteria.

Describe in detail how a stock solution is prepared from a neat compound and at what concentrations the solutions are prepared. Note the solvent in which to dilute the neat compound. State how the standards are to be stored, e.g., standards must be stored in amber vials and refrigerated at 4°C. Likewise, state the length of time the standards are good for use.

Note how and in what logbook the standard preparation must be documented.

Describe the process for standard verification and in what logbook this information is stored.

Note in this section that all Certificate of Analysis sheets accompanying standards purchased must be sent to the office of the Manager of Quality Assurance.

5.7.3 Calibration Standards

Describe the levels of standards which are to be prepared, solvent in which to prepare them, and the corresponding concentrations.

Detail the procedure for preparing the standards and note what solvent or solute in which they are to be prepared.

Provide a time period for replacement.

5.7.4 Surrogates

List all of the surrogates used in a given analysis and describe their purposes. For example, phenol-d5 is used to monitor acid compounds in semivolatile analysis.

If needed for clarity, also include a list of the surrogate compounds in tabular form (see Section 5.8.8).

Preparation instructions including concentrations, solvents in which to prepare surrogate mixes, and the concentration and volume to be spiked into a given "sample", should be clearly defined.

Note all of the "samples" into which surrogates are to be added, e.g., blanks, check standards, matrix spike/matrix spike duplicate samples, and in what laboratory spiking occurs.

5.7.5 Internal Standards

List and tabulate for clarity, as above, the internal standards to be used in the analysis and describe the purpose of them and how its behavior may indicate matrix problems.

Note what targets are quantitated from which internal standards.

Describe preparation instructions to be used for the internal standards including the solvents used to prepare them and the concentrations in which they are to be prepared and spiked into "samples". Also, describe what "samples" are to be spiked and where and at what point the spiking occurs in the preparation sequence.

5.7.6 Laboratory Control Samples (LCSs), Matrix Spikes (MSs), Matrix Spike Duplicates (MSDs)

Describe how and at what concentration levels spikes are to be prepared. Note type of solvent in which the spikes are to be prepared and the volume of spike to be used for LCSs, MSs, MSDs.

List what the frequency of the LCS, MS, and MSD are to be prepared.

5.8 Procedure - Below are topics which should be covered in standard operating procedures developed for Ross Analytical Services, Inc. Note that *not all topics* apply to all SOPs, but if the area is applicable, the underlying information must be included.

5.8.1 Instrumental Setup and Operating Conditions

This section must be very detailed and specific. Examples of some operating conditions and instrument setups for GC and GFAA analyses are listed below.

GC example

Detector type
Detector range
Column name and type
Injector temperature
Detector temperature
Gas flow information
Temperature program including initial temperature, ramp conditions, and hold times

GFAA example

Program number to run and parameters therein such as burn conditions, temperature ramp, gas flows, and wavelength
Describe any mechanical setups which are necessary

5.8.2 Calibration Procedure

5.8.2.1 Initial Calibration

The intent of this section is to provide the analyst sufficient information to set up an initial calibration run, knowing what volume of standards to inject into an instrument, understanding how to calculate a given response factor (GC), while enabling an analyst to interpret what a good % RSD is and how it applies to the analysis at hand.

Likewise, all acceptance criteria of the initial calibration must be documented along with what corrective actions to take if the criteria are not met.

5.8.2.2 Working Calibration Curve Verification

Documentation must include the following:

Frequency of verification required such as "a continuing check or continuing calibration verification standard (CCV) must be run after every ten samples on the instrument",

The concentration level of standard used for verification,

The process of how and what to compare the verification standard with, such as, "the response is checked by comparing the response factor of the continuing check (Area/concentration) to the mid-level standard response from the initial calibration",

What calculation is used to perform a comparison of the continuing check and the initial calibration (percent difference),

How to interpret the result of the percent difference and deciding what corrective actions are needed if the resulting difference exceeds established acceptance criteria.

In this section, it is recommended that a troubleshooting section be included to specifically describe what to do when a verification check does not pass acceptance criteria. This section may include maintenance items such as cleaning the torch for the ICP, changing tubing, column clipping for GCs, replacement of column inserts for GCs, or even as basic as replacing the GC septa.

5.8.2.3 Retention Time Windows

Describe the purpose of establishing retention time windows, how to establish them, how retention times are updated, and what to do if a continuing check falls outside the daily retention time windows.

5.8.3 Analysis

List the sequence of analyses which must take place prior to analysis of real-world samples, such as, run a curve, follow with an initial calibration verification standard, initial calibration blank, interferent check standards A and AB, samples, continuing calibration verifications, continuing calibration blanks, etc.

Describe when dilutions or serial dilutions must be performed. Indicate what criteria are used to determine when a dilution must be made.

Include what data are initially entered into the instrument to generate the desired results, e.g., sample weight, final volume, and percent moisture.

5.8.4 Data Interpretation and Calculations

Document what steps an analyst should take in order to interpret the data generated and place this in a logical and sequential format. If practical, place this in a decision tree, if ... then - type structure, e.g., following the verification that the continuing calibration standards and ending calibration standards have passed the acceptance criteria, calculate the LCSs to see if the analytical batch is ok. Detail every step which must take place in this process in order for the data to be deemed acceptable.

Note all formulas and calculations which must take place in order for the analytes or elements tc be hand calculated to their final form. Include all necessary formulas even if they are embedded in software programs so that the analyst will be able to verify calculations performed by the software.

Describe corrective actions specific enough that an analyst will be able to take action without supervisory assistance if a method requirement fails e.g., if a CCV or ICSA fails to meet acceptance criteria, rerun the samples.

5.8.5 Quality Control

This section must include all holding times specified by the method.

List the acceptance criteria to be used to accept internal standards. For example, if the acceptance criteria are -50 to +100 percent of the last standard check calibration, document this in the SOP. If these conditions are not met, discussion must be present regarding the corrective actions which must occur.

Likewise, discussion must be present to provide guidance when situations arise where a surrogate or multiple surrogates fall outside acceptance criteria.

Include water and soil method surrogate recoveries for those methods which indicate that laboratory generated control limits must fall within the limits specified in the particular method.

The frequency of all LCSs, MSs, MSDs should be listed herein. List their acceptance criteria and what actions to take if the criteria are not met.

5.8.6 Interferences

This section should document all known interferences present for a given matrix or compound-type. Information for this section may be present in the methods or can be based upon knowledge obtained from work with various matrices, instrumentation, or from published papers.

Note any cleanup methods which may be used to obtain better data.

5.8.7 Troubleshooting

This section is to be used to provide if ... then scenarios of problem situations which happen with some frequency and a corresponding troubleshooting exercise to perform following occurrence. It should include problems which do not result in a QC failure but nevertheless affect analytical performance or data quality. An example is that if there is a decline in response of the standards over time, what action should an analyst take in order to correct this situation before it becomes a QC problem?

This section should become an ever expanding portion of the SOP. As analysts become more familiar and knowledgeable about the SOP, they should expand on this list and attach it as a SOP modification to be included in future revisions.

5.8.8 Tables

List target analytes, EQLs or RLs, and any other groups of items which are not covered in the text sections above.

5.9 SOP Review Process and SOP Distribution

5.9.1 All SOPs must be reviewed by the Group Leader of the department from which the SOP originated, the Manager of Quality Assurance, and the Technical and Quality Director. It is highly recommended that a bench level analyst review all SOPs in order to obtain an idea of how user friendly the procedure is as written.

5.9.2 Group Leaders are responsible for maintaining current copies of applicable procedures in their respective areas. Group Leaders must review all Ross Analytical Services, Inc., SOPs annually.

Since changes in procedures occur more frequently than once a year, a SOP Modification Form must be filled out in the interim. The intent of this form is to provide a means of making minor changes to a SOP. If a major change is required for a SOP, a full fledged revision must take place. Whether a change is considered a modification or a revision is at the discretion of the Manager of Quality Assurance. This form is appended (see Figure 1) to this SOP and copies are available from the Manager of Quality Assurance.

5.9.3 Group Leaders must return all copies of SOPs which have been superseded by newer revisions upon delivery of a copy of the new revision to prevent inadvertent use. The Manager of Quality Assurance will assign and document the transfer of SOP copies to all Group Leaders and others who require them. No copies will be made by the individual group staffs. If staff require a SOP, they must see the Manager of Quality Assurance.

5.9.4 The original and all subsequent original revisions of SOPs shall be maintained in a file in the possession of the Manager of Quality Assurance.

Figure 1
SOP Modification Form

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: _____

SOP Number: _____

Date change implemented : _____

Change initiated by: _____

Operational approval: _____

QA Approval: _____

==========

Section to be changed: _____

Change(s): _____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: AD-002-02
Initial Date: 2/1/91
Page 1 of 1
Date Revised: 12/22/94

Title: Class S Weights			
Prepared by:	Approved by:	Date: 1/10/95	QA Approval:

1.0 PURPOSE

To describe the procedure for proper use of Class S weights in the calibration of balances.

2.0 SCOPE

Class S weights are used to calibrate balances before each use. Proper use and handling are important in ensuring proper calibration.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Class S weights must be certified every three years. The last certification date is written on the box. A member of the QA Department will arrange certification at one of the following agencies:

Ohio Department of Agriculture
Division of Weights and Measures
8995 East Main Street
Reynoldsburg, Ohio 43068
(614) 866-6361

State of Michigan
Department of Agriculture
P.O. Box 30017
Lansing, Michigan 48909

Certification documentation is kept in the QA Coordinator's office.

5.2 Always handle Class S weights with clean plastic forceps. Avoid transferring weights any distance with the forceps. Keep box close to the balance. If weights are needed in another room, transfer the entire box.

5.3 Return weight to box immediately after use. Keep box closed to keep weights clean.

ROSS

Ross Analytical Services, Inc.

Standard Operating Procedure

SOP NO: AD-003-01
Initial Date 2/1/91
Page 1 of 3
Date Revised: 1/16/92

Title:

Use of Balances

Prepared by:	Approved by:	Date:	QC Approval:
		3/2/92	

1.0 PURPOSE

To describe the procedure for the proper use of laboratory balances.

2.0 APPLICATION

This procedure is applicable to analytical and top-loading balances.

3.0 REFERENCES

Mettler Operating Instructions

4.0 ASSOCIATED SOPs

4.1 SOP AD-002 (current version), Class S Weights

4.2 SOP AD-004 (current version), Calibration of Balances

5.0 PROCEDURE

5.1 Calibration - see SOP No. AD-004 (current version), Calibration of Balances

5.2 Analytical Balances

5.2.1 Check if the balance is level by observing the bubble level seen on the top of the balance. If the bubble is not in the middle of the circle, adjust the leveling screws on the bottom of the balance.

5.2.2 The weighing pan must be clean and dry.

5.2.3 Weighing without a container: Close the balance doors and set the balance to zero. Open the doors and carefully place the object to be weighed in the center of the pan. Close the doors, adjust balance to the proper weight range and wait for a stable reading. Record the weight to the nearest 0.0001 g.

NOTES:
1. The object being weighed must be clean , dry, at room temperature, and must not touch the sides of the balance doors.
2. Do not handle objects with bare hands.

5.2.4 Weighing with a container: Close the balance doors and set the balance to zero. Open the doors and carefully place the container in the center of the pan. Close the doors and re-zero the balance. Open the doors and carefully add the material to be weighed into the container. When the desired weight is reached, close the doors and wait for a stable reading. Record the weight to the nearest 0.0001 g.

NOTE: If any material spills onto the pan, wipe before a final reading is taken.

5.2.5 When finished, clean the balance and surrounding area, close the doors and reset balance to zero.

5.3 Top-Loading Balances

5.3.1 The balance must be on a level, stable surface.

5.3.2 The pan must be clean and dry.

5.3.3 Place the container in the center of the pan and zero the balance.

5.3.4 Place the desired amount of material to be weighed into the container. Let the balance stabilize and record the weight.

5.3.5 Repeat the zero step for each container used.

5.3.6 When finished, clean the balance and surrounding area and leave the balance set at zero.

Title: Calibration of Balances.					
Prepared by:	Approved by:	Date:	1/22/97	QA Approval:	

1.0 PURPOSE

To describe the procedure for the calibration of balances.

2.0 APPLICATION

This procedure is applicable to both the daily, quarterly and annual calibrations of all balances.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

4.1 AD-002, Class S Weights

4.2 AD-003, Use of Balances

5.0 PROCEDURE

5.1 Daily Calibration

5.1.1 All balances must be calibrated with a series of Class S weights before the first use of each day. Record necessary information on the appropriate balance weight record log located near each balance.

5.1.2 Check the weight criteria limits on the form (See Section 6.0, Figures).

5.1.2.1 If the balance meets the acceptance criteria, proceed with the weighing.

5.1.2.2 If the balance does not meet the criteria, recheck the level of the balance, rezero and weigh the Class S weights again.

NOTE: **Do not use balance until the problem is corrected**.

If necessary, recalibrate the balance with its internal function.

If the balance fails to meet the acceptance criteria again, notify the QA Department.

5.2 Annual calibration

All balances are calibrated by an external agency annually. Certificates are maintained by the QA Department.

6.0 FIGURES

Figure 1
Example Balance Sheet

ROSS ANALYTICAL SERVICES

Analytical Balance Check Data Sheet
Balance must be checked before the first use each day.

Balance_____

	0.1 g	1 g	5 g	10 g	50 g	100 g	
Data	± 0.0005	± 0.001	± 0.005	± 0.001	± 0.001	± 0.001	Checker

Title: Calibration of Thermometers			
Prepared by:	Approved by:	Date: 1/22/97	QA Approval:

1.0 PURPOSE

To describe the procedure for the calibration of laboratory thermometers.

2.0 APPLICATION

All working thermometers must be calibrated annually with an NBS-traceable thermometer to ensure accurate temperature measurements.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Calibration of Thermometers

5.1.1 Whenever a new thermometer is purchased from a vendor, notify the QA Department of the purchase so the serial number is placed on file for the annual check against a reference thermometer.

5.1.2 Calibrate each working thermometer annually using an NBS reference thermometers. The NBS reference thermometers are kept in the QA Department.

5.1.3 For thermometers used for laboratory analyses: Calibrate at 0°C, 21°C (room temperature), and 100°C.

<u>0°C Calibration (Ice Point)</u> - Fill a beaker with ice cubes and add deionized water. Immerse the working thermometer and the reference thermometer at the same level for 10 minutes and read.

<u>21°C Calibration</u> - Allow working and reference thermometers to remain together in the same room for 24 hours. Put the bulbs together for 10 minutes and read.

<u>100°C Calibration</u> - Bring a beaker of deionized water to a boil on a plate. Immerse the working reference thermometer at the same level for 10 minutes and read.

The working and reference thermometers must agree within $\pm$ 1°C at all levels. If they do not, replace thermometers.

5.1.5 Complete a Thermometer Calibration form (Figure 1) for each thermometer calibration. Send to the QA Department where it is filed.

Figure 1

THERMOMETER CALIBRATION

Date_____

Thermometer ID_____ Date last calibrated_____

Date reference thermometer last calibrated_____

Method of Calibration_____

_____

_____

_____

_____

_____

_____

_____

Acceptance Criteria ± 1°C

Calibration Range	Reading (reference)	Reading (working)
_____	_____	_____
_____	_____	_____
_____	_____	_____

Does thermometer pass calibration?

Signed _____

When completed, please send to the QA Department.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Laboratory Waste Disposal			
Prepared by: CEO	Approved by:	Date: 5/15/96	QA Approval:

1.0 PURPOSE

To provide a safe, environmentally sound approach for handling, treatment and disposal of common laboratory waste at Ross Analytical Services, Inc. (RASI).

2.0 SCOPE AND APPLICATION

The amount of hazardous waste generated by RASI is less than 1000 Kg per month. RASI is considered a small quantity generator and is exempt from some of the U.S. and Ohio EPA Regulations impacting hazardous waste generators.

This SOP deals only with waste designated in the four groups listed below. To handle other types of waste, contact the Laboratory Waste Coordinator for guidance. Some laboratory waste is hazardous and requires special handling and disposal.

Laboratory wastes are categorized into the following four groups:

1. Paper, cardboard, plastic and other non-hazardous, non-toxic materials - This material consists primarily of used packaging material and general office waste.

2. Empty reagent and sample bottles

3. Expired reagents and standard solutions

4. Analyzed raw samples, sample residues, sample extracts, and sample digestates

This SOP has been prepared to provide direction for RASI employees for the disposal of all laboratory wastes in a safe and environmentally sound manner.

3.0 REFERENCES

DOT Hazardous Material Regulation, 49 CFR

RCRA Hazard Characterization and Manifest, 40 CFR

TSCA PCB Characterization and Manifest, 40 CFR, Part 761

Northeast Ohio Regional Sewer District

4.0 RESPONSIBILITY FOR PROPER WASTE DISPOSAL

4.1 Corporate Commitment

RASI is committed to the disposal of all waste in an environmentally safe manner. In general, non-hazardous waste is placed in a dumpster and removed by a contracted waste removal service. This waste is transported to a sanitary landfill for disposal. Special care is taken at RASI to insure that only non-hazardous waste is disposed of in this manner. Hazardous waste and non-hazardous waste generated as a result of laboratory activities is incinerated, whenever possible. When incineration is not possible or appropriate, these wastes will be treated or landfilled at an EPA approved site. Sewer disposal of waste and laboratory preparations is not permitted.

4.2 Management Responsibility

A Laboratory Waste Coordinator has been appointed at RASI. This individual is consulted whenever questions pertaining to laboratory waste disposal arise, whether or not covered by this document.

4.3 Staff Responsibility

All laboratory employees are responsible for the disposal of laboratory wastes in accordance with the guidelines contained within this document.

5.0 PROCEDURE

5.1 Paper, Cardboard, Plastics (Trash)

Within each laboratory and office area, plastic trash containers have been placed for the disposal of non-hazardous non-contact wastes. These containers are emptied by the maintenance staff into the trash dumpster (blue). Cardboard is segregated into a separate dumpster (green) for off-site recycling. To conserve room in the dumpster, all empty cardboard boxes are broken down and flattened prior to placement into the dumpster. To avoid accidental cuts, DO NOT place broken glass into these trash containers; instead, use the metal and plastic containers which are designated for broken glass.

5.2 Empty Reagent and Solvent Bottles

5.2.1 Containers from Acutely Toxic, Carcinogenic and Special Hazard Materials

Certain empty containers, due to their original contents, require special care prior to being determined "empty". The empty containers of acutely toxic reagents ("P" listed materials), carcinogenic materials such as PCBs, and special hazard materials are triple rinsed prior to disposal. Triple rinsed containers that no longer present a hazard and are no longer regulated are disposed of as non-hazardous laboratory debris which is incinerated.

5.2.2 Glassware and Containers

Empty glassware and containers are crushed and placed in the trash dumpster for disposal. Empty soil sample containers must never be placed in the dumpster. To ensure compliance with the Department of Agriculture, Plant Protection Quarantine Program, all empty soil containers are disposed of in the laboratory lab debris waste stream which is incinerated and meets the requirements of the program.

5.2.3 Plastic Containers

Empty plastic containers are shredded and placed in the dumpster for disposal. Empty soil sample containers are never be placed in the dumpster. To ensure compliance with the Department of Agriculture, Plant Protection Quarantine Program, all empty soil containers are disposed of in the laboratory debris waste stream which is incinerated and meets the requirements of the program.

5.3 Reagents and Standard Solutions

These laboratory wastes are broken into four general classes, organic and inorganic.

5.3.1 Organic Reagents and Standard Solutions

Expired or unneeded organic reagents and standards are segregated from current unexpired reagents when they are no longer usable or needed for their intended purpose. As a Pollution Prevention and Waste Minimization measure, these materials are put to other beneficial uses. In most cases, neat reference materials may be used for project-specific research and development while some expired solvents are used for cleaning glassware. Most expired standard materials are used by the Organic Preparation Lab to make the GPC deactivation solution as described in SOP # OP-052.

Note: Disposal of these materials should only be considered after it has been determined that there are no other uses for the material. Contact the SHE Officer for guidance in appropriate disposal.

5.3.2 Inorganic Reagent Solutions

Inorganic reagent solutions include acidic, basic, and neutral aqueous reagents and standards. The same Pollution Prevention and Waste Minimization philosophy as described in Section 5.3.1 is applied for these materials. Disposal of these materials is only considered after it has been determined that there are no other uses for the material. Contact the SHE Officer for guidance in appropriate disposal.

5.3.3 Waste Solvents

Solvent wastes are generated as a result of laboratory sample preparations. Solvents are segregated based on halogen content for economical disposal. Both of these waste streams are RCRA hazardous. The "Halogenated Solvent Waste" is primarily methylene chloride but contains samples residues and other solvents. The "Flammable Solvent Waste" is primarily methanol and acetone but contains sample residues and other solvents.

5.3.4 Waste Charcoal

Charcoal waste is generated from solvent cleanup procedures and the purging procedures for air sampling equipment. This waste is charcoal that is contaminated with solvents. It is disposed of as a methylene chloride waste stream.

5.4 Analyzed Samples, Sample Residues, Sample Extracts and Sample Digestates.

5.4.1 Aqueous Analyzed Samples

These include such samples as waters that have been extracted by continuous liquid/liquid or separatory funnel procedures, samples which have undergone oil & grease extraction, and some samples which have been digested for metals analysis. Once these samples have been extracted or

digested, they are segregated based on the solvent or reagents used in the extraction or digestion process. After segregation, the samples are disposed.

Samples extracted with methylene chloride or Freon are disposed of as RCRA non-hazardous (OP water) waste color coded in yellow.

Water samples extracted with such solvents as chloroform and ether are disposed of as hazardous waste because of hazardous waste characteristics (e.g. D001, D022). Samples which have been digested for metals are labeled as "Metals # 2" waste which is color coded red.

Acid solutions from metal digestates are segregated by whether (1) the digestate contained high concentrations of regulated metals or is highly colored, or (2) the digestate did not contain high concentrations of regulated metals or is clear. Digestates that contain high concentrations of regulated metals are disposed of as hazardous waste in the Metals # 2 waste stream (color code red); those which are low are disposed of in the Metals # 1 waste stream (color code blue).

Note: Acid waste is neutralized in the satellite accumulation container prior to placement in a drum for disposal.

Caustic waste generated as a result of the analysis of samples for cyanide content are disposed of as "Caustic Waste" which is color coded green. This waste stream includes both sample preparations and actual samples which are caustic. The caustic waste stream is considered hazardous waste; DO NOT neutralize this waste stream. The satellite accumulation containers of this waste stream are poured into poly-coated steal drums prior to disposal.

5.4.2 Sample Extracts

Organic sample extracts include extracts contained in auto sample vials including the portions retained by the Organic Preparation Department. These extracts are segregated according to whether they contain PCBs or not. Extracts that contain PCBs at 50 ppm or greater are disposed of in the PCB waste stream designated "PCB Lab Waste". This waste stream is both a RCRA and TSCA waste. Extracts that contain PCBs at concentrations less then 50 ppm are disposed of as RCRA wastes in a waste stream designated "Extract Vials". Both of these waste streams are placed into either paint cans or they are double bagged in polyethylene bags. These wastes are "containerized" in the department they originate and are taken to the waste accumulation area. These wastes are placed into an open-head drum until they are shipped to a RCRA or TSCA incinerator, as appropriate.

5.4.3 Sample Residues

Sample residues are the remaining portions of sample which are not used in for sample preparation or sample analyses. Samples are segregated based on sample type. All samples are inventoried prior to disposal. Analytical results for PCBs are reviewed and samples containing PCBs at 50 ppm or greater are disposed of as "PCB Lab Waste".

Samples that are clearly water samples (ground waters etc.) are disposed of in the waste stream designated "Water Samples". This is a non-hazardous waste.

Water or aqueous samples that are colored or have multiple layers are disposed of as hazardous waste in the Metals # 2 waste stream.

Soil and some solid samples such as filters are disposed of as "Soil/Solid Samples". These are not RCRA hazardous.

Waste samples such as paints are disposed of in one of two solvent waste streams generated by the lab. Both of the solvent waste streams are considered RCRA hazardous waste.

All client samples are either returned to the client or are incinerated.

5.4.4 Contact Non-Hazardous Waste Stream

Contact wastes include equipment and supplies used in the lab for the preparation and analysis of samples. This waste stream includes such items as latex gloves, weighing pans, wipes, bench coat, empty soil containers and pipets. Due to the contact nature of these items, this waste is collected in containers separate from "trash". This waste is placed into 5 gallon plastic pails which are emptied in to a Gaylord for disposal. This waste stream is designated "Lab Debris" and is considered non-hazardous. This waste is then incinerated.

6.0 STORAGE OF MATERIALS PENDING OFF-SITE REMOVAL

6.1 The Laboratory Waste Coordinator is advised of all wastes for off-site removal. Materials to be moved off-site are placed into a compatible containers which are in good condition. Temporary storage is established in a location which meets the storage needs for the identified hazard class. Flammable waste is stored in a flammable cabinet.

6.2 Weekly inspections of the storage area(s) are conducted by the Laboratory Waste Coordinator. Inspections include such items as container condition, hazard segregation, container markings, and secondary containment.

6.3 Weekly inspections of emergency equipment is conducted by the Laboratory Waste Coordinator. Inspections include items such as fire protection equipment, spill response equipment, and communication systems.

6.4 The Laboratory Waste Coordinator is responsible for maintaining inventory and arranging off-site removal in accordance with applicable Federal and State requirements. A small quantity generator can accumulate up to 1000 kg of hazardous waste each month provided removal occurs within 180 days or 270 days when shipping more than 200 miles (total quantities not to exceed 6000 kg). PCB waste must be removed within 270 days.

ROSS	SOP Standard Operating Procedure	SOP No: AD-009-00 Inital Date: 2/1/91 Page 1 of 5 Date Revised:

Title:

Training and Qualification in the Extraction Laboratory

Prepared by:	Approved by:	Date:	QC Approval:
Gary E Olson	C H Caldwell	6/11/91	

1.0 PURPOSE

To describe the training program followed within the extraction laboratory.

2.0 APPLICATION

This procedure applies to all new employees in the extraction laboratory and if used to provide guidelines to a training program and a method of documenting employee training.

3.0 REFERENCES

Ross Analytical Services, Inc., Laboratory Quality Assurance Manual

4.0 ASSOCIATED SOPs

None.

5.0 PROCEDURE

Individuals hired to work in the extraction laboratory shall be trained as follows:

5.1 First day - General Orientation

5.1.1 On the first day of work the new employee will fill out all required paper work for the personnel office. This usually takes one to two hours. During this time the employee will be issued an employee handbook and given the opportunity to briefly read the book and ask questions concerning benefits and related personnel policies.

5.1.2 After completing the required paper work, the new employee will be introduced to the extraction laboratory supervisor and taken on a "get acquainted" tour. During this tour the following objectives will be met:

1. Introduced to co-workers in the extraction lab.

2. Introduced to the Administrative Assistant who will order the lab coats.

3. Introduced to the various laboratory supervisors including GC/MS, GC, and Laboratory Director.

4. Familiarized with general lab layout and emergency exits and equipment.

5.1.3 After the tour the following materials will be issued to the employee:

1. Safety glasses.

2. Solvent gloves.

3. A laboratory coat.

4. A copy of the laboratory safety manual.

5. A copy of the pertinent extraction lab SOPs.

These materials are the property of Ross Analytical Services, Inc., and must be returned if the employee leaves the company.

5.1.4 After issuing these materials the employee will be ask to read the safety manual and complete any other required safety training. After reading the safety manual and completing the appropriate training, the employee will meet with the Laboratory Safety Officer to review the safety materials and to be given the opportunity to ask any safety related questions.

5.2 Training

5.2.1 New extraction lab employee will be assigned to work with a senior technician by the laboratory supervisor.

During this training period the senior technician will perform routine extractions and, where possible, volunteer to help other technicians by performing general methods.

When the senior technician feels that the new employee can perform a general method without supervision, the technician will notify the Group Leader. The Group Leader will initiate these steps:

1. Three samples of know concentration will be given to the new employee to extract by this newly learned method.

2. Extracts will be analyzed by the appropriate lab.

3. A successful extraction will vary from test to test and be judged by the laboratory supervisor.

4. If results are good, the new employee is judged competent in that method.

5. If the results are poor, the procedure which the employee followed will be reviewed for error. If no error can be found, the employee will repeat the extraction under supervision.

5.2. Training will continue until the new employee has learned the following extractions:

1. Method 3510 for BNAs and pest/PCBs.

2. method 3520 for BNAs and pest/PCBs.

3. Method 3540 for BNAs and pest/PCBs.

4. Method 3550 for BNAs and pest/PCBs.

5. Extraction of PCB's from oil.

After these methods have been learned, the employee will be allowed to work on his/her own and assigned to other technicians to learn new methods.

5.2.3 Progress will be reviewed at the end if each month until the probationary period is over.

5.2.4 Documentation of training will be forwarded to the Administrative Assistant for inclusion in the employee's training file.

5.3 Continuing Analyst Training

5.3.1 Extraction laboratory analysts will continue to train on new and non-routine methods by working with an analyst experienced at the method. When the analyst feels competent with the new procedure, the analyst will perform the extraction on the next set of samples received for that particular analysis. The sequence will include two blanks, a matrix spike and spike duplicate, along with 3 spiked blanks.

5.3.2 After completing the extraction, the Analyst Proficiency Documentation form (Figure 1) will be submitted to the Quality Control Coordinator. The results of the analysis will be evaluated by the group leader and Quality Control to determine proficiency.

5.3.3 If the results are within normal quality standards, the Analyst Proficiency Document will be maintained on file for all analysis the analyst has mastered.

FIGURE 1

ANALYST PROFICIENCY DOCUMENTATION

Analyst Name: _____

Department: _____

TEST	DATE PROVED	FILE REFERENCE	QA APPR.

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date
[illegible]	3/24/92

Terminated by	Date

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Storage and Handling of Compressed and Liquid Gases			
Prepared by:	Approved by:	Date: 1/23/97	QA Approval:

1.0 PURPOSE

To describe procedures for storage and handling of compressed and liquid gases.

2.0 APPLICATION

This SOP applies to all storage, transport, and use of compressed and liquid gases in the RASI Laboratory.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

Compressed and liquid gases are divided into four general categories for storage and handling: Flammable, Oxidant, Cryoliquid, and Other. Each category is treated separately below.

5.1 Purchasing

Gas grades and the cylinder and tank sizes for the gases normally used at RASI are listed in Table 1. Gas regulators have shafts and threads that prevent unsafe hookups (such as moving one from oxygen to hydrogen). The most nearly "universal" fitting is a 580; it is used for argon, nitrogen, and helium. Consult a gas vendor's literature to select the proper regulator.

5.2 General Storage and Handling

5.2.1 Compressed gas cylinders must be chained or otherwise secured to a rigid support when not being moved. Cylinders must be moved using the appropriate carts, with all safety restraints applied. Whenever possible, cylinders should, be chained individually. Never move a cylinder or tank with a regulator attached.

5.2.2 When a cylinder or tank is received, attach a yellow status tag to it (not to the cap). When the container is first used, tear off the "FULL" section. When you are finished with the tank, tear off the "IN SERVICE" section. This should be done whether the tank is truly empty or not. Store as described in 5.7, below.

5.2.3 Before attaching a regulator to a new tank, briefly and slightly open the valve to blow dust or foreign matter from the opening.

5.3 Flammable Gases

5.3.1 The flammable gases normally used at RASI are acetylene and hydrogen. Flammable gas cylinders must be kept in the cage at the outside rear of the building whether they are full or in service. The cage has separate sections for acetylene (the bay nearest the gate) and hydrogen (the bay farthest from the gate). If other flammable gases are acquired, see the Operations Manager for storage requirements.

5.3.2 Do not tip acetylene cylinders more than 30 degrees from the vertical. The gas is dissolved in acetone and acetone will get into the valve and be carried into transfer lines and instruments.

5.4 Oxidants

The oxidant gases normally used at RASI are oxygen. In-service cylinders may be secured in the chase between GC and GC/MS or at the point of use.

5.5 Cryoliquids

5.5.1 The cryoliquids normally used at RASI are nitrogen and argon. In-service tanks are stable on their wheels at the point of use and are not chained.

5.5.2 Special precautions are necessary for handling cryoliquids. Because the tanks continually vent, store and use them only in well-ventilated areas. When withdrawing liquid, wear insulated gloves and safety glasses or a full face shield.

5.6 Others

The "other" gases used at RASI are compressed air, carbon dioxide, nitrogen, helium, argon, and 5% methane in argon. Full cylinders may be stored in the chase between GC extractables and the Air lab or in the rack in the warehouse.

5.7 Empty cylinders and tanks

Cylinders and tanks whose useful life has passed are defined as empty even if they still retain some of their contents. Empty acetylene tanks are kept in the acetylene bay. All other empty tanks are placed in the cylinder storage rack located in the warehouse.

TABLE 1

GRADES AND CONTAINER SIZES OF
NORMALLY-USED GASES

Gas	Laboratory Using	Grade	Size
Acetylene	MT	99.6%	L5
Air	GC	Zero	J
Argon (liquid)	MT	99.99%	Bulk tank
Carbon dioxide	II	99.99%	J
Helium	GC, GC/MS, II	99.999%	J
Hydrogen	GC	99.999%	J
5% Methane in argon	GC		J
Nitrogen (gas)	MT, OP	99.998%	Bulk tank
	GC, GC/MS	99.998%	Bulk tank
Nitrogen (liquid)	OP		PG45 HP
	GC/MS		PG45 LP
Oxygen	II	Medical	J
	II	99.995%	J
	WC		J

ROSS	SOP Standard Operating Procedure	SOP No: AD-015-00 Inital Date: 2/1/91 Page 1 of 10 Date Revised:

Title:

Preventive Maintenance in the Inorganic Instrumentation Laboratory

Prepared by:	Approved by:	Date:	QC Approval:
Belinda M Winter	C H Caldwell	6/13/91	CHC

1.0 PURPOSE

To help keep an instrument running properly, routine daily, weekly, or monthly maintenance must be performed. Maintaining an effective routine preventive maintenance schedule requires proper documentation. The purpose of this SOP is to assist the analyst in keeping an effective, well-documented routine preventive maintenance log book.

2.0 APPLICATION

This SOP applies to the following instruments:

- Thermal Jarrell Ash Enviro 36 ICP
- Varian SpectrAA-20 Flame Atomic Spectrometer
- Varian SpectrAA-400Z graphite Furnance Atomic Spectrometer

- Dohrman/Rosemount DC-80 TOC Analyzer
- Dohrman/Rosemount DC-20 YOX Analyzer
- Lachat Automated Ion Analyzer

3.0 REFERENCES

3.1 Operation Manuals for the Thermal Jarrell Ash Enviro 36 ICP.

3.2 Operation Manuals for the Varian SpectrAA-20 Flame Atomic Absorption Spectrometer.

3.3 Operation Manuals for the Varian SpectrAA-400Z Graphite Furnace Spectrometer.

3.4 Operation Manuals for the Dohrman/Rosemount DC-80 TOC Analyzer.

3.5 Operation Manuals for the Dohrman/Rosemount DX-20A TOX Analyzer.

3.6 Operation Manuals for the Lachat Automated Ion Analyzer.

4.0 ASSOCIATED SOPs

None.

5.0 PROCEDURE

5.1 Preventive Maintenance Schedules

5.1.1 Thermal Jarrell Ash Enviro 36 ICP

Maintenance	Check Frequency	Replace Frequency	Clean Frequency
Pump tubing	Daily	Every 2 days	When needed
Capillary tubing	Daily	When needed	When needed
Quartz tubing	Daily	When needed	When needed
Injector tube	Daily	When needed	When needed
Nebulizer insert	When needed	When needed	When needed
RF coil	Daily	When needed	When needed
Quartz bonnet	Daily	When needed	When needed
Drain bottle	Daily		When needed
Purge gas	Daily	When needed	When needed
Purge windows	Daily	When needed	When needed
Vacuum pump oil	Daily	Every 6 mos.	
Adsorption trap		Every month	
Exhaust filter		Annually	
Nitrogen filter		Annually	

5.1.2 Varian SpectrAA-20

Maintenance	Check Frequency	Replace Frequency	Clean Frequency
Contact rings	Daily	Every 6 mos.	Daily
Graphite tube	Daily	When needed	
Quartz windows	Daily	When needed	When needed
Injector tubing	Daily	When needed	When needed

5.1.3 Varian SpectrAA-400Z

Maintenance	Check Frequency	Replace Frequency	Clean Frequency
Quartz tube assembly	Daily		When needed
Quartz windows	Daily	When needed	When needed

5.1.4 Dohrman/Rosemount DC-80 TOC Analyzer

Maintenance	Frequency
Replace septa	As needed
Replace glass wool in boat	Daily, when in use
Replenish reaction vessel reagent	Daily, when in use
Repack tine and copper scrubbers	As needed
Replace pump tubing	As needed
Repack lithium hydroxide scrubber	As needed
Repack cupric oxide furnace tube	As needed
Wipe down outside of instrument	As needed

5.1.5 Dohrman/Rosemount DX-20A TOX Analyzer

Maintenance	Frequency
Check inlet and outlet tubes for residue buildup	Daily, when in use
Clean or replace inlet and outlet tubes	As needed
Check all electrodes for residue buildup	Daily, when in use
Clean or replace cell electrodes	As needed
Replenish electrolyte solution	As needed
Replace glass columns and "O" rings	As needed
Wipe down outside of instrument	As needed

5.1.6 Lachat Quick Chem Automated Ion Analyzer

Maintenance	Frequency
Check helium tank	Daily, when in use
Change pump tubing	Monthly
Replace colorimeterlamp	Yearly
Replenish reagents	As needed
Wipe down outside of instruments	As needed

5.2 Documentation in Logbook

Each day, before an analyst begins to run an instrument, the routine preventive maintenance check sheet must be filled out (Form A).

5.2.1 Always begin by entering the date and the instrument checked.

5.2.2 After the daily checks are made the analyst enters his or her initials in the appropriate column in the check sheet.

5.2.3 In the event that an item on the check sheet is replaced or cleaned, the analyst's initials are entered in the appropriate column on the preventative maintenance check sheet or on the parts inventory check sheet (Form B).

5.2.4 The comments column of the check sheet is to be used for referencing detailed accounts of all uncustomary repairs, replacements and maintenance to the back side of the check sheet (Figure 2) by entering "on back" in the space provided. The analyst's initials and the date are to be entered immediately after any comments are entered on the back page.

5.2.5 A yearly calendar is kept in the log book to help remind the analyst of monthly, semi-annual, and annual maintenance.

5.2.6 Preventive maintenance documentation is reviewed monthly by the Group Leader.

Form A

MAINTENANCE LOG BOOK

Date	Instrument	Description of Maintenance	Initial	Parts Needed	Parts Ordered

Form B

Parts Inventory Log Book

Date	Instrument	Part and Part Number	Date Replaced	Date Taken	Initials

Figure 1

PREVENTIVE/ROUTINE MAINTENANCE

Date:

ICP	Check	Replace	Clean	Comments
Pump Tubing				
Capillary Tubing				
Quartz Tubing				
Injector Tube				
Nebulizer Insert				
RF Coil				
Quartz Bonnet				
Drain Bottle				
Purge Gas				
Purge Windows				
Vacuum Pump Oil				

SpectrAA-20	Check	Replace	Clean	Comments
Contact Rings				
Graphite Tube				
Quartz Windows				
Injector Tubing				

SpectrAA-400Z	Check	Replace	Clean	Comments
Quartz Tube Assembly				
Quartz Windows				

Figure 2

PREVENTIVE / ROUTINE MAINTENANCE
DOHRMANN DC-80 TOC ANALYZER

Daily Basis

Replace Glass Wool

Date	Initials	Date	Initials	Date	Initials

Daily Basis

Replenish Persulfate Sol'n

Date	Initials	Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Repack Tin & Copper Scrubbers		
Replace pump tubing		
Repack Lithium Hydroxide Scrubber		
Repack Cupric Oxide tube		

Checked By: _____ Date: _____

Figure 3

PREVENTIVE / ROUTINE MAINTENANCE
DOHRMANN DX-20 TOX ANALYZER

Daily Basis

Check Inlet & Outlet Tubes For Residue Buildup

Date	Initials	Date	Initials	Date	Initials

Daily Basis

Check Cell Electrodes For Residue Buildup & Replenish Electrolyte

Date	Initials	Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Clean Inlet & Outlet Tubes		
Replace Inlet & Outlet Tubes		
Clean Cell Electrodes		
Replace Cell Electrodes		

Checked By:_____ Date:_____

Figure 4

PREVENTIVE/ROUTINE MAINTENANCE
LACHAT QUICKCHEM AUTOMATED ION ANALYZER

Daily Basis

Check Sample Valve, Valve Lines, and Manifolds

Date	Initials	Date	Initials

Yearly Basis

	Date	Initials
Replace Colorimeter Lamp		

Checked By:_____ Date:_____

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date

Terminated by	Date

ROSS	SOP Standard Operating Procedure	SOP No: ~~AD 016 00~~ Initial Date: 2/1/91 Page 1 of 3 Date Revised:

Title:

Glassware Cleaning for Inorganic Analyses

Prepared by:	Approved by:	Date:	QC Approval:
Rey Bro	CHCaldwell	6/11/91	CHC

1.0 PURPOSE

To describe the procedure for cleaning glassware to be used for analyses in the inorganic laboratory.

2.0 APPLICATION

This procedure applies to all glassware and plastic ware in the inorganic lab.

3.0 REFERENCES

3.1 EPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories, 1979.

4.0 ASSOCIATED SOPs

None.

5.0 PROCEDURE

5.1 General Wet Chemistry Glassware

5.1.1 Immediately after use, glassware should be rinsed with tap water to remove residues.

5.1.2 Wash all glassware with a brush or scrubbing pad in a 1% solution of Liquid-Nox detergent.

5.1.3 Rinse well with tap water to remove all traces of detergent, then follow with several deionized water rinses.

5.1.4 Place glassware in the drying cabinet until dry.

5.2 Pipets

5.2.1 Immediately after use, rinse pipets with tap water and place into the basket inside the soaking cylinder filled with detergent solution.

5.2.2 When the basket is full, place it into the siphon washer and drop in an Alco-Tabs tablet.

5.2.3 Connect washer to tap water faucet and turn on hot water at a rate which will allow siphon to work correctly. Run for 30 minutes.

5.2.4 Turn off tap, switch connection to deionized water faucet and run with deionized water for an additional ten minutes.

5.2.5 Place pipets in the drying cabinet until dry.

5.3 Special Cleaning Requirements

5.3.1 Glassware with stubborn deposits may be soaked in Nochromix overnight and then rinsed well with tap water and deionized water.

5.3.2 Greasy residues may be removed with acetone or by soaking with warm NaOH (~1 g/50 mL H_2O) for 10 to 15 minutes.

5.3.3 All glassware used for metals analysis must be washed and rinsed as above, then immersed in a batch of 1:1 nitric acid for 30 minutes. Carry items from the bath to the nearest sink in a plastic tub, and rinse with copious amounts of deionized water.

NOTE: Always wear rubber apron, runner gloves, and eye protection when working with the acid bath.

5.3.4 Glassware to be used for phosphorus analysis should never be washed in phosphate-containing detergent. prior to first use, soak this glassware in hot 1:1 HCl for 30 minutes. Thereafter, rinsing well with hot tap water followed by deionized water is sufficient unless visible residues remain.

5.3.5 Glassware to be used for oil and grease analysis should be rinsed with Freon and allowed to dry under a hood, then placed in a desiccator until needed.

6.0 QUALITY CONTROL

6.1 Spot check randomly selected items periodically with pH indicating solution to assure no trace of acid or alkali remains after cleaning.

6.2 Monitor reagent blanks for all analyses performed. A high blank often indicates improper cleaning of the glassware.

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date

Terminated by	Date

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Chain-of-Custody and Sample Security			
Prepared by:	Approved by:	Date: 12/7/94	QA Approval:

1.0 PURPOSE

This SOP defines the procedure for maintaining chain-of-custody for samples and related sampling materials. It will ensure the traceability of samples should their data be questioned.

2.0 APPLICATION

This procedure applies to all samples processed through the laboratory.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

SOP No. SC-002, Sample Receipt, Log-in and Storage

5.0 PROCEDURE

5.1 Chain-of-Custody

5.1.1 The chain-of-custody on samples begins in the field. Chain-of-custody forms (Figure 1) may be obtained from Sample Control or clients may use their own forms.

5.1.2 The sample identification, time of sampling, date of sampling, sample descriptions, sampler's name, and company name. Note that if a chain-of-custody form is used from another company, information on these forms may vary from the RASI forms.

5.1.3 When the RASI lab receives the samples, the information is checked to be accurate. The chain-of-custody seals (Figure 2) ,if present, are inspected to ensure they are intact, legible, and dated correctly. If the sample arrives by a common carrier (e.g. Fed-Ex or UPS) the airbill is signed and the date and time of receipt is noted. The samples are inspected for information accuracy. The chain-of-custody forms are signed and dated by the recipient and the conditions of the chain-of-custody seals upon receipt is noted. Any problems are noted on the chain-of-custody form.

5.1.4 Once the samples are in Sample Control's custody, an internal chain-of-custody system is initiated. The samples are under the custody of the Sample Control Department. Anyone who removes a sample from the Sample Control area must log it out using the Internal Chain-of-Custody (Sample Control Record) (Figure 3). This is done by recording the RASI identification number, the test to be performed, the initials of the remover and the date and time of removal. Once a sample is signed out by remover, he/she is responsible for that sample until it is returned

to Sample Control. The only exception to this general sample documentation are the CLP and similar programs, for which each SDG has its own internal chain-of-custody.

5.1.5. Samples must be returned to Sample Control at the end of each day, noting the date and time of return, and storage location in the logbook. Any analyst who wishes to transfer a sample to another analyst must first return to Sample Control and make the changes in the log book.

5.1.6 The log books are reviewed daily and initialed by a sample custodian. If a person has failed to abide by the established protocol, a mark is made to highlight the problem. If an individual repeatedly fails to follow the established protocol, a memo is sent to his/her supervisor.

5.1.7 The chain-of-custody forms received from the field are transferred from Sample Control to the Reporting Department once logging is complete. They are filed there until the data report is completed. The chain-of-custody documents are then filed with the report.

5.1.8 If a sample is returned to the client for a reason other than routine disposal, the chain-of-custody is signed and a copy is put in the file for that project. The original is sent on with the samples.

5.1.9 If a portion of a sample is sent to a subcontractor, a chain-of-custody form is generated by Sample Control to accompany the samples. A copy is placed in the files and the original is sent back to RASI with the written report.

5.2 Sample Security

5.2.1 The door to the Sample Control room is kept locked unless occupied. Only Sample Control personnel have keys to the sample control areas. All samples must be returned to Sample Control each night. During times which sample control personnel are unavailable, such as after normal business hours, one laboratory person will be the designated "sample custodian". If there is a time when only one individual is working in the laboratory, he or she will be the designated "sample custodian". On these occasions samples will be returned to Sample Control, signed in and placed on a cart inside the walk-in cooler. The next morning sample control personnel will make sure that all samples on the cart are signed in. Sample Control personnel will then put samples away.

Figure 1
Chain-of-Custody Form

ROSS • Ross Analytical Services, Inc.
16433 Foltz Industrial Parkway • Strongsville, Ohio 44136
(216) 572-3200 • Fax (216) 572-7620 • 1-800-325-7737

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

Page ____ of ____

Report to: _____

Lab Contact _____

Bill to: _____

Purchase Order No. _____

Project Reference _____

Telephone: _____

ONE CONTAINER PER LINE

Sample Number	Sample Type/Description	Date Collected	Container Type	Pre-servative	Required Tests	Condition on Receipt (Lab)

Possible Hazard Identification: Non-hazard Flammable Skin Irritant Other	Sample Disposal: Return to Client Disposal by Lab Archive (mos)
Turnaround Time Required: Normal Rush Report Required By	QA Requirements:
1. Relinquished by *Signature/Affiliation* Date	1. Received by Signature/Affiliation Date
2. Relinquished by *Signature/Affiliation* Date	2. Received by Signature/Affiliation Date
Special Instructional Comments:	

Figure 2
Chain-of-Custody Seals

CUSTODY SEAL
ROSS ANALYTICAL SERVICES, INC.
16433 FOLTZ PARKWAY · STRONGSVILLE, OH 44136

SITE:_____
SAMPLE NO.:_____
DATE:_____

SIGNATURE:_____ PRINTED NAME:_____

CUSTODY SEAL
ROSS ANALYTICAL SERVICES, INC.
16433 FOLTZ PARKWAY · STRONGSVILLE, OH 44136

SITE:_____
SAMPLE NO.:_____
DATE:_____

SIGNATURE:_____ PRINTED NAME:_____

ROSS ANALYTICAL SERVICES, INC.

INTERNAL CHAIN OF CUSTODY (SAMPLE CONTROL RECORD)

Sample(s) Taken	Test(s)	Taken by	Date and Time Out	Date and Time In	Storage Site

Figure 3
Internal Chain-of-Custody

ROSS Ross Analytical Services, Inc.	SOP Standard Operating Procedure	SOP No: SC-002-00 Initial Date: 2/1/91 Page 1 of 8 Date Revised:

Title:

Sample Receipt, Log-in and Storage for Non-CLP Samples

Prepared by:	Approved by:	Date:	QC Approval:
[signature]	[signature]	5/10/91	[signature]

1.0 PURPOSE

This procedure defines the actions taken to properly log and store samples received at the RASI Laboratory.

2.0 APPLICATION

This procedure applies to all samples sent through the Laboratory for analysis or other processing, other than those received through the US EPA Contract Lab Program.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

SOP No. SC-001-00, Chain of Custody and Sample Security
SOP No. SC-003-00, Monitoring Sample Control Refrigerator Temperature

5.0 PROCEDURE

5.1 Sample Receipt

5.1.1 If there is paperwork on the outside of the container used for transporting the samples, read to see if it notes any hazards associated with the samples.

5.1.2 If the samples are in a cooler or if they are suspected of being hazardous, open the transportation container in the Sample Control hood. The appropriate protection should be used by the person handling the samples. Normally this means gloves and protective glasses. In some cases a face mask may be needed.

5.13 Remove the samples from the transportation container. Line the samples up in the most logical order. (This may be by sample date, numeric, alphabetic, or according to the paperwork if there is no other logical order.)

5.2 Sample Inspection

5.2.1 Check the sample against the paperwork to verify the paperwork is accurate. Note any discrepancies on the chain of custody, if applicable, and also on a nonconformance memo.

5.2.2 The condition of these specific samples should be checked as follows. All discrepancies between the sample condition and the desired condition as mapped out in the following paragraphs will be noted on the chain of custody if applicable and on (Figure 1).

Figure 1

NONCONFORMANCE MEMO

Client: _____ Work Order No.: _____ Filed by: _____ Date: _____

Sample(s) Affected: _____

Area: __ SC __ OP __ IP __ II __ GC __ GC/MS __ MT __ Other _____

Nonconformance

Please explain nonconformances (issues which affect data quality) in the space below for reasons such as (1) holding times exceeded; (2) samples which are lost during extraction or analysis, especially when no re-prep. is possible; (3) quality control (QC) data is outside of defined limits; (4) when reported detection limits are higher than specified in contract, method, and/or project plan; (5) sample received in a broken condition, without proper preservation, or in an improper container; (6) or, any other problem issues which may arise in the course of sample receipt, preparation, or analysis.

(Note that nonconformance memos are to be used to provide accurate and concise information regarding problems associated with our client's samples.)

_____

_____

_____

_____

_____

_____

_____

_____

_____

CORRECTIVE ACTION

__ Client informed verbally on _____ by _____

__ Client informed in writing on _____ by _____

__ Sample(s) processed "as is". Comments: _____

__ Sample(s) on hold until _____ If released, notify: _____

Corrective action initiated/performed by: _____ Date _____

Operations concurrence: _____ Date _____

QA Concurrence: _____ Date: _____

CORRECTIVE ACTION VERIFICATION

__ Verified __ Cannot verify. Reason: _____

Verified by: _____ Date _____

ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

5.2.2.1 Hivol filters for TSP analysis

5.2.2.1.1 Remove filters from envelopes and sleeves and inspect for holes, tears, and excess particulate matter. If necessary, void the sample and fill out a Nonconformance Memo. Leave voided filters in the envelope.

NOTE: Wear gloves when handling filters.

5.2.2.1.2 Place filters in a rack as they are logged. (No numbers are placed on the filters; the lab number is placed in the envelope.)

5.2.2.2 Method 5 Stack Testing Samples

Check each sample for accuracy against the recovery sheets. Note any discrepancies on the recovery sheets and a Nonconformance Memo. Check the container numbers, filters, and liquid levels carefully.

5.2.3 (Figure 2) describes the necessary requirements for the more common sample types received. The aqueous fractions that should be preserved are checked with pH paper. The temperature of one sample of a shipment is taken with a calibrated thermometer and recorded on the work order. Problems are documented as nonconformances.

5.3 Sample Log in

5.3.1 Each sample submitted to the lab is assigned a unique identity. Logging in is performed on a computerized system called Sample and Analysis Management (SAM), developed by Radian Corp. The following information common to all samples in a batch is entered:

Client code and project number
The person within RASI requesting the work
Date of receipt
Date the report is due
Date samples can be discarded and the method of discarding

A work identification (such as case number)
Text comments applying to all samples
The following information specific to each sample is entered:

Sample matrix and the client's identification of the sample
Date of sample collection
Storage location in the laboratory
Test(s) to be performed
Text comments applying to individual samples

5.3.2 SAM assigns a hyphenated number to each sample and fraction within a sample. This number is written on the sample bottle in indelible marker. A fraction is usually a separate container appropriate for a specific set of tests; for instance, a sample being analyzed for cyanide, metals, and extractable organics would have three fractions. Sample Number 91-02-040-03B is interpreted as follows:

91 - Sample batch received in 1991
02 - Sample batch received in January (the first month)
040 - Sample batch is the 40th received in January
03 - Sample is the third in the batch
B - Fraction is the second on the sample

5.3.3 For SAM, the laboratory is divided into departments such as organic preparation, GC/MS, classical wet tests, and AA/ICP. Each test is assigned to one department. The system prints a master work order listing all samples and their tests and a work order for each department represented in the order. Each department work sheet lists only those samples which will be prepared or analyzed in the department. The system also prints a label for each sample container. Copies of the master work order are distributed to the Technical Director and to the appropriate Supervisors. The original is kept in a three-ring notebook in sample control. Copies of the department work orders are distributed to the appropriate departments supervisors'. (A copy of the master work order is filed with the raw data and report.) Dates of completion, reporting, and invoicing are later entered into SAM. The work order is archived (removed from the active database) about 3 months after it is invoiced.

5.3.4 The samples remain in Sample Control until removed for analysis. The temperatures of the refrigerators in the sample storage are monitored to ensure that proper storage temperature is maintained (See SOP No. SC-003-00). Transfer is documented on a Sample Control Record which is maintained by the sample custodian. The Sample Control Record documents all custody changes that occur in the laboratory and each procedure performed on the sample (See SOP No. SC-001-00).

5.4 Sample Storage

5.4.1 Hivol filters for TSP Analysis

5.4.1.1 Store in the balance room.

5.4.1.2 Place a Kimwipe over the rack.

5.4.2 Method 5 stack testing sample

5.4.2.1 Store on the metal shelves in sample control.

5.4.2.2 If it is a modified Method 5, contact the project manager, QA Coordinator, or Technical Director on how to store the samples.

5.4.3 Store highly concentrated samples in coolers in the cold box.

5.4.4 Extracts

5.4.4.1 Store extract samples on shelves (for metals) RT 1A-F or (for all others) CB 12A-F

NOTE: The refrigerator is separated into areas for Base-Neutrals, PCB, Acids, and Pesticides. These areas are clearly marked.

5.4.5 Use the chart (Figure 2) to determine where to store other common types of samples.

5.4.6 Non-routine samples will be stored according to the project manager, QA Coordinator or Technical Director instructions.

	Preserved to pH =	Bottle Type	Temperature	Storage Location
Water Samples				
Metals	2	any	room	RT
Hardness	2	any	room	RT
Oil & Grease	2	glass*	4 C	CB
Phenolics	2	glass	4 C	CB
COD	2	any	4 C	CB
TKN	2	any	4 C	CB
NH3	2	any	4 C	CB
NO3	2	any	4 C	CB
Phosphorus	2	any	4 C	CB
TOC	2	any	4 C	CB
Cyanide	10	any	4 C	CB
Sulfide	10	any	4 C	CB
pH	none	any	4 C	CB
Other Inorganics	none	any	4 C	CB
624	**	glass*	4 C	VOA Ref.
8240	**	glass*	4 C	VOA Ref.
SELVA	**	glass*	4 C	VOA Ref.
625	none	glass	4 C	VOA Ref.
8270	none	glass	4 C	VOA Ref.
Pesticides	none	glass	4 C	VOA Ref.
PCB	none	glass	4 C	VOA Ref.
Other Extractables	none	glass	4 C	VOA Ref.
Soil Samples				
Metals	none	any	room	RT
Other Inorganic	none	any	4 C	CB
624	none	glass*	4 C	VOA Ref.
8240	none	glass*	4 C	VOA Ref.
Other Volatiles	none	glass*	4 C	VOA Ref.
625	none	glass	4 C	VOA Ref.
8270	none	glass	4 C	VOA Ref.
Pesticides	none	glass	4 C	VOA Ref.
PCB's	none	glass	4 C	VOA Ref.
Other Extractables	none	glass	4 C	VOA Ref.

* Must have at least 2 bottles per sample
** Do not open bottles to take pH or temperature.

Figure 2

	Preserved to pH =	Bottle Type	Temperature	Storage Location
<u>Other Samples Types</u>				
Oil for PCB's	none	glass	room	RT
Other Oils	none	any	4 C	CB
Sludges	none	any	4 C	CB
VOST Tubes	none	glass	4 C	VOA Freezer
XAD-2 Resin	none	any	4 C	
Canisters	none	any	room	RT
PUF Filters	none	any	4 C	CB
Filters for Metals		any	room	RT
Coal	none	any	room	RT
QC Ampules	none	glass	room	RT
Charcoal Tubes	none	glass	4 C	VOA Freezer

"Any" means plastic or glass

Figure 2
(Continued)

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Sample Control Refrigerator Temperature Monitoring			
Prepared by:	Approved by:	Date: 6/8/95	QA Approval:

1.0 PURPOSE

To describe the procedure for monitoring and controlling the temperatures of refrigerators used for sample storage.

2.0 SCOPE

Most samples are maintained under refrigeration to preserve integrity. As a control measure, the temperatures of all sample storage refrigerators must be checked routinely. If readings are beyond acceptable limits, corrective action must be taken.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

SOP AD-005, Calibration of Thermometers

5.0 EQUIPMENT

5.1 Glass mercury thermometers which have been calibrated against an NBS-traceable thermometer within the past year

5.2 Mineral oil

5.3 Vials, 40 mL

6.0 PROCEDURE

6.1 Thermometer Preparation

6.1.1 Immerse the end of the thermometer in a 40 mL vial containing approximately 20 mL mineral oil. Seal the top to prevent spillage.

6.1.2 Place the thermometer vial near the center of the refrigerator.

6.2 Temperature Monitoring

6.2.1 Refrigerators are uniquely identified. The temperature of each refrigerator must be checked twice daily. The first temperature check must be done before 10:00 a.m., and the second in the mid-to-

late afternoon. The reading is recorded along with the date, time, and initials of the person takir the reading on a Refrigerator Temperature Record sheet (Section 7.0, Figure 1). Log sheets are kept in a folder with a magnetic clip, and are attached to a cabinet next to the cold box in Sample Control until completed. They are checked periodically and signed by a person from Quality Assurance. Completed pages will be filed in the QA Department.

6.2.2 The acceptable range for the refrigerator temperature is 4°C ± 2°C (or 39.2°F ± 3.6°F). The acceptance range for the organics extract freezer is -10 to -20 °C.

6.2.3 If a reading is found to be unacceptable, corrective action must be taken. A Refrigerator Nonconformance Memo (Section 7.0, Figure 2) which documents the actions taken must be filed.

6.2.4 Below are the recommended procedures to be followed when an unacceptable reading is obtained. Remember that each case may be unique and that good judgement will be necessary to decide on specific actions.

6.2.4.1 If the temperature is below 2°C:

1. Open door to warm the refrigerator

2. If problem cannot be corrected immediately, consult the Technical Director or QA Manager for actions to be taken (See Section 6.2.5)

6.2.4.2 If the temperature is above 6°C:

1. Check to see that the refrigerator is getting power.

2. Assume excess activity and recheck temperature after two hours.

3. Consult Technical Director or QA Manager for immediate actions to be taken. (See Section 6.2.5)

6..2.5 For cases where a serviceman is required, it may be necessary to take immediate actions to preserve the integrity of samples. The Technical Director or QA Manager must decide what these actions will be. They may include:

1. Do nothing until repairman comes.

2. Put ice or blue ice in malfunctioning refrigerator.

3. Move samples to another refrigerator.

6.2.6 If moved, samples may be returned to a refrigerator when two consecutive readings two hours apart are within acceptance limits.

7.0 FIGURES

Figure 1
Example Refrigerator Temperature Record

ROSS ANALYTICAL SERVICES
Refrigerator Temperature Record

Temperature, degree Celcius

	Date	Checker	SC 1	Cold Box	Time	Comments/Corrective Actions
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						
1						
2						

Acceptable Range = 4 +/− 2 degrees Celcius
If you have any problems or questions, contact the QA Department.

Figure 2
Example Refrigerator Nonconformance Memo

ROSS ANALYTICAL SERVICES

Refrigerator Nonconformance Memo

Date Reported _____ Refrigerator _____

Reported by _____ Refrigerator Contents _____

Acceptable Range: 2–6°C

1) Record temperatures when outside of acceptable range
2) Note the following corrective action(s) taken

A. Checked to see if refrigerator is on and has power
B. Opened door to raise temperature
C. Adjusted thermostat
D. Assumed heavy activity -- rechecked after 2 hours
E. Put ice or refrigerant in refrigerator
F. Called repair service
G. Moved samples to another refrigerator
H. Other _____

TIME	TEMP in °C	CORRECTIVE ACTION TAKEN (document all actions taken with the reference letter or a statement)

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: SC-004-01
Initial Date: 2/1/91
Page 1 of 1
Date Revised: 11/14/94

Title: Review of Sample Control Log Books			
Prepared by:	Approved by:	Date: 11/14/94	QA Approval:

1.0 PURPOSE

To describe the procedure for reviewing sample control records in the RASI Laboratory.

2.0 APPLICATION

This procedure applies to the review of the log book used to track sample removal from and return to Sample Control.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Sample Control log books are reviewed each day by the Sample Control Supervisor or designate for completeness and legibility. The reviewer will also check to ensure that all samples signed out the day before have been signed back into Sample Control.

5.2 Any nonconformances will be highlighted in the log book.

5.3 The nonconformance will be discussed immediately with the appropriate party. If sample integrity was affected, a Nonconformance Memo will be filed.

5.4 If a person is habitually delinquent, a memo will be sent to his/her supervisor.

5.5 All log book pages will be initialed and dated after review.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Sample Container Kit Preparation/Sample Collection and Preservation			
Prepared by:	Approved by:	Date: 12/7/94	QA Approval:

1.0 PURPOSE

This SOP describes how to assemble containers, preservatives, chain of custody papers, and instructions for sample collection.

2.0 APPLICATION

This SOP applies to projects in which the samples to be collected are water, soil, sediment, waste, and similar materials. It does not address air sampling media.

3.0 REFERENCES

3.1 Test Methods for Evaluating Solid Waste, SW-846.

3.2 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020.

4.0 ASSOCIATED SOP's

None.

5.0 PROCEDURE

5.1 Most water and soil sampling projects require RASI to provide sample containers. The Sales Department or other client contact person notifies Sample Control of the project scope, including the number of samples of each matrix and the tests to be performed. A RASI Bottle Kit List (Figure 1) may be filled out by the client contact person. If not, the appropriate containers and preservatives are selected based on the information in Table 1.

5.2 Members of the Sample Control Department assemble the kit. Pre-cleaned and certified bottles are used. Certificates of analysis provided by the bottle supplier are filed with the QA Coordinator. Glass bottles are wrapped in bubblepack or otherwise cushioned to prevent breakage. Bottles are packed in insulated coolers or, for some projects, left in their original packages with empty coolers provided.

5.3 Sample bottle labels, Request for Analysis forms, Chain of Custody forms, Chain of Custody seals, and solid coolant ("Blue Ice") are provided with all kits. Appropriate preservatives are provided if requested by the client.

5.4 A copy of "Sample Containers and Preservation for Water, Soil/Sediment, and Waste" (Figure 2) is annotated as necessary and provided with the kit.

5.5 Tables 1, 2 and 3 contain more detailed accounts of tests, containerization, sample volumes needed, preservatives, and holding times.

Table 1
Sample Containers, Amounts, Preservatives, and Holding Times for Routine Analyses of Water Samples

Test	Container	Min. Volume (mL)	Preservative	Hold time
Acidity	Plastic or glass	50	4° C	14 days
Alkalinity	Plastic or glass	50	4° C	14 days
Biochemical oxygen demand	Plastic or glass	1000	4° C	48 hrs.
Bromide	Plastic or glass	100	None required	28 days
Chemical oxygen demand	Plastic or glass	50	4° C; H_2SO_4 to pH<2	28 days
Chloride	Plastic or glass	50	None required	28 days
Chlorine, residual	Plastic or glass	200	None required	Immediate
Conductivity	Plastic or glass	100	4° C	28 days
Cyanide, total	Plastic or glass	500	4° C; NaOH to pH>12 (1)	14 days
Cyanide, amen. to chlorination	Plastic or glass	500	4° C; NaOH to pH>12 (1)	14 days
Fluoride	Plastic only	100	4° C	28 days
Hardness	Plastic or glass	100	HNO_3 to pH<2	6 mos.
Iodide	Plastic or glass	100	4° C	24 hrs.
MBAS (Surfactants)	Plastic or glass	500	4° C	48 hrs.
Nitrogen, ammonia	Plastic or glass	100	4° C; H_2SO_4 to pH<2	28 days
Nitrogen, total Kjeldahl	Plastic or glass	100	4° C; H_2SO_4 to pH<2	28 days
Nitrogen, nitrate + nitrite	Plastic or glass	100	4° C; H_2SO_4 to pH<2	28 days
Nitrogen, nitrate	Plastic or glass	50	4° C; H_2SO_4 to pH<2	28 days
Nitrogen, nitrite	Plastic or glass	50	4° C	24 hrs.
Oil and grease	Glass only	1000	4° C; H_2SO_4 to pH<2	28 days
Petroleum hydrocarbons	Glass only	1000	4° C; H_2SO_4 to pH<2	28 days
pH	Plastic or glass	25	4° C	24 hrs.
Phenolics	Glass only	100	4° C; H_2SO_4 to pH<2	28 days
Phosphorus, ortho	Plastic or glass	50	4° C	48 hrs.
Phosphorus, total	Plastic or glass	50	4° C; H_2SO_4 to pH<2	28 days
Residue, filterable (TDS)	Plastic or glass	100	4° C	7 days
Residue, non-filterable (TSS)	Plastic or glass	100	4° C	7 days
Residue, total (TS)	Plastic or glass	100	4° C	7 days
Residue, volatile (TVS)	Plastic or glass	100	4° C	7 days
Turbidity	Plastic or glass	100	4° C	48 hrs.
Silica	Plastic only	50	4° C	28 days.
Sulfate	Plastic or glass	50	4° C	28 days.
Sulfide	Glass BOD bottle (2)	300	4° C; ZnOAc; NaOH to pH>9	28 days.
Sulfite	Plastic or glass	50	None required	Immediate
Total organic carbon (TOC)	Plastic or glass	25	4° C; H_2SO_4 to pH<2	28 days
Total organic halogen (TOX)	Glass septum cap bottle	100	4° C (3)	28 days
Chromium, hexavalent	Plastic or glass	100	4° C	24 hrs.
Mercury, dissolved	Plastic or glass	100	Filter on site; HNO_3 to pH<2	28 days
Other metals, dissolved	Plastic or glass	100	Filter on site; HNO_3 to pH<2	6 mos.
Mercury, total	Plastic or glass	100	HNO_3 to pH<2	28 days
Other metals, total	Plastic or glass	100	HNO_3 to pH<2	6 mos.
Purgeable organics (VOC's)	Septum cap glass vial	40	4° C; HCl (4)	14 days (5)
Semivolatile organics (BNA's)	Amber glass only	1000	4° C (4)	7 days (6)
Organochlorine pesticides/PCB's	Amber glass only	1000	4° C (4)	7 days (6)
Organophosphate pesticides	Amber glass only	1000	4° C (4)	7 days (6)
Phenoxy acid herbicides	Amber glass only	1000	4° C (4)	7 days (6)
TCLP for VOC's (ZHE)	Septum cap glass	100	4° C	14 days (7)
TCLP for metals	Plastic or glass	200	None needed	28 days (7)
TCLP for extr. organics	Glass only	1000	4° C	14 days (7)

Notes:

1. If the sample is known or suspected to contain oxidants such as residual chlorine, also add 0.6 g ascorbic acid.
2. A 250 mL septum cap bottle may be substituted.
3. If the sample is known or suspected to contain oxidants such as residual chlorine, add a few grains of NaSO3.
4. If the sample is known or suspected to contain oxidants such as residual chlorine, add a few grains of $Na_2S_2O_3$ before the HCl.
5. It is a common belief that VOC analyses have a 7 day holding time if not preserved. This is not supported by the EPA methods.
6. Time to extraction is 7 days. The extract must be analyzed within 40 days of extraction.
7. This time is for starting the TCLP leaching. Normal analytical holding times begin with completion of the TCLP leaching.

Table 2
Sample Containers, Amounts, Preservatives, and Holding Times for Routine Analyses of Soils, Sediments and Waste Samples

Test	Container	Min. g	Preservative	Hold time
Cyanide, total	Glass only	20	4° C	14 days
Cyanide, amen. to chlorination	Glass only	20	4° C	14 days
Moisture/solids	Glass only	10	4° C	7 days
Oil and grease	Glass only	50	4° C	28 days
Petroleum hydrocarbons	Glass only	50	4° C	28 days
pH	Glass only	25	4° C	24 hrs.
Phenolics	Glass only	10	4° C	28 days
Total organic carbon (TOC)	Glass only	10	4° C	28 days
Total organic halogen (TOX)	Glass only	10	4° C	28 days
Mercury	Glass only	10	4° C	28 days
Other metals	Glass only	10	4° C	6 mos.
Purgeable organics (VOC's)	Glass only	5	4° C	14 days
Semivolatile organics (BNA's)	Glass only	30	4° C	14 days (1)
Organochlorine pesticides/PCB's	Glass only	30	4° C	14 days (1)
Organophosphate pesticides	Glass only	30	4° C	14 days (1)
Phenoxy acid herbicides	Glass only	30	4° C	14 days (1)
TCLP for VOC's (ZHE)	Glass only	40	4° C	14 days (2)
TCLP for metals	Glass only	150	None needed	28 days (2)
TCLP for extr. organics	Glass only	150	4° C	14 days (2)

Notes:

1. Time to extraction is 14 days. The extract must be analyzed within 40 days of extraction.
2. This time is for starting the TCLP leaching. Normal analytical holding times begin with completion of the TCLP leaching.

Table 3
Allowable Combinations of Tests From Each Container Type

Container and Preservative Combination	Allowed Tests
Plastic or glass, preserved at 4° C	Acidity Alkalinity Biochemical oxygen demand Bromide Chloride Chlorine, residual Chromium, hexavalent Conductivity Fluoride (only if bottle is plastic) Iodide MBAS (surfactants) Nitrogen, nitrite pH Phosphorus, ortho Residue, filterable (TDS) Residue, non-filterable (TSS) Residue, total (TS) Residue, volatile (TVS) Turbidity Silica (only if bottle is plastic) Sulfate Sulfite
Plastic or glass, preserved at 4° C with H_2SO_4 to pH<2	Chemical oxygen demand Nitrogen, total Kjeldahl Nitrogen, nitrate + nitrite Nitrogen, nitrate Phenolics (only if bottle is glass) Phosphorus, total Total organic carbon (TOC)
Plastic or glass, preserved with HNO_3 to pH<2	Hardness Metals (all)
Plastic or glass, preserved with NaOH to pH>12	Cyanide, total Cyanide, amen. to chlorination
Amber glass, preserved at 4° C	Semivolatile organics (BNA's) Organochlorine pesticides /PCB's Organophosphate pesticides Phenoxy acid herbicides TCLP for extr. organics TCLP for metals
Glass, preserved at 4° C (non-aqueous samples)	All tests except VOC's (total or TCLP)

Note: Aqueous samples for oil and grease, petroleum hydrocarbons, sulfide, and total organic halide (TOX) require individual bottles for each test despite having in some cases common preservation requirements.

Figure 1
RASI Bottle Kit Checklist

RASI BOTTLE KIT CHECKLIST

SEE OTHER SIDE FOR IMPORTANT SAMPLING INSTRUCTIONS

Drop Off / Ship (Circle one)

CLIENT_____ SHIP DATE_____

WATER SAMPLES

PARAMETER	BOTTLE TYPE	#/SAMPLE	PRESERVATIVE	NUMBER OF SAMPLES	TOTAL ENCLOSED
Volatile Organics	40 mL vial		HCl		
BNA's	1/2 gallon glass		NONE		
Extractable Organics	1/2 gallon glass		NONE		
Herbicides	1000 mL glass		NONE		
Dioxins/Furans	1000 mL glass		NONE		
Unpreserved Wet Tests	Plastic		NONE		
Preserved Wet Tests	Plastic		H_2SO_4		
Preserved Wet Tests	Glass		H_2SO_4		
Metals/Hardness	Plastic		HNO_3		
Oil & Grease/TRPH	1000 mL glass		H_2SO_4		
TOX	250 mL w/septum		H_2SO_4		
Cyanide	1000 mL plastic		NaOH		
Sulfide	300 mL BOD		ZnOAc,NaOH		
Phenolics	500 mL glass		H_2SO_4		
TCLP for Volatiles	250 mL w/septum		NONE		
TCLP for Others	1/2 gallon glass		NONE		

SOIL & WASTE SAMPLES

PARAMETER	BOTTLE TYPE	#/SAMPLE	PRESERVATIVE		
Volatile Organics	4 oz. glass		ICE		
Extractable Organics	8 oz. glass		ICE		
Metals & Other Tests	8 oz. glass		ICE		

RFA/COC FORMS Yes [ ] No [ ]
LABELS Yes [ ] No [ ]
BLUE ICE Yes [ ] No [ ]
TRIP BLANKS Yes [ ] No [ ]
How Many?
CUSTODY SEALS Yes [ ] No [ ]
PRESERVE BOTTLES? Yes [ ] No [ ]

SPECIAL COMMENTS/SHIPPING INFO

PACKED BY_____
CHECKED BY_____

Figure 2
Sample Containers and Preservation for Water, Soil/Sediment, and Waste

ROSS • Ross Analytical Services, Inc.
16433 Foltz Industrial Parkway • Strongsville, Ohio 44136
216/572-3200 • Fax 216/572-7620 • 1-800-325-[illegible]

SAMPLE CONTAINERS AND PRESERVATION FOR WATER, SOIL/SEDIMENT, AND WASTE

The table on the back of this sheet lists the bottles and preservatives to use for your project. Listed quantities are per sample. Unless otherwise noted below, fill bottles only to their shoulder and add acids (H_2SO_4, HCl, or HNO_3) to pH<2 and NaOH to pH>12. Samples for all tests except metals require storage and shipment at 4°C.

Volatile Organics: 40 mL septum cap vials. If residual chlorine or other oxidants are present (as in, for instance, a chlorinated effluent), add 4 drops of 10% sodium thiosulfate solution to the vial. With or without thiosulfate, fill partway, add 5 drops of 1:1 HCl, then fill to just overflowing. Pop the septum out of the cap and slide it, shiny side down, across the top of the vial. Screw the cap on partway, and then push down gently on the septum to expel air (the eraser end of a pencil works well for this). Tighten the cap and invert; if there are bubbles, empty the vial and start over.

Zero Headspace Extraction (ZHE): Fill and expel air as described for volatile organics above, but DO NOT add preservative.

Extractable Organics: If residual chlorine or other oxidants are present add 1-2 mL of 10% sodium thiosulfate ($Na_2S_2O_3$) solution to each 1/2 gallon jug. Do the same for herbicides and dioxins/furans (use 1 mL for a 1 liter bottle).

Cyanides: If residual chlorine or other oxidants are suspected, add a few crystals of ascorbic acid in addition to the NaOH.

Sulfide: Fill bottle nearly to the ground glass area, add NaOH to pH>9, then 2 mL zinc acetate solution. Fill further into the ground glass are, put in the stopper (sample will overflow) and twist to set it.

Total Organic Halogen (TOX): Fill partway, add 10 drops of 1:1 H2SO4, then fill and expel air as described for volatile organics above.

Unpreserved Wet Tests:

Acidity	Alkalinity	BOD
Bromide	Chloride	Color
Fluoride	Surfactants (MBAS)	Nitrite-N
Chromium +6	Orthophosphate	pH
TSS	TDS	TS
Sulfate	Sulfite	Turbidity
Conductivity	Residual Chlorine	Odor

Sulfuric Acid Preserved Wet Tests:

Ammonia-N	Total Phosphorus	Kjeldahl-N
Nitrate-N	Nitrate/Nitrite-N	COD

Material Safety Data Sheets for the necessary preservatives are provided. These sheets are those of the manufacturer from which we purchase the materials, annotated as necessary to reflect the dilutions which we make.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Screening of potentially radioactive samples.				
Prepared by:	Approved by:	Date:		QA Approval:

1.0 PURPOSE

This SOP describes what to do when receiving potentially radiological materials.

2.0 APPLICATION

This SOP applies specifically to all samples received from Department of Defense (DOD), Department of Energy (DOE), and other sites that potentially have radiological material contamination.

3.0 REFERENCES

10 CFR, Section 20.205 (C)(1).

4.0 ASSOCIATED SOPs

None.

5.0 EQUIPMENT

5.1 Eberline Geiger Counter

5.2 Eberline ^{137}Cs - 5 μCi source

6.0 PROCEDURE

6.1 The Geiger counter will be checked at least each month by placing the probe against the open top of the lead container holding the ^{137}Cs - 5 μCi source. The instrument reading is recorded in the logbook (see Figure 1) and the value is compared to previous readings to ensure proper performance.

6.2 Packages must be screened as soon as they arrive, no later than 3 hours after arrival. Packages received after normal working hours must be screened within 2 hours after the beginning of the next working day.

Latex gloves must be worn when performing this procedure. If a package is determined to be "hot", the gloves must be removed and left with the package. Wash hands immediately.

6.3 The background radiation reading is taken in the warehouse area away from the packages to be screened. The average reading on the meter is taken and recorded.

6.4 Prior to opening the container, use the Geiger counter to check the package for any radioactivity present at a distance of three feet from the package. If radioactivity is detected at a factor of 2 times or greater above the normal background level, the package is considered hot. Immediately contact the Radiation Safety Officer (RSO).

6.5 If the measurement is less than 2 times above the background level, determine the radiation level at the surface of the package by placing the detector against the package. If no radiation is detected at a level of 2 times above the background, open the package and determine the radiation level around each sample container. If samples are contained in canisters, use the probe to check around the valves of the canisters. Whenever a reading is detected at a level of greater than 2 times above background, immediately contact the RSO.

6.6 Record the meter reading of the sample in the Geiger counter check log and note if the sample passed or failed the screening procedure. Any reading 2 times background or greater fails the screening procedure and the RSO must be notified.

Figure 1
Geiger Counter Check Log

[illegible]

GEIGER COUNTER CHECK LOG

ROSS ANALYTICAL SERVICES, INC.

INSTRUMENT SERIAL #	CHECK SOURCE	BACKGROUND LEVEL	SAMPLE READING	SOURCE READING	DATE	INITIAL	PASS FAIL	WORK ORDER #

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Sample Preservative Shipping Procedures			
Prepared by:	Approved by:	Date: 4/5/95	QA Approval:

1.0 PURPOSE

This SOP describes the procedures used to ship sample preservatives to clients.

2.0 APPLICATION

This SOP applies to Ascorbic Acid, Hydrochloric Acid, Nitric Acid, Sodium Hydroxide, Sodium Thiosulfate, Sulfuric Acid, and Zinc Acetate which are routinely shipped to clients as a sample preservatives.

3.0 REFERENCES

49 CFR
International Air Transportation Association (IATA), Dangerous Goods Regulations

4.0 ASSOCIATED SOP's

None

5.0 EQUIPMENT AND SUPPLIES

5.1 30 mL plastic container with screw cap

5.2 UN designated 4G fiberboard box

5.3 Corrosive resistant absorbent material

5.4 Plastic bags zip-lock

5.5 Electrical tape

5.6 Packaging tape

5.7 Packing Materials (bubble wrap, polyfoam, vermiculite, expanded clay/diatomaceous earth)

5.8 Shipping labels

5.8.1 Corrosive

5.8.2 Package orientation (arrows)

5.8.3 Danger Do Not Load In Passenger Aircraft

5.8.4 UPS and Federal Express Labels (as needed)

5.9 Shipping papers

5.9.1 Federal Express

5.9.2 UPS

5.10 Paint can

5.11 Material Safety Data Sheets

5.12 Instructions for using the shipped chemicals

5.13 Hammer

5.14 Ascorbic acid

5.15 Hydrochloric acid (19%)

5.16 Nitric acid (20%)

5.17 Nitric acid (35%)

5.18 Sodium hydroxide solution (25%)

5.19 Sodium thiosulfate (10%)

5.20 Sulfuric acid (48%)

5.21 Zinc acetate (10%)

6.0 PROCEDURE

The procedures for shipping the materials listed in Section 2.0 has been divided into five separate sections.

1. Section 6.1 details the proper way to ship nitric acid (20%), hydrochloric acid (19%), sulfuric acid (48%), and sodium hydroxide solution (25%) by Federal Express.

2. Section 6.2 details the proper way to ship nitric acid (35%).

3. Section 6.3 details the proper shipping method for hydrochloric acid, Sodium hydroxide solution, and sulfuric acid by UPS.

4. Section 6.4 describes the procedure for shipping different dangerous goods including 35% Nitric Acid in the same package by Federal Express.

5. Section 6.5 describes the procedure for shipping ascorbic acid, sodium thiosulfate, and zinc acetate which are not regulated under DOT or IATA regulations.

6.1 SHIPPING VIA FED-EX (Dangerous Goods in Excepted Quantities)

This procedure is for shipping hazardous goods under IATA regulations for "Dangerous Goods in Excepted Quantities" by Federal Express. This procedure applies to Nitric acid (20% or less), Hydrochloric acid (19%), Sulfuric acid (48%), Sodium hydroxide solution (25%) and non-regulated materials shipped alone or in combination.

This is the primary method used to ship these materials.

6.1.1 Nitric acid must be 20% or less.

6.1.2 The inner container of each of these hazardous goods must contain 30 mL or less and the liquids must not completely fill the inner container. The cap of plastic bottles must be secured with electrical tape. The containers must be marked to identify the contents.

6.1.3 The container is placed in an individual zip-lock bag with sufficient absorbent material to contain the entire contents of the inner container.

6.1.4 This package is placed in a UN designated 4G fiberboard box with the MSDS, usage instructions and additional packaging material (e.g. vermiculite) to ensure the inner contents are held securely. The box (outer package) is taped shut with strong packaging tape.

The total volume of all inner containers must not exceed 500 mL in any outer package.

6.1.5 Package orientation labels are placed on opposing sides of this package or the cooler that contains this package.

6.1.6 The "Dangerous Goods in Excepted Quantities" label (see Appendix A) must be placed on the outer package or the cooler that contains the package.

This label must have the signature, title and address of the shipper, and the date. The hazard class box must be marked and the UN numbers of each material(s) that is/are contained in the package.

Nitric Acid, Class 8, UN2031, II

Hydrochloric Acid, Class 8, UN1789, II

Sulfuric Acid, Class 8, UN1830, II

Sodium Hydroxide, Solution, Class 8, UN1824, II

6.1.7 Non-hazardous goods shipping papers must note "Dangerous Goods In Excepted Quantities" (see Appendix B). Because there is no designated area on the shipping paper for this notation, any unused space is acceptable.

6.2 SHIPPING VIA FED-EX (35% Nitric Acid Solution)

Nitric Acid (35%) - There are no "Limited Quantity Exceptions" or "Exceptions for Small Quantities" in the DOT regulations (49 CFR 173.4) for nitric acid. Shipments by Federal Express under IATA regulations for nitric acid at 35% will require dangerous goods paper work, packaging, marking and labeling. This procedure is intended for the shipment of nitric acid only and not in combination with any other material.

Nitric acid is shipped Federal Express non-passenger air only.

6.2.1 This acid is shipped in 30 mL plastic bottles or 1 to 30 mL glass ampules labeled "NITRIC ACID". The cap of plastic containers must be secured with electrical tape to prevent leakage during transportation.

6.2.2 This bottle or ampule is placed in a zip-lock plastic bag.

6.2.3 The acid, now in a bag, is overpacked in a friction lid can (paint can) with sufficient absorbent capable of absorbing the entire contents of the container. Additional absorbent material should be added to the can to prevent movement of the inner receptacle during transportation. The friction lid is sealed with a hammer.

6.2.4 The can is placed in a UN designated 4G fiberboard box with the MSDS, usage instructions and sufficient packaging material (e.g. vermiculite, polyfoam) to prevent movement of the can inside of the box. The box is closed and taped with strong packaging tape.

6.2.5 The package **must** be labeled with:

6.2.5.1 Package orientation labels (arrows) on opposing sides (see Appendix C).

6.2.5.2 **"CORROSIVE"** on one side (see Appendix C).

6.2.5.3 **"DANGER DO NOT LOAD IN PASSENGER AIRCRAFT"** (see Appendix C).

6.2.6 The package **must** be marked with:

6.2.6.1 Shipper's name and address

Ross Analytical Services, Inc.
16433 Foltz Industrial Parkway
Strongsville, Ohio 44136

6.2.6.2 The recipient's name and address

6.2.6.3 Proper shipping name, class, UN number, and packing group.

NITRIC ACID, 8, UN2031, II 30 mL
(35%)

This information must be printed exactly as written above including commas.

Following the name, print the actual volume of nitric acid being shipped and the concentration in parentheses below the name.

6.2.7 The Federal Express Dangerous Goods shipping papers must be complete (see Appendix D).

6.2.7.1 Section 1 - Include the Federal Express account number, your name, company phone number, and company (RASI) address.

6.2.7.2 Section 2 - Complete the recipient's name, phone number, company name and address.

6.2.7.3 Section 3 - Payment - check the appropriate box.

6.2.7.4 Section 4 - Service - check the desired box (check only one box).

6.2.7.5 Section 5 - Complete delivery and special handling as desired.

6.2.7.6 Section 6 - Complete the number of packages, weight, and declared value (if applicable).

6.2.7.7 For the purpose of shipping nitric acid, under TRANSPORT DETAILS "Passenger and Cargo Aircraft" must be crossed out, and under Shipment Type, "Radioactive" must be crossed out.

6.2.7.8 In the section called "NATURE AND QUANTITY OF DANGEROUS GOODS", the proper shipping name is NITRIC ACID with "(35%) printed directly below the name.

The class or division is "8", the UN or ID No. is UN2031.

The subsidiary risk column is left blank.

In the quantity and type of packing column, "1 Fiberboard Box X 30 mL " (use actual volume) must appear. This volume can not legally exceed 30 L, and we should never need to send more than 100 mL .

The packing instruction column has "813 II".

The authorization column is left blank.

6.2.7.9 Emergency phone number - **CHEMTREC 1 (800) 424-9300** - This is a 24 hour emergency phone number as required by the regulations.

6.2.8 The finished package can be placed inside of a cooler for shipping if the information described in Sections 6.2.5 and 6.2.6 is on the cooler. In addition to this information, "INSIDE PACKAGES COMPLY WITH PRESCRIBED SPECIFICATION" must be marked on the cooler.

6.3 SHIPPING BY UPS (Small Quantities)

Hydrochloric acid (19%), Sodium hydroxide solution (25%), and Sulfuric acid (48%) can be shipped by UPS air under "Exceptions for Small Quantities" in 49 CFR 173.4. Under this exception, there are minimum requirements for packaging, labeling and marking. Hazardous labels, proper shipping name, and dangerous goods shipping paper is not required.

This method does not apply to Nitric Acid.

6.3.1 For this exception to apply to the materials listed in 6.3, the quantity shipped must be 30 mL or less.

6.3.2 The inner receptacle is a 30 mL plastic bottle or glass ampule containing 30 mL or less of the materials described in Section 6.3. The cap of the plastic bottle must be secured with electrical tape to ensure the container will not leak in transportation. The container must be marked to identify the contents.

6.3.3 The container is placed in an individual zip-locked plastic bag with sufficient absorbent material capable of absorbing the entire contents of the inner container.

6.3.4 This package is placed in a UN designated 4G fiberboard box with the MSDS, usage instructions and additional packaging material (e.g. vermiculite, polyfoam. bubble wrap) to ensure the inner contents are held securely. The box is taped shut with strong packaging tape.

6.3.5 Package orientation labels on opposing sides of the package is the only labeling requirement.

6.3.6 The package must be marked with:

6.3.6.1 The shipper's name and address.

6.3.6.2 The recipient's name and address.

6.3.6.3 The words "THIS PACKAGE CONFORMS TO CONDITIONS AND LIMITATIONS SPECIFIED IN 49 CFR 173.4".

6.3.7 Multiple 30 mL bottles of the material listed in 6.3 can be shipped in the same box. Each individual bottle must be packaged as described in 6.3.1 through 6.3.4 before being placed in the 4G fiberboard box.

The total volume of all inner containers must not exceed 500 mL in any one outer container.

The gross weight of the completed package cannot exceed 29 Kg (64 pounds).

6.3.8 The finished package can be placed inside of a cooler for shipping if the information described in Sections 6.3.5 and 6.3.6 is on the cooler. The gross weight of the completed package cannot exceed 29 Kg (64 pounds).

6.3.9 The UPS shipping papers must be complete (See Appendix G).

6.3.9.1 Section 1 -**SHIPMENT FROM**- include the UPS account number, your name, company telephone number, and the company (RASI) address.

6.3.9.2 Section 2 -**EXTREMELY URGENT DELIVERY TO**- Fill in the recipient's name, telephone number (if known), and the company's name and address.

6.3.9.3 Section 3 -**WEIGHT AND ZONE**- In the box labeled "WEIGHT" fill in the weight (in pounds) of the package. In the box labeled "ZONE" write "022" in the spaces provided.

6.3.9.4 Section 4 -**TYPE OF SERVICE**- Check the box labeled "NEXT DAY AIR".

6.3.9.5 Section 5 -**OPTIONAL SERVICES**- Select any of the listed services, ONLY if specifically requested.

6.3.9.6 Section 6 -**ADDITIONAL HANDLING CHARGE**- Do not fill in any information in these spaces.

6.3.9.7 Section 7 -**METHOD OF PAYMENT**- Check the box labeled "BILL SHIPPER".

6.3.9.8 Section 8 -**RECEIVER'S INFORMATION**- Do not fill in any information in these spaces.

6.3.9.9 Section 9 -**SHIPPER'S SIGNATURE**- Put your signature and the date in the space provided.

6.4 SHIPPING VIA FED-EX (Dangerous Goods)

Nitric acid (35%), Hydrochloric acid (19%), Sodium hydroxide solution (25%), and Sulfuric acid (48%) can be shipped together in the same UN designated 4G box by Federal Express under the IATA dangerous goods shipping regulations. Proper packaging, labeling, marking and hazardous goods papers are required.

6.4.1 The inner receptacle is a 30 mL plastic bottle or glass ampule containing 30 mL or less of the material described in Section 6.3. The cap of the plastic bottle must be secured with electrical tape to ensure the container will not leak in transportation. The container must be marked to identify the contents.

6.4.2 Each container is placed in an individual zip-lock bag with sufficient absorbent material capable of absorbing the entire contents of the inner container.

6.4.3 Nitric acid must be overpacked in a friction lid can with sufficient absorbent material capable of absorbing the entire contents of the inner container.

6.4.4 The packages are placed in a UN designated 4G fiberboard box with the MSDS's, usage instructions, and sufficient packaging material to cushion and prevent movement of the contents in transportation. The box is closed and taped shut with strong packaging tape.

6.4.5 The package must be labeled with:

6.4.5.1 Package orientation labels (arrows) on opposing sides (see Appendix C).

6.4.5.2 "CORROSIVE" on one side (see Appendix C).

6.4.5.3 "DANGER DO NOT LOAD IN PASSENGER AIRCRAFT" (see Appendix C).

6.4.6 The package must be marked with:

6.4.6.1 Shipper's name and address

6.4.6.2 The recipient's name and address.

6.4.6.3 Proper shipping name, class, UN number, and packing group.

NITRIC ACID, 8, UN2031, II 30 mL
(35%)

HYDROCHLORIC ACID, 8, UN1789, II 30 mL
(19%)

SODIUM HYDROXIDE SOLUTION, 8, UN1824, II 30 mL
(25%)

SULFURIC ACID, 8, UN1830, II 30 mL
(48%)

name and actual volume must be included on the package.

6.4.7 The Federal Express Dangerous Goods shipping paper must be complete (see Appendix E).

6.4.7.1 Complete the paper work as described in Sections 6.2.7.1 through 6.2.7.9.

6.4.7.2 Nature and Quantity of Dangerous Goods section. In this section, the proper shipping name, class, UN number, quantity and packing instructions must be written separately for each of the materials being shipped (see Appendix E). The package type and "Q" value is written at the bottom of the "Quantity and type of packing" column. The subsidiary risk and authorization columns are left blank.

Only the actual material(s) and volume(s) being shipped will be in this section. See Appendix E for details.

Calculating the "Q" value - The quantities of different dangerous goods contained in one outer package must be such that "Q" does not exceed the value of 1 where "Q" is calculated using the formula:

$$Q = n1/M1 + n2/M2 + n3/M3 \ldots$$

Example $Q = .030L/30L + .030L/30L = 0.002$

where n1, n2 etc. are the net quantities per package of dangerous goods and M1, M2, etc. are the maximum net quantities per package permitted for the shipment of each material by cargo aircraft.

6.4.7.3 Emergency phone number - **CHEMTREC 1 (800) 424-9300** - This is a 24 hour emergency number as required by the regulations.

6.4.8 The finished package can be placed inside of a cooler for shipping if the information described in Sections 6.4.5 and 6.4.6 is on the cooler along with the words "INSIDE PACKAGES COMPLY WITH PRESCRIBED SPECIFICATIONS".

6.4.9 The gross mass of the completed package cannot exceed 29 Kg (64 pounds).

6.5 Shipping Procedures for Ascorbic acid, Sodium thiosulfate (10%), and Zinc acetate.

These are not dangerous goods and no specific packaging, labeling, marking or shipping papers are required.

6.5.1 These materials can be shipped in plastic or glass containers.

6.5.2 The identity of the contents must be marked on the container and the lid of the container must be secured with electrical tape to ensure the lid will not come off in shipping.

6.5.3 All liquids should be shipped in a plastic bag with sufficient absorbent material capable of absorbing the entire contents of the inner receptacle.

6.5.4 UN specified packaging is not required.

6.5.5 Hazardous Goods shipping papers are not required. See Appendix F and G.

7.0 JOB TRAINING

Job specific training is required for shipping hazardous goods. Anyone using this SOP must be trained before performing the tasks described in this procedure.

APPENDIX A

2.7.5 Labelling

2.7.5.1 Each package containing dangerous goods in excepted quantities must be labelled with a label having minimum dimensions of 100 × 100 mm (4 × 4 in) which conforms in shape, colour, format and text to the specimen reproduced in Figure 2.7.B. A label must be affixed or printed on the package on a contrasting background. If the package is included in an overpack, this label must be clearly visible or appear on the overpack also.

2.7.5.2 The shipper must enter the required information on the label and sign it as indicated. The signature may be written by hand or it may be facsimile reproduced by a stamp or by printing. A typewritten signature is not acceptable. The label must not be completed or signed by a consolidator, a forwarder or an IATA cargo agent.

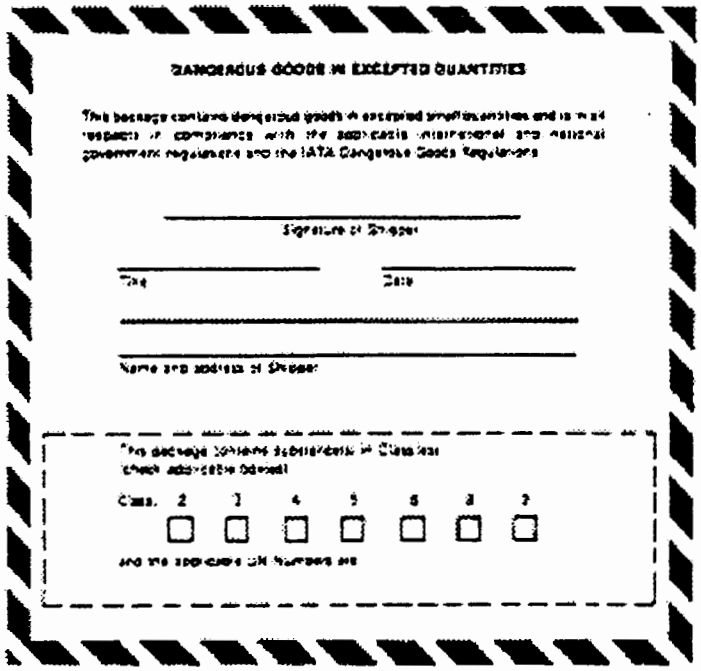
2.7.5.3 If required, the wording in English may be supplemented by an accurate translation in another language. Alternatively, a second label in a different language may be affixed. The text appearing in the space delineated by dotted lines may be changed to accommodate the shipper's requirements as long as it provides the same information.

2.7.5.4 The border of the label must have red diagonal hatching. The label may be printed in black and red on white paper or it may be printed in red only on white paper.

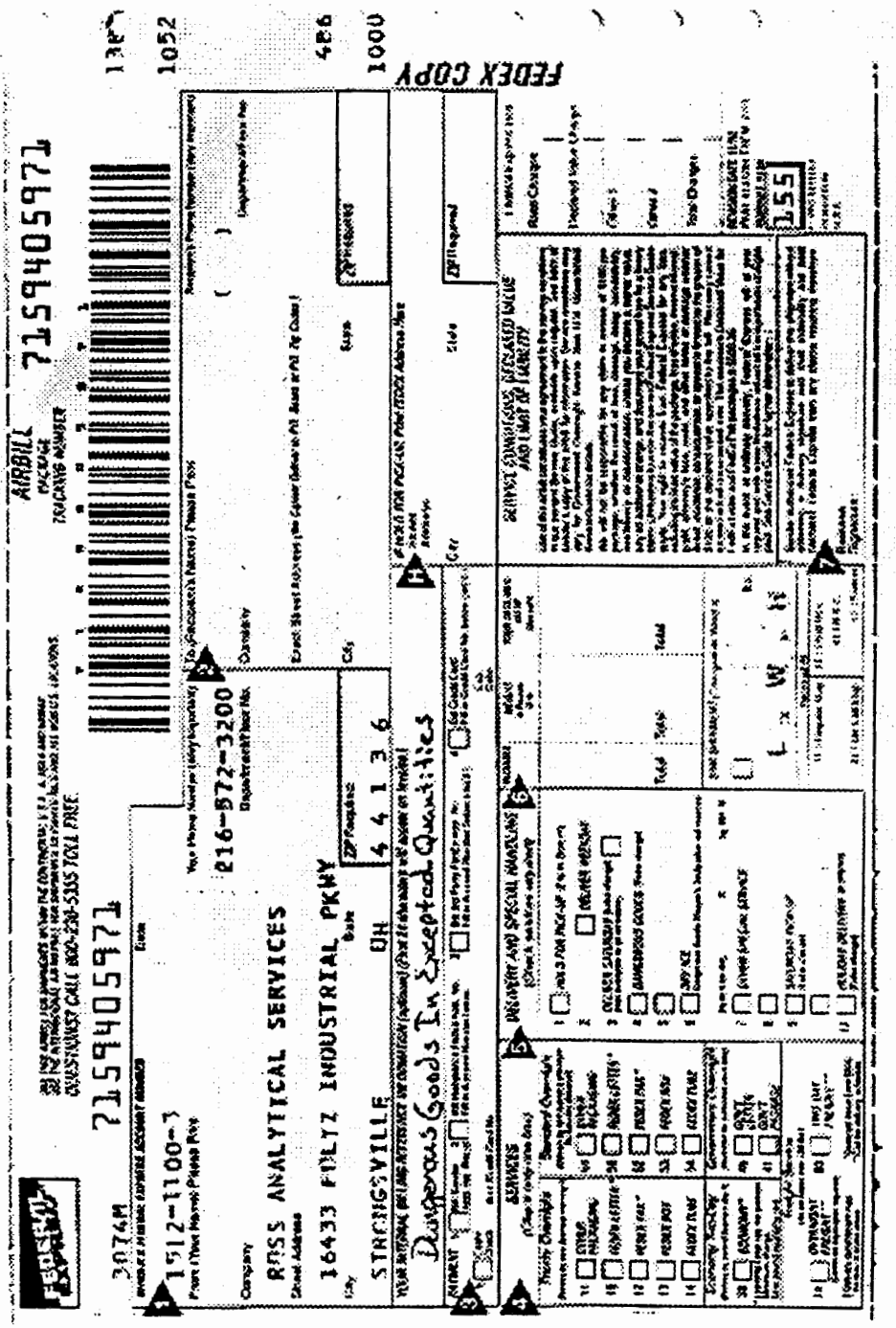
2.7.5.5 The label may contain identification information, including the name of its printer, provided that information is printed outside the border is no larger than 10 point type.

2.7.5.6 The label is shown in Figure 2.7.B.

FIGURE 2.7.B
Label for Dangerous Goods in Excepted Quantities



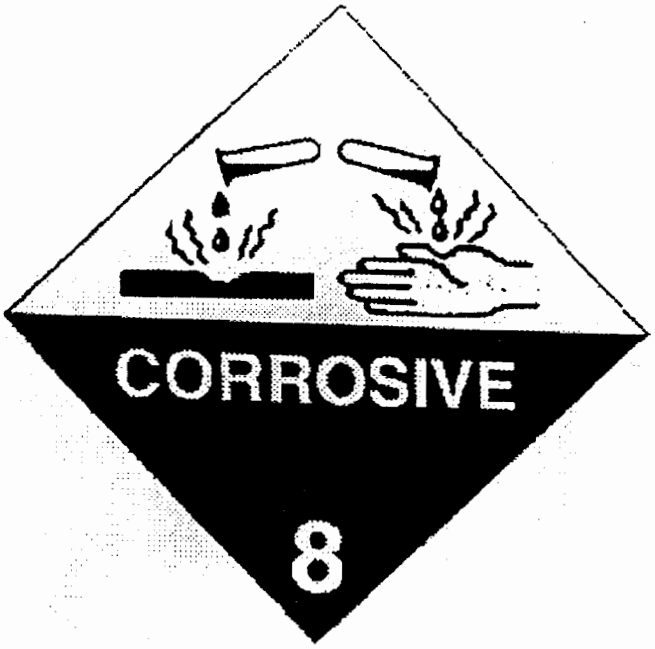
APPENDIX B



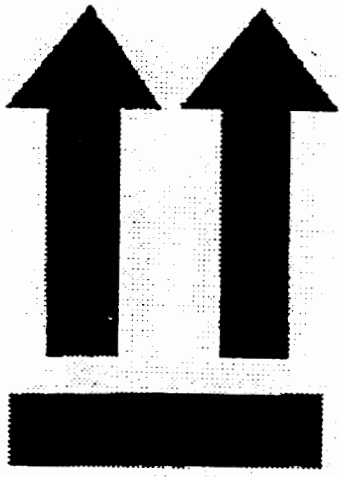
7159405971

7159405971

APPENDIX C



CORROSIVE LABEL



PACKAGE ORIENTATION LABEL

NO PASSENGER AIRCRAFT LABEL

APPENDIX D

AIRBILL 1648460273

4087 1648460273

1512-1100-3

216-572-3200

ROSS ANALYTICAL SERVICES

16433 FOLTZ INDUSTRIAL PKWY

STRONGSVILLE OH 44136

SIGNATURE RELEASE UNAVAILABLE

1648460273

TRANSPORT DETAILS

CARGO AIRCRAFT ONLY

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

Shipment type: NON-RADIOACTIVE | RADIOACTIVE

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification

Proper Shipping Name	Class or Division	UN or ID No.	Subsidiary Risk	Quantity and type of packing	Packing Inst.	Authorization
Nitric Acid (35%)	8	UN2031		1 Fiberboard Box x 30ml	813 II	

Additional Handling Information

I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in the proper condition for transport by air according to the applicable International and National Government Regulations.

Emergency Telephone Number (Required for all Class or Division 9 shipments)

Chemtrec Phone # 1-800-424-9300

* ACCEPTABLE FOR PASSENGER AIRCRAFT. THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

APPENDIX E

1648460273

4087A 1648460273

1512-1100-3

216-572-3200

ROSS ANALYTICAL SERVICES

16433 FOLTZ INDUSTRIAL PKWY

STRONGSVILLE OH 4 4 1 3 6

SERVICE

DELIVERY AND SPECIAL HANDLING

SEPARATE CONDITIONS, DECLARED VALUE AND COST OF CARRIAGE

L x W x H

157

SIGNATURE RELEASE UNAVAILABLE

1648460273

TRANSPORT DETAILS

Airport of Departure

Airport of Destination

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

Shipment type: NON-RADIOACTIVE | RADIOACTIVE

NATURE AND QUANTITY OF DANGEROUS GOODS

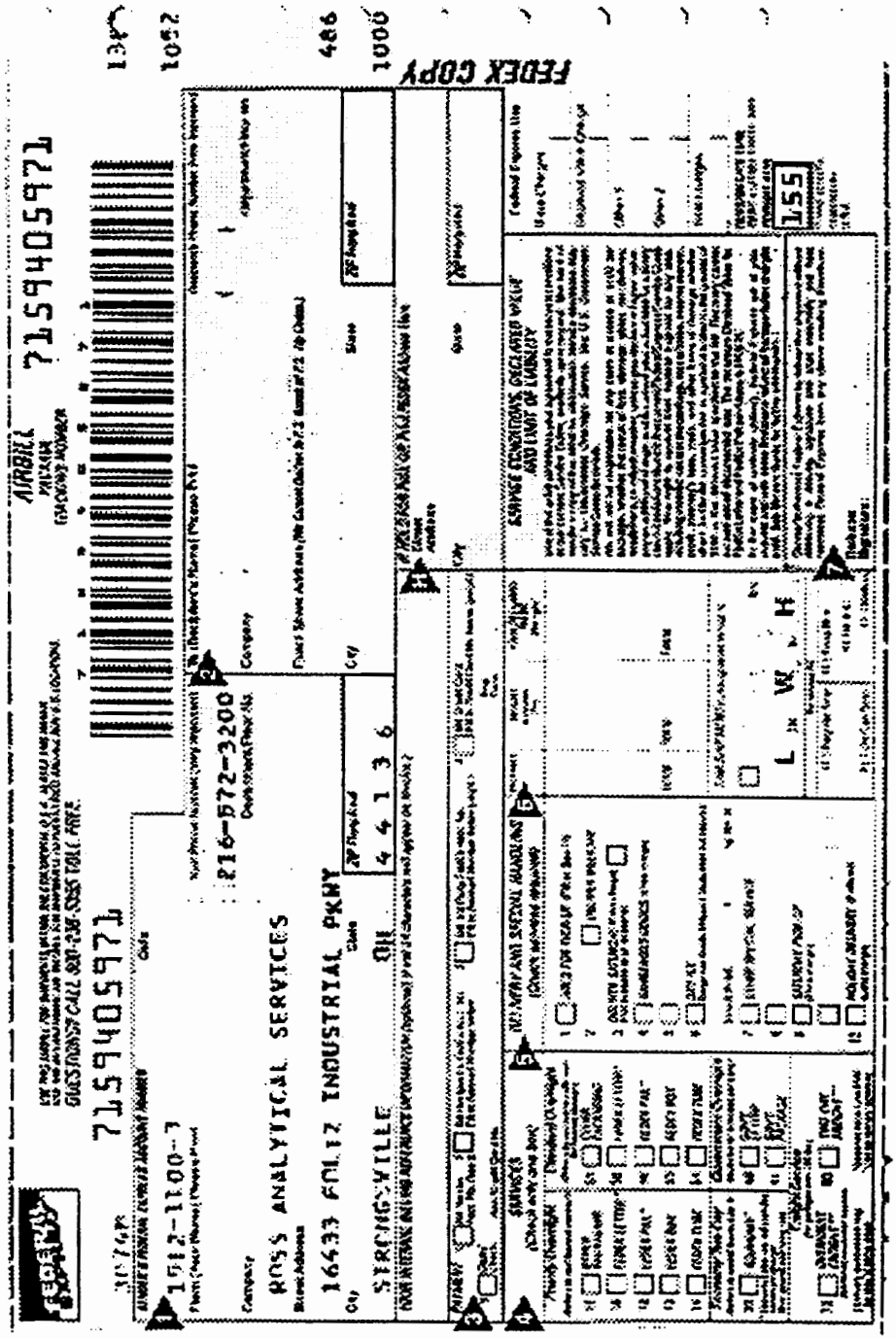
Dangerous Goods Identification						
Proper Shipping Name	Class or Division	UN or ID No.	Subsidiary Risk	Quantity and type of packing	Packing Inst.	Authorization
Nitric Acid (35%)	8	UN2031		30ml	813 III	
Sulfuric Acid (35%)	8	UN1830		30ml	813 II	
Sodium Hydroxide Solution (25%)	8	UN1824		30ml	813 II	
Hydrochloric Acid (35%)	8	UN1789		30ml Packed in 1 Fiberboard Box - 9 x 9 x 9	813 II	

Additional Handling Information

Shipper's Phone No. 1-800-924-9300

ACCEPTABLE FOR PASSENGER AIRCRAFT. THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS OR TREATMENT.

APPENDIX F



7159405971

7159405971

APPENDIX G

SHIPPER'S COPY

UPS Next Day Air®
UPS Worldwide Express
Shipping Document

See instructions on back. For additional information, call 1-800-PICK-UPS (800-742-5877).

TRACKING NUMBER 0745 3547 540

1 SHIPMENT FROM

SHIPPER'S UPS ACCOUNT NO. 426986 | UPS SHIPPER NO./UPS BILLING NO. | FOR UPS USE

REFERENCE NUMBER

NAME | TELEPHONE 216-572-3200

COMPANY ROSS ANALYTICAL SERVICE

STREET ADDRESS 16433 FOLTZ INDUSTRIAL

CITY STRONGSVILLE | STATE OH | ZIP CODE 44136

2 EXTREMELY URGENT DELIVERY TO

NAME | TELEPHONE

COMPANY

STREET ADDRESS | DEPT./FLOOR

CITY | STATE | ZIP CODE

3 WEIGHT AND ZONE — WEIGHT | DIMENSIONAL WEIGHT | ZONE 022

4 TYPE OF SERVICE — [X] NEXT DAY AIR [ ] WORLDWIDE EXPRESS (INTERNATIONAL) [ ] DOCUMENTS ONLY — CHARGES $

5 OPTIONAL SERVICES — [ ] SATURDAY PICKUP [ ] SATURDAY DELIVERY $ [ ] DECLARED VALUE AMOUNT $ $ [ ] C.O.D. AMOUNT $ $

6 ADDITIONAL HANDLING CHARGE — [ ] An Additional Handling Charge applies for certain items. See instructions. $

TOTAL CHARGES $

7 METHOD OF PAYMENT — [X] BILL SHIPPER [ ] BILL RECEIVER [ ] BILL THIRD PARTY [ ] AMERICAN EXPRESS [ ] MASTER CARD [ ] VISA [ ] CHECK [ ] UPS USE CARD

8 RECEIVER'S/THIRD PARTY'S UPS ACCOUNT NO. OR MAJOR CREDIT CARD NO. | EXPIRATION DATE

THIRD PARTY'S COMPANY NAME

STREET ADDRESS

CITY | STATE | ZIP CODE

9 SHIPPER'S SIGNATURE X | DATE

910191120 2/94 U

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: SC-012-00
Initial Date: 3/4/97
Page 1 of 21
Date Revised: NA

Title: Operation of the Infrared Thermometer and Measuring Sample Temperatures			
Prepared by:	Approved by:	Date: 3/5/97	QA Approval:

1.0 PURPOSE

This SOP describes the operating procedure for the C-1600 infrared thermometer to be used for measuring sample temperatures.

2.0 APPLICATION

This procedure applies to all samples received in Sample Control for analysis by the laboratory.

3.0 REFERENCES

C-1600 Series User Manual

4.0 ASSOCIATED SOPs

SC-002, Sample Receipt, Log-in and Storage for Non-CLP Samples

SC-006, Sample Receipt, Log-in and Storage for CLP Samples

5.0 PROCEDURE

5.1 Measuring Temperatures

5.1.1 Turn on the unit using the switch located on the left side of the display unit. Flip the switch up to measure in Celsius and down to measure in Fahrenheit.

5.1.2 Immediately upon sample receipt, remove the container lid and hold the probe of the unit just above the top of the sample. **Do not let the probe touch the sample.** Read the temperature of the sample in degrees Celsius. This procedure is applicable to samples for all analyses **except volatiles**.

5.1.3 For sample shipments containing samples **for volatiles analyses only**, the temperature of the cooler must be taken. The temperature of the cooler is measured by holding the probe of the unit inside the cooler immediately upon opening.

5.1.4 If there are greater than 10 samples in a container, take a random 25% selection of samples and measure their respective temperatures. For CLP, measure the temperature blank of the cooler, if one is contained.

5.1.5 Upon measuring the temperature of the samples, record the temperature on the chain-of-custody, and on the sample log-in checklist.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: B/N/A Extraction of Water Samples Using SW-846 Methods 3510B, 3520B			
Prepared by: BES	Approved by:	Date: 10/24/96	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to describe the method used to extract B/N/A compounds from aqueous samples.

2.0 APPLICATION

This method applies to aqueous samples requiring extraction for BNAs including TCLP leachates, but excluding samples requiring CLP protocols, EPA "600" series analyses, and EPA "500" series analyses.

3.0 REFERENCES

"Separatory Funnel Liquid-Liquid Extraction", Test Methods for Evaluating Solid Waste (SW-846), Method 3510B, September, 1994

"Continuous Liquid-Liquid Extraction", Test Methods for Evaluating Solid Waste (SW-846), Method 3520B, September, 1994

"Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Test Methods for Evaluating Solid Waste (SW-846), Method 8270B, September, 1994

4.0 ASSOCIATED SOPs

OP-004, GPC Operating Procedure

OP-037, Solvent Extract Waste Disposal in the Organic Preparation Laboratory

OP-039, Wastewater Disposal in the Organic Preparation Laboratory

GX-001, Organic Standards Preparation

5.0 PROCEDURE

5.1 Reagents

5.1.1 Reagent water, LabConco or equivalent

5.1.2 Acetone - Pesticide grade or equivalent

5.1.3 Glass wool

5.1.4 pH paper

5.1.5 Sodium hydroxide solution (10N) - dissolve 40 g of sodium hydroxide in reagent water and dilute to 100 mL. Store in Teflon containers.

5.1.6 Sulfuric acid solution (1:1) - slowly add 500 mL of concentrated sulfuric acid to 500 mL of reagent water (Use caution!)

5.1.7 Methylene chloride - Pesticide grade or equivalent

5.1.8 Sodium sulfate (anhydrous, granular) baked at 400°C for 4 hours prior to use

5.1.9 BNA spike solutions as prepared by GC/MS. Surrogate and spiking solutions are prepared at concentrations of 200 ug/mL for acids and 100 ug/mL for base neutral compounds in methylene chloride. See Section 10.0, Tables, for a list of the acid and base neutral surrogate and matrix spiking compounds.

5.1.10 Teflon boiling chips washed in a Soxhlet for 8 hours using methylene chloride.

5.2 Equipment

For Continuous Extraction

5.2.1 Continuous extractor and condenser

5.2.2 24/40 ball flask, 500 mL

5.2.3 Stirring rod

For Separatory Funnel Extraction

5.2.4 Erlenmeyer, 500 mL

5.2.5 Separatory funnel

For Extraction

5.2.6 Funnel

5.2.7 Concentrator: Kuderna-Danish

5.2.8 Snyder column

5.2.9 Water steam bath

5.2.10 Nitrogen blow down apparatus

5.2.11 Disposable pipet

5.2.12 Calibrated 2 mL amber autosampler vials. Vials are calibrated using a 1 mL syringe. (1 mL of methylene chloride is added to the vial and the meniscus is marked with a permanent marker. The methylene chloride is then discarded.)

5.2.13 Centrifuge

5.2.14 Syringe

6.0 PROCEDURE

6.1 Extraction Procedure for Separatory Funnel

Acid Extraction

6.1.1 If the extraction must be completed quickly (less than one day) or the sample volume is less than 500 mL, then continue to follow the method below, otherwise, go to the Continuous Extraction Method, Section 6.2.

6.1.2 Assemble appropriate glassware and rinse with extraction solvent (methylene chloride). Assemble a short stem funnel with anhydrous sodium sulfate retained with a glass wool plug and solvent wash.

6.1.3 Choose a separatory funnel of appropriate size. For example, for a one liter extraction use a 2 liter separatory funnel.

6.1.4 Measure out 1000 mL of sample and add it to the separatory funnel. Also pour out a small aliquot into the sample cap. Measure and record the pH of the sample in the sample cap with pH paper and then discard. Prepare two blanks per 20 or fewer samples in a set, using reagent water. If the entire sample is used, rinse the sample container with methylene chloride and add to the separatory funnel.

6.1.5 Spike 0.5 mL of surrogate into each sample, matrix spike and blank. Spike 0.5 mL of the base neutral acid spike into each of the MS/MSD sample aliquots and LCS.

6.1.6 Adjust the samples, blanks, matrix spike, and LCS spikes to a pH lower than 2 with 1:1 H_2SO_4 (use pH paper).

6.1.7 Add 100 mL of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for 3 minutes. Vent the funnel several times during the three minutes to relieve pressure. Allow the solvent water layers to separate for 10 minutes. Drain the solvent into a 500 mL Erlenmeyer flask which has been labeled with green tape to indicate that it is the base neutral fraction.

Separation of Emulsions

Option 1 If the layers do not separate and time permits, prepare a continuous extractor and transfer the entire contents of the separatory funnel to the continuous extraction method.

Option 2 If the layers do not separate, try to break the emulsion with a stirring rod. If the layers still do not separate, pour the emulsion into a centrifuge tube and separate the layers be centrifuged. Add the water back to the separatory funnel and transfer the solvent to the labeled Erlenmeyer flask. Proceed to extract the sample two more additional times by this method. When finished, thoroughly rinse the centrifuge flask with solvent and add to the Erlenmeyer flask.

Option 3 Make a saturated solution of sodium sulfate in deionized water, pour approximately 300 mL of this solution into the separatory funnel and stir with a glass rod.

6.1.8 Repeat the extraction two more times after breaking up emulsions (if present) using 60 mL of methylene chloride. Add the solvent layer to the labeled Erlenmeyer flask after each extraction. Cover with foil and set aside the base neutral fraction for concentration.

Basic Extraction

6.1.9 Make samples, blanks, matrix spike and LCS basic in the separatory funnel using 10 N sodium hydroxide to a pH of 12 measured with pH paper.

6.1.10 Add 100 mL of methylene chloride to the separatory funnel and shake the funnel for 3 minutes. Vent the funnel several times during the three minutes to relieve pressure. Allow the solvent/water layers to separate for 10 minutes and then drain the solvent into a 500 mL Erlenmeyer which has been labeled with red label tape to indicate that it contains the acid fraction.

For separation of emulsions, see Options 1 through 3 above.

6.1.11 Extract the sample two more times with 60 mL of methylene chloride and add the solvent to the Erlenmeyer containing the acid fraction after each extraction. This sample is now ready for concentration.

6.2 Continuous Extraction

For **Method 8270 non-extended list**, continuous extraction is performed on the acid side only. RASI has demonstrated the CLP method of extraction (acid side only) produces recoveries for the compounds of interest on this target list which are equivalent to recoveries produced using both acid side followed by base neutral side extractions. **The test code for acid only BNA extraction is 3520AO.**

Acid Extraction

6.2.1 Rinse all continuous extractor parts with methylene chloride. Rinse all condenser bases with acetone.

6.2.2 Add 300 mL of methylene chloride to the base of the continuous extractor.

6.2.3 Add a few boiling chips to a 500 mL boiling flask and affix it to the extractor.

6.2.4 Label the extractor with green and red label tape. The base neutral (green fraction) is extracted second, if required. Add one liter of sample to the continuous extractor. If a smaller sample volume is being used add reagent water to the continuous extractor by pouring slowly into top of extractor. Bring to one liter or high enough to ensure proper flow in the extractor.

If the entire sample is used, rinse the bottle with approximately 40 mL of methylene chloride and dump rinsate into extractor.

6.2.5 Pour a little of the sample into the sample cap. Measure and record the pH of the sample in the sample cap with pH paper and then discard.

6.2.6 Spike 0.5 mL of surrogate into each sample, matrix spike and blank. Spike 0.5 mL of the base neutral acid spike solution into each the MS/MSD samples and LCS.

6.2.7 Adjust the pH of the samples, blanks, and matrix spike to less than 2 with 1:1 H_2SO_4 (measure the pH with pH paper).

6.2.8 Connect the condenser to the continuous extractor. Turn the cooling water on. Extract the samples for 18-24 hours.

IF BASIC EXTRACTION IS REQUIRED, PROCEED WITH THE FOLLOWING BASIC EXTRACTION

Basic Extraction

6.2.9 After 18-24 hours, pour 250 mL of methylene chloride and a few boiling chips into another ball flask which has been solvent rinsed. Affix the green label to the base neutral extract flask, remove this flask and swap it with the new solvent-filled flask.

6.2.10 Remove the condenser and adjust the pH of all the samples, blanks, and matrix spike to pH of 12 using 10 N sodium hydroxide (measured with pH paper). Mix with a glass rod.

6.2.11 Reconnect the condenser and extract the samples for another 18-24 hours.

6.2.12 After 18 - 24 hours: Remove ball flask and wash joint into flask with 2 to 3 mLs of solvent. Affix red label to this ball flask containing the acid extract.

6.3 Sample Concentration by Kuderna-Danish (KD)

6.3.1 Place a funnel on top of a KD extractor and add a glass wool plug in the funnel. Pour enough sodium sulfate to fill funnel at least half full. Rinse this whole apparatus by pouring approximately 40 mL methylene chloride through the sodium sulfate funnel. Swish the solvent around sides of KD, then dump out this rinse. Pour first the acid extract, then the basic (if extracted) extract through the funnel. Rinse ball flasks with methylene chloride into funnel. Rinse the sodium sulfate funnel into KD using a 30 mL methylene chloride rinse. Remove funnel, then add two boiling chips to the KD.

6.3.2 With a three ball Snyder column, evaporate the extract down to about 5 mL on a hot water bath. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. There will be about a 10 mL volume after the KD cools.

6.3.3 After the KD has cooled, wipe the outside moisture off with a Kimwipe. Remove the receiver and rinse the joint with methylene chloride into the receiver.

6.3.4 Place the receiver on the N-EVAP and gently blow down the extract with nitrogen to about 1 mL.

6.3.5 Calibrate a 2.0 mL vial to 1.0 mL with methylene chloride. Transfer the contents of the receiver to the labeled vial and continue to blow down the extract. Do not reduce the volume to <0.2 mL. Rinse the receiver three times with methylene chloride and add each rinse to the vial. Reduce the volume in the vial to the 1.0 mL mark using a nitrogen micro blower. Cap the vials and assemble them in an extract tray. Deliver to Sample Control and store them in the extract refrigerator.

6.4 Clean up extracts by Gel Permeation Chromatography (GPC) if the extract is oily or very dark. See SOP OP-004, GPC Operating Procedure.

6.5 Extracts containing sediment can be cleaned by diluting extract to 5 mL then passing through a 1 micron syringe filter. Rinse some methylene chloride through the syringe following the sample then blow extract down to 1 mL again.

7.0 QUALITY CONTROL

7.1 Extract two blanks with each sample batch or for every 20 samples extracted, whichever is more frequent, or . client specifies otherwise. Once a batch has been completed discard one blank. Send only one blank with each batch to GC/MS.

7.2 Extract a matrix spike/matrix spike duplicate pair for every 20 samples, or one pair every two weeks, whichever is more frequent, or if client specifies otherwise.

7.3 Prepare a laboratory control sample (LCS) with every batch.

7.4 The holding time for extraction of BNAs from waters as specified in SW-846, Method 8270, is 7 days from date of sample collection.

8.0 INTERFERENCES

8.1 Care must be taken to avoid latex glove contact with sample, solvent, or interior of extraction glassware. Phthalate contamination can be introduced by plastics. Avoid any plastic when handling samples or extracts.

8.2 Avoid scratched KD receiver tubes as they tend to inhibit good surrogate recovery.

9.0 TROUBLESHOOTING

9.1 Frequently a sample requiring a MS/MSD per client request will arrive with insufficient volume to accommodate MS/MSD extraction. Usually the recommended process is to: Use one liter for the sample, then only 500 mL for the MS and MSD. Half the normal surrogate (0.5 mL) and half the normal spike (0.5 mL) would then be added to the MS and MSD. The final concentration of both the MS/MSD would be taken to 0.5 mL. This will keep the surrogate and spike to final volume ratio consistent.

9.2 Extracts which do not blow down to the specified 1 mL final volume will require a dilution. A dilution of 5 mL final volume is usually sufficient to decrease possible viscosity or compounds from precipitating out of solution is required. 5 mL final volume is usually sufficient.

NOTE: Extracts requiring such dilutions should receive GPC clean-up prior to attempting final vialing of the extract. Some extracts will not concentrate to 0.5 mL even after GPC clean up. No choice exists except to dilute.

9.2.1 Ensure that dilutions or GPC clean up is noted on the extraction summary and on a narrative form..

10.0 TABLE(s)

10.1 Surrogate Table

Base/Neutral Compounds	Final Concentration
2-Fluorobiphenyl	50 ug/mL
Nitrobenzene-d5	50 ug/mL
Terphenyl-d14	50 ug/mL

Acid Compounds	Final Concentration
2-Fluorophenol	100 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Phenol-d6	100 ug/mL

10.2 Matrix Spiking Table

Base/Neutral Compounds	Final Concentration
1,2,4-Trichlorobenzene	50 ug/mL
Acenaphthene	50 ug/mL
2,4-Dinitrotoluene	50 ug/mL
Pyrene	50 ug/mL
N-Nitroso-di-n-propylamine	50 ug/mL
1,4-Dichlorobenzene	50 ug/mL

Acid Compounds	Final Concentration
Pentachlorophenol	100 ug/mL
Phenol	100 ug/mL
2-Chlorophenol	100 ug/mL
4-Chloro-3-methylphenol	100 ug/mL
4-Nitrophenol	100 ug/mL

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Pesticide and PCB Extraction Using SW-846, Methods 3510B; 3520B			
Prepared by: BES	Approved by: CK	Date: 12/9/94	QA Approval:

1.0 PURPOSE

This procedure describes the method used to extract pesticides and PCBs from water samples.

2.0 APPLICATION AND SCOPE

This procedure applies to water samples to be extracted for Pesticides and PCBs (Non-CLP). This procedure is to be used when samples are to be analyzed by SW-846, Method 8080 or 8081. This procedure is not to be used for Method 608 sample preparation. Information about interferences can be found in Section 9.0.

3.0 REFERENCES

EPA SW-846 Method 3510B; 3520B

4.0 ASSOCIATED SOPs

SOP No. OP-004, GPC Operating Procedure
SOP No. OP-037, Solvent Extract Waste Disposal in the Organic Preparation Lab
SOP No. OP-039, Wastewater Disposal in the Organic Preparation Lab
SOP No. GX-001, Organic Standards Preparation

5.0 EQUIPMENT

Separatory Funnel Extraction

5.1 Separatory funnel, appropriate size

5.2 500 mL Erlenmeyer flask

Continuous Extraction

5.3 Glass stirring rod

5.4 Continuous extractor and condenser

5.5 500 mL 24/40 joint ball flask

Either Extractions

5.6 Concentrator: Kuderna-Danish

5.7 10 mL calibrated receiver with reservoir tip

5.8 Snyder column

5.9 Florisil chromatography column.

5.10 Hot water bath

5.11 Nitrogen concentration apparatus

5.12 Vial, 10 mL calibrated to 5.0 mL with hexane

5.13 Centrifuge

5.14 Teflon-lined caps for 10 mL vials

5.15 Auto sampler vials - 2 mL with Teflon-lined screw caps

6.0 REAGENTS/SOLUTIONS

6.1 Methylene chloride, Pesticide grade or equivalent

6.2 Boiling chips

6.3 Hexane, Non-Spectra grade

6.4 Sodium sulfate, anhydrous, baked at 450°C for 4 hours prior to use

6.5 Florisil - 60/100 mesh

6.6 Sulfuric acid (1:1 vol:vol) - slowly add 500 mL of concentrated sulfuric acid to 500 mL of reagent water.

6.7 10 N Sodium hydroxide - dissolve 40 g of sodium hydroxide in reagent water and dilute to 100 mL.

6.8 Reagent water, LabConco

6.9 Glass wool

6.10 Surrogate spike mixture, (See Table 1)

6.11 Pesticide and PCB matrix spike solution, (See Table 2)

7.0 EXTRACTION PROCEDURES

If the extraction must be completed quickly (less than two days) or the sample volume which will be extracted is less than 500 mL, follow the Separatory Funnel Extraction Method; otherwise follow Continuous Extraction Method.

7.1 Separatory Funnel Extraction

7.1.1 Choose a separatory funnel of appropriate size. For example, for a one liter extraction use a 2 liter separatory funnel.

7.1.2 Assemble all glassware and rinse with extraction solvent (methylene chloride). Assemble a short stem funnel with anhydrous sodium sulfate retained with a glass wool plug and solvent wash.

7.1.3 Measure out the sample volume and add it to the separatory funnel. Also, pour out a small aliquot of sample into the sample cap. Measure and record the pH of the sample in the sample cap with pH paper and then discard. If the pH is <5 or >9, adjust the pH to between 5 and 9 with either 1:1 sulfuric acid or 10 N sodium hydroxide. Prepare two blanks per 20 or fewer samples in a set, using reagent water.

7.1.4 Spike 1 mL of the matrix spike solution into each of the MS, MSD, and LCS sample aliquots. Spike 1 mL of surrogate mixture into all extractions including the blanks.

7.1.5 Add 60 mL of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for 2 minutes. Vent the funnel several times during the two minutes to relieve pressure. Allow the solvent: water layers to separate for 10 minutes. Collect the solvent in a 500 mL Erlenmeyer flask which has been properly labeled with white label tape.

NOTE: If an emulsion forms, use the below options to try to separate the layers.

Option 1 If time permits, prepare a continuous extractor and transfer the entire contents of the separatory funnel to the continuous extractor. Proceed with the continuous extraction method.

Option 2 Try to break the emulsion using a glass stirring rod. If the layers still do not separate, pour the emulsion into a centrifuge tube and separate the layers by centrifuge. Add the water back to the separatory funnel and transfer the solvent to the labeled Erlenmeyer flask. When finished, thoroughly rinse the centrifuge tube with solvent and add to the Erlenmeyer flask.

Option 3 Make a saturated solution of sodium sulfate in deionized water, pour approximately 300 mL of this solution into the separatory funnel, and stir with a glass rod.

7.1.6 Repeat the extraction two more times after breaking up the emulsion (if present) using 60 mL of methylene chloride. Add the solvent layer to the labeled Erlenmeyer flask after each extraction. Rinse the Na_2SO_4 in the funnel with about 20 mL of methylene chloride. Cover the extracts with foil.

7.1.7 Rinse sodium sulfate and funnel with 20-30 mL methylene chloride after final extraction.

7.2 Continuous Extraction

7.2.1 Assemble all glassware and rinse all continuous extractor parts with methylene chloride. Rinse all condenser bases with acetone.

7.2.2 Add a few boiling chips to a 500 mL boiling flask and affix it to the extractor.

7.2.3 Add 300 mL of methylene chloride to the base of the continuous extractor.

7.2.4 Label the extractor with white label tape. Add one liter of sample to the continuous extractor. If a smaller sample volume is being used, add reagent water to a total volume of approximately one liter to the continuous extractor by pouring slowly into the top of the extractor.

7.2.5 Pour a little of the sample into the sample cap. Measure and record the pH of the sample in the cap with pH paper and then discard.

7.2.6 If the pH <5 or >9, adjust the pH with either 10 N sodium hydroxide or 1:1 sulfuric acid to between 5 and 9.

7.2.7 Spike 1.0 mL of surrogate into each sample, matrix spike, LCS, and blank. Spike 1 mL of the matrix spike solution into each of the MS, MSD, and LCS sample aliquots.

7.2.8 Connect the condenser to the continuous extractor. Turn the cooling water on. Extract the samples for 18-24 hours.

7.3 Sample Concentration by Kuderna-Danish (KD)

7.3.1 Place a funnel on top of a KD extractor, add a glasswool plug in the funnel. Pour enough sodium sulfate to fill the funnel just below half full. Rinse this whole apparatus by pouring approximately 40 mL of methylene chloride through the sodium sulfate funnel. Swish the solvent around the sides of the KD, then dump out this rinse. Add the extracts, then rinse the ball flasks with methylene chloride directly into the funnel. Rinse the sodium sulfate funnel into the KD, using a 30 mL methylene chloride rinse. Remove funnel then add two boiling chips.

7.3.2 Attach a 3 ball Snyder column. Evaporate the extract down to about 5 mL on a hot water bath. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. Immediately add 25 mL of hexane through the Snyder column and continue to concentrate to about 8 mL. Add a second 25 mL portion of hexane and concentrate to about 8 mL.

Note: If during concentration the extract appears colored or viscous do not perform a hexane exchange since these colored extracts will be run through the GPC for clean-up. (See SOP OP-004, GPC Operating Procedure.)

7.3.3 After the KD has cooled, wipe the outside moisture off with a Kimwipe. Remove the receiver and rinse the joint with hexane into the receiver.

7.3.4 Place the receiver on the N-Evap and gently blow down the extract with nitrogen to about 3 mL.

7.3.5 Transfer the extract to a culture tube calibrated to 10 mL. Complete the transfer with three 1 mL rinses of hexane. Bring the extract to 10 mL final volume with hexane. (Extracts run through the GPC will go to 1/2 of their normal final volume or 5 mL.)

7.3.6 Florisil clean-up is not usually required on waters. If extracts are distinctly colored, proceed with SOP OP-006, Florisil Pasteur Pipet Column Clean-up for CLP and Non-CLP Pesticide and PCB Samples.

7.3.7 GPC-cleaned extracts are KD'ed following GPC and solvent exchanged as described above. Extracts run through GPC <u>do not</u> need to be dried with sodium sulfate prior to KD'ing.

7.3.8 If 1 mL of extract is used in the Florisil clean-up, the extract must be concentrated to 1 mL final volume on the N-Evap.

7.3.9 Cap the vials and assemble them in an extract tray. Deliver to Sample Control storing them in the extract refrigerator.

8.0 QUALITY CONTROL

8.1 Extract two blanks with each sample batch or for every 20 samples extracted, whichever is more frequent, or if client specifies. Note the numbers of both blanks which you prepare on the extraction summary sheet. Retain one blank in the OP lab except under the following conditions:

1. If one of the preparation blanks gets misplaced or lost, then supply the second blank to the analytical group.

2. If GPC is needed, one of the blanks should be sent through the GPC.

The second blank is not to be provided to the analytical groups if one surrogates fails or contamination is found in the one blank. Either of these occurrences requires reextraction of the batch.

8.2 Extract a matrix spike and matrix spike duplicate for every 20 samples or every two weeks, which ever is more frequent, or if a client specifies otherwise.

8.3 Run a laboratory control sample (LCS) with every batch.

8.4 Holding times for pesticides in waters, as specified in SW-846, Method 8080, is 7 days from sample collection.

9.0 INTERFERENCES

Florisil must be rinsed with at least 6 mL of hexane/acetone during the Florisil process. Florisil shipped from manufacturers may already be "very active" so no additional baking will be necessary. This should be determined by performing Florisil lot checks on each new shipment.

10.0 TROUBLESHOOTING

Frequently, a sample requiring an MS/MSD pair per client request will arrive with insufficient volume to accommodate the extraction. Usually the recommended process is to use one liter of sample for the sample extraction, and only 500 mL for the MS and MSD. Half of the normal surrogate (0.5 mL) and half of the normal spike (0.5 mL) would then be added to the MS and MSD. Final concentration of the MS and MSD pair would be taken to 5 mL. This will keep the surrogate and spike final volume ratio consistent.

Otherwise, another sample from the same client is selected.

11.0 TABLES

Table 1
Concentrations of the Pesticide and PCB surrogate spikes used in the extraction process.

ANALYSIS	Sample Weight or Volume	Final Volume	Concentration of surrogate	Amount added
PEST/PCB (8080 or 8081)				
Waters	1L	5 mLs		
SURROGATES				
TCMX			0.20 ug/mL	1.0 mL
DCB			0.20 ug/mL	

Table 2
Concentration of the Pesticide and PCB spiking solutions used in the extraction of LCSs and matrix spikes. All spikes made up in acetone.

ANALYSIS	Sample Weight or Volume	Final Volume	Concentration of spike	Amount added
PEST/PCB (8080 or 8081)				
Waters	1L	5 mLs		
TARGETS				
Lindane (g-BHC)			0.50 ug/mL	
Heptachlor			0.50 ug/mL	
Aldrin	MIX		0.50 ug/mL	
Dieldrin			1.00 ug/mL	1.0 mL
Endrin			1.00 ug/mL	
4,4'-DDT			1.00 ug/mL	
TCLP PEST	0.200L	5 mLs		
TARGETS				
Lindane (g-BHC)			0.20 ug/mL	
Heptachlor			0.20 ug/mL	
Heptachlor Epoxide	MIX		0.20 ug/mL	1.0 mL
Endrin			0.20 ug/mL	
Methoxychlor			2.00 ug/mL	
Toxaphene	0.200L	5 mLs	5.0 ug/mL	1.0 mL
Tech. Chlordane	0.200L	5 mLs	5.0 ug/mL	1.0 mL
PCBs (8080 or 8081)				
Waters				
1260	1L	5 mLs	2.00 ug/mL	1.0 mL

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: OP-003-01
Initial Date: 2/1/91

Date Revised: 4/15/96

Title:	Organic Labware Cleaning						
Prepared by:	BES	Approved by:	[illegible]	Date:	4/16/96	QA Approval:	[illegible]

1.0 PURPOSE

To describe the procedure for the cleaning and preparation of labware used in the organic laboratory.

2.0 APPLICATION

This procedure applies to labware used for all organic analyses, including ZHE.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Remove surface residuals immediately after use by rinsing glassware with deionized water.

5.2 Wash glassware in a hot solution of cleaning reagent (liquid Alconex, Micro or equivalent) and tap water. If necessary, remove any marker with acetone. Use scrub pad to remove residue.

5.3 Rinse with hot tap water to flush away floated residue.

5.4 Rinse with deionized water to remove metallic deposits from the tap water.

5.5 Rinse with methanol (distilled in glass grade). DO NOT rinse microdistillation glassware with methanol.

5.6 Air dry or oven dry prior to storing.

5.7 Rinse the item immediately before use with some of the same solvent that will be used in the analysis.

NOTE: For labware to be used for ZHE analysis, omit the solvent rinsing (Steps 5.5 and 5.7).

6.0 QUALITY CONTROL

Glassware cleanliness will be demonstrated by the cleanliness of the method blanks.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: AD-012-01
Initial Date: 2/1/91
Date Revised: 6/28/94

Title: Preparation of Reports to Clients			
Prepared by: CHeldwell	Approved by: CHeldwell	Date: 6/28/94	QC Approval:

1.0 PURPOSE

This SOP provides guidelines for preparing RASI Level I, II, and III reports in a uniform manner.

2.0 APPLICABILITY

This SOP applies to reports of analyses not performed by CLP protocol.

3.0 REFERENCES

None

4.0 RELATED SOP's

None

5.0 GENERAL PROCEDURE

5.1 Regardless of the level of the report to be submitted, each Group Leader is responsible for ensuring that all of the necessary records relating to his or her group's work is present and correct before submitting it to the Reporting Department. Blocks of data may be submitted before all work of a given group or department is complete. Each block should, however, be the complete record of all the tests submitted.

5.2 Large raw data packages which have data from several work orders need not be copied and included in the file of every work order. Examples are GC and GC/MS calibrations and ICP instrument outputs. If raw data are not included, the data summary must prominently state where they are filed, by client code and work order number.

5.3 Data are generally entered into SAM by the Reporting Department. Selected test codes are set up so only raw data need be entered; most others require that results be calculated to their final value before submission.

5.4 Data from a subcontractor are always identified as being from the subcontractor. If sub data are a minor part of the full job, for instance one or two wet tests, they may be included in the body of the SAM output. If they are a significant fraction of the job, for instance dioxins, the entire subcontractor's report is included with the RASI report.

5.5 The Reporting Department performs an overall review of the data to eliminate inconsistencies between departments' results.

5.6 A senior member of the Reporting Department proofreads the results in SAM, either on draft printouts or on screen, and makes any corrections and format changes. He or she writes a narrative "Report Comments" section to point out or explain deviations from normal practice or presentation.

5.7 The report is printed from SAM and assembled with any necessary supporting documents, signed, copied, and the original sent to the client. Sections 6.0, 7.0, and 8.0 below describe the contents of each level of report commonly provided by RASI. The copy of the report is filed with the raw data.

5.8 Data packages are filed alphabetically by client code. Within a given client code, packages are filed by work order number. Very large or continuing projects are often filed directly in bankers' boxes. Smaller packages are filed in filing cabinets.

5.9 The usual order for documents in the file is listed in Table 1. Variations are made as necessary.

6.0 RASI LEVEL III REPORT

6.1 This is the simplest report, a Certificate of Analysis. It contains the following items in this order:

Cover Page
Report Comments, if applicable
Test Methodologies page(s)
Analytical results
Chain of custody

6.2 In addition to the basic Certificate of Analysis, some clients receive data for organic blanks. These are prepared through a combination of SAM and the DOS editor. They are inserted after the analytical results.

7.0 RASI LEVEL II REPORT

7.1 The RASI Level II report is a Certificate of Analysis with a Quality Control section which is inserted after the Report Comments. The QC section contents are variable depending on the client's expectations. The section can include any of LCS recoveries, MS/MSD results, duplicate analysis results, and similar data.

8.0 RASI LEVEL I REPORT

8.1 The RASI Level I report is a "CLP-style" data package for analyses performed by other than CLP protocols. (CLP protocol data review and reporting are addressed in other SOP's.) A Level I report follows, as closely as practical, the inorganic and organic CLP SOW's. Instrument logs, internal chain of custody forms, and Forms DC-1 and DC-2 are normally not part of a Level I report.

8.2 The wet chemistry section of the report includes a summary of all QC (spikes, duplicates, LCS's) performed, the analytical results, and the raw data. TRPH, TOC, and TOX results are reported like the wet chemistry.

8.3 The metals section of the report includes analytical results (from SAM or on EPA Form 1's as appropriate); EPA Forms 2A, 2B, 3, 4, 5A (if MS's were performed), 6 (if duplicates were performed), 7, 10, 11A, 11B, 12, 13, and 14; and raw data. They are arranged as in the inorganic SOW.

8.4 The sections for volatiles and semivolatiles by GC/MS include analytical results (from SAM or on EPA Form 1's as appropriate); EPA Forms 2, 3 (if MS/MSD's were performed), 4, 5, 6, 7, and 8; and raw data. They are arranged as in the organic SOW.

8.5 The sections for GC analyses mimic the GC/MS sections rather than the CLP SOW pesticide/PCB section. They include analytical results from SAM; forms similar to EPA Forms 2, 3, 4, 6, and 7; and raw data. They are arranged as in the GC/MS sections of the organic SOW.

8.6 The report packages are sequentially numbered before copying. The original of the signed cover page and copies of all other pages are sent to the client. RASI retains the copy of the cover page and the originals of the other report pages and the raw data.

TABLE 1

DATA FILE ORDER

1.0 Copy of the final report, not stapled or bound.

2.0 Originals of Request for Analysis and Chain of Custody forms, airbills, and any other paperwork which accompanied the samples.

3.0 Copy of the work order.

4.0 Instrument printouts, bench sheets, etc., in the following general order. If there are multiple runs for a given test code, arrange them chronologically.

Wet Chemistry data (usually alphabetical by test)
Metals data (Hg, GFAA alphabetically, ICP)
TRPH
Gas Chromatography/Mass Spectrometry data
- Volatiles
- Semivolatiles

Gas Chromatography data
- Volatiles (purge and trap)
- Alcohols
- Organochlorine pesticides and/or PCB's
- Herbicides
- Organophosphorus pesticides
- Other

ROSS	SOP Standard Operating Procedure	SOP No: AD-014-01 Initial Date: 2/1/91 Page 1 of 6 Date Revised: 10/7/91

Title:

Preventive Maintenance in the Wet Chemistry Laboratory

Prepared by:	Approved by:	Date:	QC Approval:
Peggy J Bo	[illegible]	10/7/91	[illegible]

1.0 PURPOSE

This SOP describes the maintenance procedures taken on a periodic basis to prevent avoidable breakdowns of instruments in the wet chemistry laboratory.

2.0 APPLICATION

This SOP applies to the following instruments:

- Hitachi U2000 UV/VIS Spectrophotometer with sample sipper
- Orion Model 720A pH/mV meter
- YSI Model 35 Specific Conductance Meter
- Deionized water system
- Mettler electronic balances, AE 100, AE 200, and BB 300
- HACH COD Reactor
- CEM MDS - 81D Microwave

3.0 REFERENCES

3.1 Operator's Manuals for the Hitachi U2000 UV/VIS Spectrophotometer and the Sample Sipper.

3.2 Operator's Manual for the Orion Model 720A pH/mV Meter.

3.3 Operator's Manual for the YSI model 35 Specific Conductivity Meter.

3.4 Operator's Manual for the LabConCo Water System.

3.5 Operator's Manual for Mettler AE 100, AE 200, and BB 300 Balances.

3.6 Operator's Manual for the HACH COD Reactor.

3.7 Operator's Manual for the CEM MSD - 81D Microwave

4.0 ASSOCIATED SOPs

4.1 SOP NO. QC-004-00, Daily water check

4.2 SOP NO. AD-004-00, Balance calibration

5.0 PROCEDURE

5.1 Preventive Maintenance Schedules

5.1.1 Hitachi U2000 UV/VIS Spectrophotometer

Maintenance	Frequency
Check instrument response with commercially purchased solutions	Quarterly
Check wavelength with internal wavelength scan	Quarterly
Wipe down outside of instrument	As needed
Flush lines and fill with detergent	After each use

5.1.2 Orion Model 720A pH/mV Meter

Maintenance	Frequency
Clean electrode	As needed
Wipe down outside of instrument	As needed
Check electrolyte level and electrode slope	When used

5.1.3 YSI Model 34 Specific Conductivity Meter

Maintenance	Frequency
Clean conductivity cell	Quarterly
Replatinize cell	As needed
Wipe down outside of instrument	As needed

5.1.4 Water System

Maintenance	Frequency
Change deionizing tanks	As needed

Change LabConCo cartridges	As needed
Conductivity check	Daily

5.1.5 Mettler AE 100, AE 200, and BB 300 Electronic Balances

Maintenance	Frequency
External service	Annually
95% capacity check	Quarterly
Class S weight check	Daily
Clean chamber/pan	After each use
Wipe down outside of balance	As needed

5.1.6 HACH COD Reactor

Maintenance	Frequency
Temperature check	Quarterly
Wipe down outside	As needed

5.1.7 CEM MDS - 81D Microwave

Maintenance	Frequency
Flush pressure line	After each use
Clean chamber	After each use
Wipe down outside	As needed

5.2 Documentation of Maintenance

5.2.1 Each time a maintenance check or procedure is performed it is recorded on the appropriate maintenance log sheet (Figures 1) and kept in the Maintenance Log Book.

5.2.2 When a routine check or procedure is performed, the analyst dates and initials the appropriate line on the log sheet. If a replacement is made, it is noted in the space provided and the spare parts log (Figure 2) is updated.

5.2.3 When a non-routine maintenance procedure is performed, a description is entered in the space provided as well as the date and the name of the person performing the procedure.

5.2.4 All preventive maintenance documentation is reviewed by the lab supervisor monthly.

Figure 1

Date	Instrument	Description of Maintenance	Initial	Parts Needed	Parts Ordered

Figure 2

Date	Instrument	Part and Part Number	Date Replaced	Date Taken	Initials

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date
[illegible]	5/24/92

Terminated by	Date

ROSS

Ross Analytical Services, Inc.

Standard Operating Procedure

SOP NO: AD-013-01
Initial Date 2/1/91
Page 1 of 3
Date Revised: 1/15/92

Title:

Inorganic Data Validation

Prepared by:	Approved by:	Date:	QC Approval:
Belinda Minter	[illegible]	3/3/92	[illegible]

1.0 PURPOSE

To outline the procedure used to validate the data generated in the inorganic laboratory.

2.0 APPLICATION

This procedure applies to all data generated by Classical Wet Chemistry and Inorganic Preparation, Inorganic Instrumentation and Metals Laboratories.

3.0 REFERENCES

None

4.0 ASSOCIATED SOP'S

None

5.0 PROCEDURE

5.1 All data sheets completed by the analysts are placed in a tray on the Team or Group Leader's desk to be reviewed.

5.2 The first check of all data is done on a 100% basis.

5.3 Items reviewed:

5.3.1 If the data have been calculated by hand, the calculations are also done by the reviewer and checked for accuracy.

5.3.2 If the data are calculated by computer spreadsheets or other in-house generated programs, the data are checked for the correctness of entry and for accuracy.

5.3.3 If the data are generated by an instrument data system, the data are checked for accuracy of blank corrections and dilution corrections.

5.3.4 If a standard curve is generated, the correlation coefficient and/or the hard copy graphic of the curve are reviewed. Either a linear regression or a second order polynomial algorithm is acceptable. (Nothing larger than second order polynomial equations is accepted.) When available, a correlation coefficient of 0.995 or better is acceptable. Any coefficient <0.990 is unacceptable and the analysis must be redone.

5.3.5 QC sample results are reviewed against established acceptance criteria.

5.4 If random error is detected, the reviewer draws a line through the incorrect data and writes in the correct value and initials and dates it.

5.5 If a systematic error is detected in the data, a Nonconformance Memo is issued and the data are returned to the analyst for corrections.

5.6 If an error in methodology or calibration is discovered, or if QC data do not meet accepted criteria, a Nonconformance Memo is initiated and the analysis is rescheduled.

5.7 As the data are checked, the reviewer places a RED check (✓) mark next to each data item that is correct or at the bottom of the corrected column.

5.8 After all the entries on the data set have been checked, the reviewer initials and dates the data sheet and then it is submitted to the Group Leader for a second data review.

5.9 The Group Leader gives all data a second check on a 10% basis.

5.10 If an error is detected, the supervisor can either enter the correct value or return it to the Team Leader or analyst for correction. A Nonconformance Memo would be issued in the latter case.

5.11 After the second check is completed, the supervisor initials and dates the data sheet.

5.12 When both reviews are completed, the data can then be summarized and reported after verifying that the appropriate analytical method was used, the detection limit is appropriate, samples are properly identified, the proper number of significant figures is determined, and the data were calculated properly.

5.13 If applicable, the Group Leader transfers the approved data to a project summary sheet, and either he/she or an analyst enters the data into the SAM System.

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date
[illegible]	5/24/92

Terminated by	Date

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: CLP Pesticide Extraction of Soil Samples by OLM03.X				
Prepared by: BES ESK	Approved by:	Date: 5/31/95	QA Approval:	

1.0 PURPOSE

This procedure describes the method used to extract pesticide compounds from soil samples using the CLP 0LM03.X method.

2.0 APPLICATION

This method applies to all soil samples which are to be analyzed by CLP Statement of Work OLMO3.X. Note: This SOP replaces OP-008, CLP Pesticide/PCB Extraction Procedure.

3.0 REFERENCES

CLP Statement of Work OLMO3.X

4.0 ASSOCIATED SOPs

4.1 OP-004, GPC Operating Procedure

4.2 OP-003, Organic Labware Cleaning

4.3 OP-037, Solvent Extract Waste Disposal in the Organic Preparation Lab

4.4 OP-012, Extraction Solvent Suitability Check

4.5 OP-039, Wastewater Disposal in the Organic Preparation Lab

5.0 APPARATUS/EQUIPMENT/REAGENTS

5.1 Apparatus/Equipment

5.1.1 Funnels, short stem

5.1.2 Kuderna-Danish concentrator, 10 mL receiver

5.1.3 Snyder column

5.1.4 Steam bath

5.1.5 Nitrogen blowdown apparatus

5.1.6 Disposable pipets

5.1.7 Boiling chips - Soxhlet extracted with methylene chloride

5.1.8 Balance capable of weighing to 0.01 g

5.1.9 Beakers, 400 mL

5.1.10 Whatman filter paper, #41

5.1.11 Test tubes, 16X100 mm

5.1.12 Erlenmeyer flasks, 250 mL and 500 mL

5.1.13 Drying oven

5.1.14 Sonicator with 3/4 inch disrupter horn

5.1.15 Glass wool

5.2. Reagents

5.2.1 Acetone, pesticide grade

5.2.2 Methylene chloride, pesticide grade

5.2.3 Sodium sulfate, anhydrous (granular), baked at 450°C for 4 hours prior to use

5.2.4 PEST CLP surrogate spike solution as prepared by GC group, (See Section 10.0, Table 1)

5.2.5 PEST CLP matrix spike solution as prepared by GC group, (See Section 10.0, Table 2)

5.2.6 Methylene chloride:acetone (1:1). Add 1 L of acetone to 1 L of methylene chloride.

5.2.7 Hexane, pesticide grade

6.0 PROCEDURE

6.1 Decant and discard any water layer on a sediment sample. Record that water was decanted. Discard any foreign objects such as sticks, leaves, and rocks. Mix samples thoroughly.

Note: Percent moisture and pH can be done simultaneously for the pesticide and semivolatile fractions of the sample. It is important to perform these steps rapidly to avoid the loss of the more volatile extractables.

6.2 Percent Moisture

6.2.1 Label the bottom of an aluminum dish with ball-point pen. Record its tared weight. Place 5-10 g sample on dish.

6.2.2 Record dish + sample weight.

6.2.3 Place the aluminum dish containing sample for percent moisture in 105°C oven for 12 hours.

6.2.4 Remove sample from oven and let it cool in dessicator. Re-weigh this sample and record weight. Calculate percent moisture using the following formula:

% Moisture = [loss on drying/((dish + sample weight) - tare weight))] • 100.

6.3 pH Determination

6.3.1 Label a 250 mL Erlenmeyer with white labeling tape. Weigh 50 g of sample into it for pH determination. Note: If limited sample volume is received use a smaller weight.

6.3.2 Add the same amount of water in mL to the sample as you weighed sample in grams. For example, if you weighed 50 g of sample, add 50 mL of water. Determine the pH of sample with a pH meter. Document on Case Narrative is pH is >11 or <5.

6.4 Rinse 400 mL beaker with methylene chloride and attach white labeling tape with a pink square on it. Weigh and record 30.0 g ± 0.1 g into beaker for PEST extraction.

6.5 Add 60 g of sodium sulfate. Mix well with wooden spatula. Remove wooden spatula and add 2.0 mL of CLP PEST surrogate spiking solution to all samples. Add 1.0 mL of CLP PEST matrix spiking solution to the MS/MSD pair. Then immediately add about 100 mL of 1:1 methylene chloride:acetone.

6.6 Attach 3/4 inch disrupter horn and rinse with methylene chloride.

6.7 Tune Sonicator by pressing TUNE and setting output control to 0, 3, 6 and 10. Adjust bar graph display to a minimum setting at each output interval using tuning knob.

6.8 Program Sonicator by setting PROCESS TIME to 1:30, PULSAR ON to 1.0 and PULSAR OFF to 1.0.

6.9 Position disrupter horn 1/2 inch below surface of solvent and press START to begin sonication. This will take 3 minutes.

6.10 Place #41 Whatman filter paper in short stem funnel and place over 500 mL Erlenmeyer. Rinse with methylene chloride and discard rinse.

6.11 Decant and filter extracts through the filtered funnel into Erlenmeyer.

6.12 Repeat the extraction two more times with 2 additional portions of 1:1 methylene:chloride acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with a clean spatula.

6.13 On the final extraction, pour the entire sample into the filtered funnel and rinse with 1:1 methylene chloride:acetone.

6.14 Preparation for GPC (mandatory)

6.14.1 Sample concentration by Kuderna-Danish (KD)

6.14.1.1 Place a funnel on top of a KD extractor and add a glasswool plug in the funnel. Pour enough sodium sulfate to fill funnel at least half full. Rinse this whole apparatus by pouring approximately 40 mL methylene chloride through the sodium sulfate funnel. Swish the solvent around sides of KD, then discard this rinse.

6.14.1.2 Add the extracts. Break up the residue in the beaker and rinse beaker with methylene chloride into funnel. Using 30 mL of methylene chloride, rinse the sodium sulfate funnel into KD. Remove funnel then add two boiling chips to the KD extractor.

6.14.2 With a pre-wet three ball Snyder column, evaporate the extract down to about 5 mL on a hot bath. This will be about a 10 mL volume after the KD cools. At the proper rate of distillation, the balls will actively chatter but the chambers will not flood.

6.14.3 After the KD has cooled, wipe the outside moisture off with a Kimwipe. Remove the receiver and rinse the joint with methylene chloride into the receiver.

6.14.4 Place the concentrator tube in the N-Evap unit. Immerse in warm water bath (30-35°C) and evaporate the solvent volume to just below 1 mL.

6.14.5 Transfer the extract with three rinses of methylene chloride to a 10 mL calibrated test tube. Take to the mark with methylene chloride and cap with Teflon lined cap.

6.14.6 Proceed with SOP-004, GPC Operating Procedure.

6.15 Finalizing sample after GPC

6.15.1 Remove extract from GPC. Attach a receiver tube to KD and rinse with methylene chloride. Pour extract in KD. Rinse flask with methylene chloride into KD.

6.15.2 With a pre-wet three ball Snyder column, evaporate the extract down to about 5 mL on hot water bath. At the proper rate of distillation, the balls will actively chatter but the chambers will not flood. Immediately add 25 mL of hexane through the Snyder column and continue to concentrate to about 8 mL. Add a second portion of hexane and concentrate to about 8 mL.

6.15.3 After the KD has cooled, wipe the outside moisture off with a Kimwipe. Remove the receiver and rinse the joint with hexane into the receiver.

6.15.4 Place the concentrator tube in the N-Evap unit. Immerse in warm water bath (30-35°C) and evaporate the solvent volume to just about 3 mL.

6.15.5 Transfer the extract with three rinses of hexane to a test tube calibrated to 5 mL(using hexane) with three rinses of hexane. Take to the 5 mL mark with hexane.

6.15.6 All extracts must undergo Florisil cleanup (mandatory). Proceed with SOP-006, Florisil Pasteur Pipet Column Cleanup for CLP and Non-CLP Pesticide and PCB samples.

Note: The statement of work describes using a vacuum manifold in the Florisil cartridge cleanup. The cartridges described in SOP-006 do not require a vacuum manifold.

6.15.7 After Florisil cleanup, cap the vials and assemble in an extract tray. Deliver to Sample Control and store them in extract refrigerator.

7.0 QUALITY CONTROL

7.1 Extract two sodium sulfate blanks per batch and unique to each SDG. These blanks should be carried through all stages of the extraction process. Only one of the two blanks is to be turned into Sample Control. The second is in reserve in case one is lost due to breakage, not if it fails acceptance criteria.

7.2 A MS/MSD pair is required for each matrix per SDG. Note that each batch does not necessarily have a MS/MSD pair. It should be noted on workorder what sample to perform the MS/MSD.

7.3 Extraction (e.g., sonication) must be completed within 10 days from date of receipt.

8.0 INTERFERENCES

Due to low levels associated with this analysis, glassware must be meticulously clean. Rinse all appropriate glassware with methylene chloride prior to use.

9.0 TROUBLESHOOTING

If a sample requiring a MS/MSD pair is of insufficient quantity to extract 30 g for both pesticides and semivolatiles, another sample may be chosen. However, the Project Manager must be notified so he/she can notify the EPA. The reason for choosing the substitute must be provided to the EPA.

Remember a reduced volume can be used for pH determination.

10.0 TABLE(S)

Table 1

Surrogate Table

Surrogate Compound	Final Concentration (ug/mL)
Tetrachloro-m-xylene	0.2
Decachlorobiphenyl	0.2

Table 2

Matrix Spiking Solution

Pesticide	Final Concentration (ug/mL)
gamma-BHC (Lindane)	0.5
4,4'-DDT	1.0
Endrin	1.0
Heptachlor	0.5
Aldrin	0.5
Dieldrin	1.0

ROSS	SOP Standard Operating Procedure	SOP No: CW-006-00 Inital Date: 2/1/91 Page 1 of 3 Date Revised:

Title:

Use of the UV/Vis Spectrophotometer

Prepared by:	Approved by:	Date:	QC Approval:
	CHCaldwell	6/11/91	

1.0 PURPOSE

To describe the procedure for using the Hitachi U2000 Spectrophotometer for colorimetric analyses.

2.0 APPLICATION

This procedure is applicable to all manual colorimetric analyses.

3.0 REFERENCES

3.1 Operator's Manual for the Hitachi U2000 UV/VIS Spectrophotometer.

4.0 ASSOCIATED SOP'S

4.1 SOP NO. CW-004-00, Total Cyanide.

4.2 SOP NO. CW-005-00, Cyanide Amenable to Chlorination.

4.3 SOP NO. CW-007-00, Total Phenolics.

4.4 SOP NO. CW-008-00, Hexavalent Chromium.

4.5 SOP NO. CW-014-00, Chemical Oxygen Demand.

4.6 SOP NO. CW-015-00, Ammonia Nitrogen.

4.7 SOP NO. CW-016-00, Total and Ortho Phosphorus.

5.0 PROCEDURE

5.1 Equipment

5.1.1 Hitachi U2000 UV/VIS Spectrophotometer with sample sipper.

5.1.2 Colorized standards and samples.

5.2 Instrument Setup

5.2.1 Turn on instrument and allow to warm up for at least 10 minutes prior to use.

5.2.2 Open sipper compartment and pull the sample tube taut until it clicks in place.

5.2.3 Release the pinchcock on the drain tube.

5.2.4 Place sipper tube in a beaker of deionized water and hold the wash button in for 30 seconds to flush the sample cell.

5.2.5 If screen does not display the main menu, press the "Main Menu" button on the keypad.

5.2.6 From the main menu, pick "Photometry".

5.2.7 Choosing each menu item in turn, set the parameters for the analysis being performed, including: wavelength, number of standards, values of standards, unit of measurement, and name of analysis. Skip the "Save Parameters" choice until the sipper parameters have been set (see next step).

5.2.8 In "Sipper Setup", set sipping time to 4 seconds and delay to 8 seconds. The other choices can be left on their default values.

5.2.9 Choose "Save Parameters" from the Photometry menu and pick a slot to save it in (1-20, or "." for the next available slot).

NOTE: If the analysis being performed has been saved previously, the parameters can be recalled directly from the Main Menu by choosing "Testing Menu" and then the number that it was saved under.

5.2.10 When all the parameters have been set and saved, push the "Forward" button to advance to the calibration screen.

5.3 Calibration

5.3.1 Place the sipper tube into the blank (referred to as Standard 1 by the instrument), and hold in the "Wash" button in until the absorbance displayed no longer changes.

5.3.2 Wait approximately 8-10 seconds, then push the "Autozero" button on the keypad. When the absorbance displayed changes to .000, push the "Start" button to read in the value. It should display as .000 or close to it.

5.3.3 At the prompt, place the sipper tube into the second standard and push the sipper lever. When the beep is heard, remove the standard and wait for the reading.

5.3.4 Repeat the previous step for all standards. After the last standard has been read, the screen will display the curve. Choose "Print Curve" from the menu bar at the bottom of the screen.

5.4 Sample Analysis

5.4.1 Place the sipper tube into each sample and read each one the same as for the standards. The values will be printed out in whatever units were chosen for calibrating.

5.5 Cleanup

5.5.1 After the last sample has been read, flush the sample line with deionized water for at least 30 seconds.

5.5.2 Fill the sample cell with detergent solution by washing it through the cell for 5-10 seconds.

5.5.3 Close the pinchcock on the drain line.

5.5.4 Open the sipper panel and release the tension on the sample line by pushing the release button.

5.5.5 Turn off the instrument.

6.0 QUALITY CONTROL

6.1 Analyze quality control samples as appropriate for the particular analysis being performed.

6.2 Maintain the instrument as per the schedule of preventative maintenance.

ROSS	SOP Standard Operating Procedure	SOP No: CW-007-00 Inital Date: 2/1/91 Page 1 of 5 Date Revised:

Title:

Total Phenolics

Prepared by:	Approved by:	Date:	QC Approval:
[signature]	[signature]	5/23/91	[signature]

1.0 PURPOSE

To describe the distillation and spectrophtometric procedures for the determination of total phenolics.

2.0 APPLICATION

This procedure is applicable to the analysis of waters, wastewaters and groundwaters for total phenolics. It does not differentiate between different kinds of phenols.

3.0 REFERENCES

3.1 EPA 600, Revision 1983, Method 420.1.

3.2 Standard Methods for the Examination of Water and Wastewater, 17th edition, Method 5530.

3.3 EPA SW-846, 3rd edition, Method 9065.

4.0 ASSOCIATED SOPs

4.1 SOP NO. CW-006-00, Use of the UV-VIS Spectrophotometer.

5.0 PROCEDURE

5.1 Equipment

5.1.1 Distillation apparatus, see Figure 1.

5.1.2 Separatory funnels, 125 mL and 250 mL.

5.1.3 Spectophotometer for use at 460 nm and 510 nm.

5.1.4 100 mL graduated cylinders.

5.2 Reagents

5.2.1 Copper sulfate solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in deionized water and dilute to one liter.

5.2.2 Concentrated H_2SO_4.

5.2.3 Buffer solution: Dissolve 16.9 g NH_4Cl in 143 mL concentrated NH_4OH and dilute to 250 mL with deionized water.

5.2.4 Aminoantipyrine solution: Dissolve 2 g of 4-aminoantipyrine in deionized water and dilute to 100 mL.

5.2.5 Potassium ferricyanide solution: Dissolve 8 g of $K_3Fe(CN)_6$ in deionized water and dilute to 100 mL.

5.2.6 Stock phenol solution, 1000 ppm: Dissolve 1 g of phenol in deionized water and dilute to one liter. Store in amber glass. Shelf-life is 6 months.

5.2.7 Working solution A, 10 ppm: Dilute 10.0 mL of stock phenol solution to one liter with deionized water. Make fresh daily.

5.2.8 Working solution B, 1 ppm 10.0 mL of working solution A to 100 mL with deionized water.

5.2.9 Chloroform - use only in hood.

4.2.10 NaOH solution, 50%: commercially purchased.

5.3 Chloroform cleanup extraction - used if necessary to remove oily or tarry substance from the sample.

5.3.1 Adjust pH of 100 mL sample to 12.5 with NaOH.

5.3.2 Extract with 50 mL of chloroform in separatory funnel. Discard chloroform layer and transfer water layer to a beaker.

5.3.3 Place beaker on a hot plate and heat gently to remove any chloroform.

5.3.4 Proceed with distillation.

Figure 1

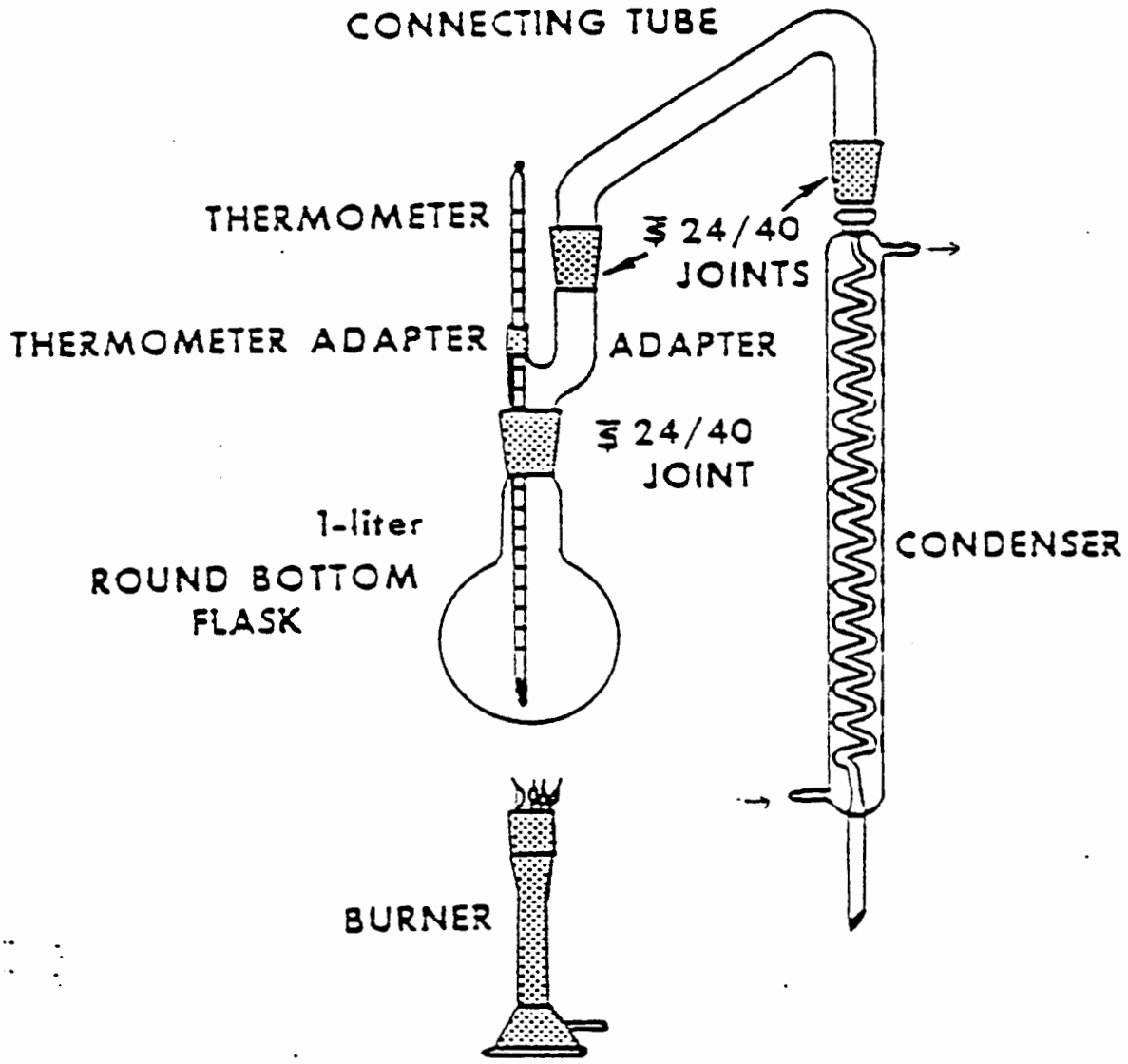


Figure 1

5.4 Distillation

5.4.1 Measure 100 mL of sample into the distillation flask. Add 3 drops of methyl orange - the color should be reddish-orange. If not, add H_2SO_4 dropwise to lower the pH.

5.4.2 Add 1 mL of copper sulfate solution.

5.4.3 Set up apparatus as in Figure 1, using a 150 mL beaker as the receiving vessel. Turn on condenser water.

5.4.4 Distill sample until 90 mL is collected, remove heat and allow to cool. Add 10 mL deionized water and continue distillation until approximately 100 mL sample is collected. Measure volume to nearest 1 mL in a graduated cylinder.

5.4.5 Turn off condenser water.

5.5 Direct Photometric Method

5.5.1 Standards - Pipet 0, 0.5, 1.0, 2.0, 3.0, 4.0, 5.0 ml of working solution A into spectrophotometer tubes. Dilute to 10 mL with deionized water.

mL, Working solution A	Concentration, µg
0	0
0.5	5
1.0	10
2.0	20
3.0	30
4.0	40
5.0	50

5.5.2 Samples - Pipet 10.0 mL of distillate or an aliquot diluted to 10 mL into a spectrophotometer tube. If distillate is turbid, filter through a membrane filter first.

5.5.3 Add 0.2 ml buffer solution and mix.

5.5.4 Add 0.2 mL aminoantipyrine solution and mix.

5.5.5 Add 0.2 mL potassium ferricyanide solution and mix.

5.5.6 After 15 minutes calibrate spectrophotometer with standards at 510 nm. Read concentration of samples (in µg) directly from the instrument.

5.6 Chloroform, Extraction Method - concentration step for better detection limit

5.6.1 Standards - Pipet 0, 3.0, 5.0, 10.0, 20.0 and 25.0 mL of Working solution B into 125 mL separatory funnels and dilute to 50 mL with deionized water.

mL Working solution A	Concentration, µg
0	0
3.0	3
5.0	5
10.0	10
20.0	20
25.0	25

5.6.2 Samples - Transfer 50 mL distillate to a 125 mL separatory funnel.

5.6.3 Add 1 mL buffer and mix.

5.6.4 Add 0.3 mL aminoantipyrine solution and mix.

5.6.5 Add 0.3 mL potassium ferricyanide solution and mix.

5.6.6 After 3 minutes, extract with 25 mL chloroform. Shake the funnel 10 times, let settle, shake 10 more times and let settle again.

5.6.7 Filter chloroform layer through Whatman 40 filter paper for analysis.

5.6.8 Calibrate spectrophotometer with standards at 460 nm. Read concentration of samples (in µg) directly from the instrument.

5.7 Calculation

$$\text{mg/l phenolics} = \frac{\mu g \text{ from curve}}{\text{aliquot for color}} \times \frac{\text{mL distillate}}{\text{mL original sample}}$$

6.0 Quality Control

6.1 Distill a minimum of one SRS with every set.

6.2 Analyze an undistilled SRS at the time of the spectrophotometric analysis.

6.3 Analyze a minimum of one matrix spike/matrix spike duplicate pair once every 20 samples.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Fluoride by Ion Selective Electrode			
Prepared by: Sharon Jefferson	Approved by:	Date: 7/15/96	QA Approval:

1.0 PURPOSE

To outline the procedure for measuring the fluoride in sample. This process may include a distillation process (contained herein) followed by the use of the use of an Ion Selective Electrode (ISE) for actual quantitative analyses.

2.0 APPLICATION

This procedure is applicable to drinking water, wastewaters, and groundwaters. If a water sample does not contain interferences in excess of the tolerances listed in Section 11.0, Interferences, the sample may be analyzed directly starting in Section 8.0, Quantitative Analysis Using Fluoride Electrode; if the analysis is for compliance with NPDES regulations, the sample must be distilled. Standard Methods indicates that samples with dissolved solids at a concentration which exceeds 10,000 mg/L should be distilled.

3.0 REFERENCES

"Fluoride, Total (Colorimetric, SPADNS with Bellack Distillation)", Methods for Chemical Analysis of Water and Wastes (EPA-600), Method 340.1, Revised 1978

"Fluoride, Total (Potentiometric, Ion Selective Electrode)", Methods for Chemical Analysis of Water and Wastes (EPA-600), Method 340.2, Revised 1974

"Fluoride; A, Introduction; B, Preliminary Distillation Step; C, Ion-Selective Electrode Method", Standard Methods for the Examination of Water and Wastewater, 19th Edition, Method 4500-F^- (A,B,C), Approved 1994

4.0 ASSOCIATED SOPs

None

5.0 REAGENTS

5.1 Buffer solution, pH 5.0 - 5.5

To approximately 500 mL of deionized water in a 1 liter beaker, add 57 mL of glacial acetic acid, 58 g of sodium chloride and 4 g of 1,2-cyclohexylene dinitrilo tetracetic (CDTA). Stir to dissolve and allow to cool. Adjust pH of the solution to between 5.0 and 5.5 with 5N sodium hydroxide (approximately 150 mL). Transfer solution to a 1 liter volumetric and dilute to mark. If commercially available, this buffer solution my be purchased.

5.2 Sodium fluoride stock solution, 100 mg/L

Dissolve 0.2210 g of sodium fluoride (NaF) in deionized water and dilute to mark in a 1 liter volumetric flask. Store in plastic.

5.3 Sodium fluoride, standard solution, 10.0 mg/L

Dilute 10 mL of stock solution to 100 mL of deionized water in a plastic volumetric flask. Store in plastic.

5.4 Sodium hydroxide, 5N

Dissolve 200 g sodium hydroxide in deionized water, cool and dilute to 1 liter in a volumetric flask. Alternatively, dilute 1 volume of 50% NaOH solution with 4 volumes of deionized water.

5.5 Silver sulfate, Ag_2SO_4, crystals - Reagent grade

6.0 EQUIPMENT

6.1 Distillation apparatus - See Section 12, Figure 1

6.2 Plastic beakers and volumetric flasks

6.3 Combination Fluoride ISE

6.4 pH/ISE meter, Orion 720 or equivalent

7.0 DISTILLATION PROCEDURE

7.1 Into clean distillation flasks, pour 200 mL of deionized water. To each flask carefully add 100 mL of concentrated H_2SO_4 and MIX THOROUGHLY. Add several boiling beads.

7.2 At first, gently, and then vigorously heat the contents of each flask to 180°C. To prevent overheating and carryover, discontinue heating when the temperature reaches 178°C. Discard the distillate. This process removes fluoride contamination (with the exception of high chloride interferences which are addressed in Section 7.4) and adjusts the acid-water ratio for subsequent distillations. Allow the contents of the flask to cool below 60°C.

7.3 Add 100 mL of sample (or an aliquot diluted to 100 mL) to the flask and mix well. Heat the mixture to 180°C and collect the distillate in a plastic beaker. To prevent overheating and carryover, discontinue heating when the temperature reaches 178°C. Record the amount of distillate recovered (measured by a graduated cylinder) and store in plastic.

7.4 Add Ag_2SO_4 at a rate of 5 mg per mg of chloride (chloride actually in the sample) when high chloride samples are distilled. (See Section 11.0 for interfering levels of chloride.)

7.4 The sulfuric acid mixture in the distillation flask can be used a maximum of 3 times or until it is discolored by a sample. Then discard the mixture and repeat Steps 7.1 and 7.2 to regenerate the mixture.

8.0 QUANTITATIVE ANALYSIS USING FLUORIDE ELECTRODE

8.1 Standards Preparation

Prepare a total of 4 standards in the minimum range of 1.0 to 10. An additional 5th standard may be prepared to extend the range of the curve. Prepare two standards which bracket the sample concentrations then divide the range into two additional, evenly spaced concentrations for a total of 4 standards.

8.2 Turn on the ISE meter and set the mode to CON. Push "calibrate", then enter the number of standards at the prompt and push "yes".

8.3 Add 25 mL of buffer solution to 25 mL of the lowest standard in a plastic beaker and mix. Insert a Teflon stir bar and begin stirring the solution. Place the fluoride electrode in the solution.

8.4 At the "ready" prompt, enter the actual concentration and push "yes". Remove the electrode, rinse with deionized water and blot dry very gently.

8.5 Repeat Steps 8.3 and 8.4 for all the standards. Record the actual readings onto the Analytical Data Worksheet. After the last one has been entered, the meter will display the average slope of the curve. Document the slope of the curve on the Analytical Data Worksheet. (If the slope is outside the range of -56 to -60, consult the manufacturer's instructions.)

8.6 Analyze the samples the same way as the standards beginning with Step 8.3, reading the concentration in mg/L directly from the meter at the "ready" prompt. Record the readings of the samples onto the Analytical Data Worksheet.

9.0 CALCULATION

$$\text{mg/L F} = \text{mg/L F from meter} * \frac{\text{(mL distillate)}}{\text{(mL distilled)}}$$

10.0 QUALITY CONTROL

10.1 A minimum of one LCS per batch is analyzed. The LCS spike must be from a different vendor than the spike used to prepare the curve.

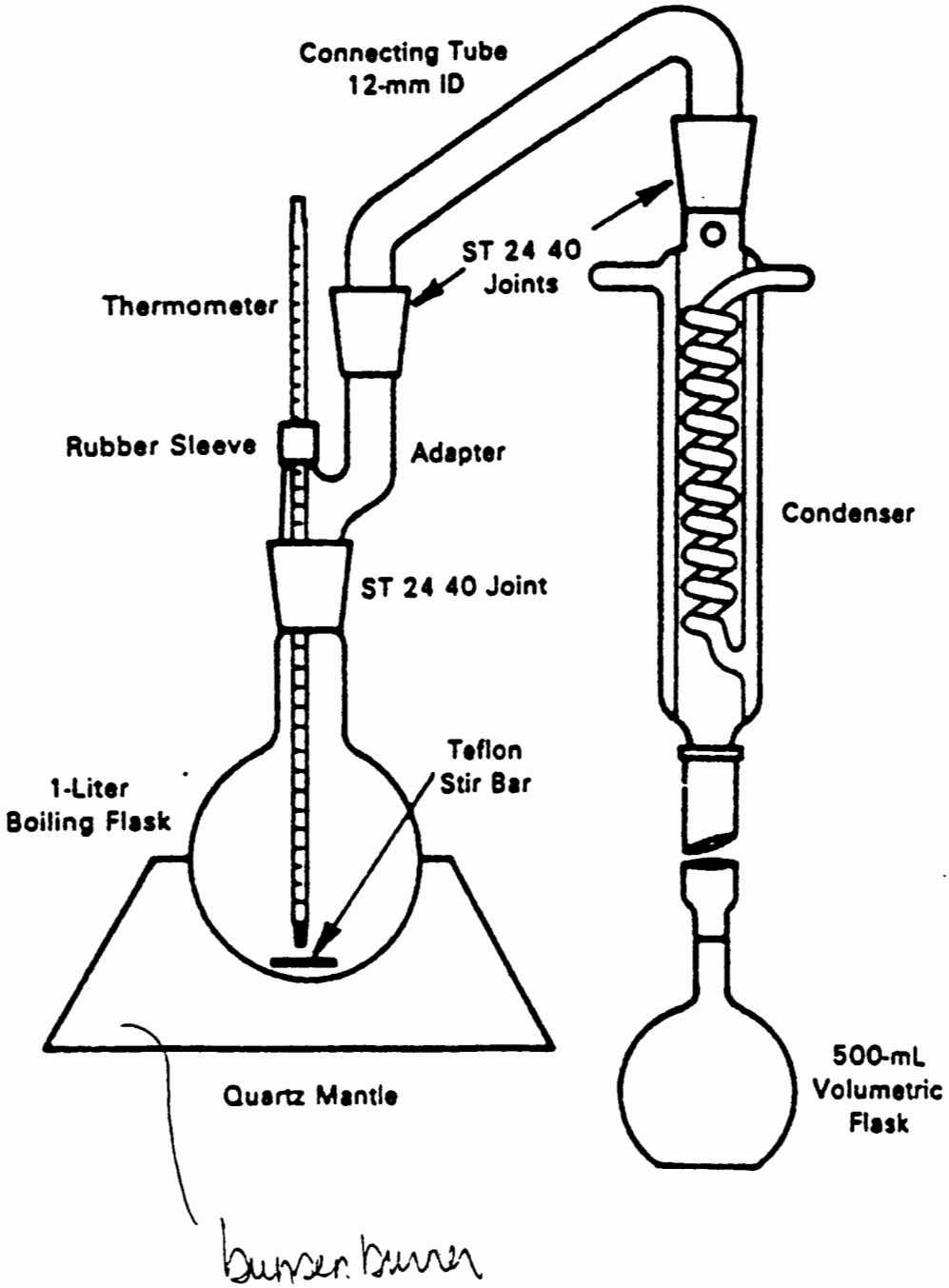
10.2 One MS/MSD pair is analyzed for every 20 samples.

11.0 INTERFERENCES

Substance	Conc. (mg/L)	Type of Error
Alkalinity ($CaCO_3$)	7000	Positive
Aluminum (Al^{3+})	3.0	Negative
Chloride (Cl^-)	20000	
Chlorine	5000	
Iron	200	Negative
Hexametaphosphate ($NaPO_3$)	50000	
Phosphate (PO_4^{3-})	50000	
Sulfate (SO_4^{2-})	50000	

12.0 FIGURE(S)

Figure 1
Distillation Apparatus for Fluoride



ROSS	SOP Standard Operating Procedure	SOP No: CW-011-00 Inital Date: 2/1/91 Page 1 of 3 Date Revised:

Title:

Total Dissolved Solids (TDS)

Prepared by:	Approved by:	Date:	QC Approval:
		6/3/91	

1.0 PURPOSE

To describe the procedure used for the determination of total dissolved solids (filterable residue).

2.0 APPLICATION

This procedure applies to any aqueous sample which can be filtered through a glass fiber filter.

3.0 REFERENCES

3.1 EPA 600, Revision 1983, Method 160.1.

3.2 Standard Methods for the Extraction of Water and Wastewater, 17th edition, Method 2540C.

4.0 ASSOCIATED SOPs

4.1 SOP No. CW-012-00, Total Suspended Solids (TSS).

4.2 SOP No. CW-013-00, Total Solids

5.0 PROCEDURE

5.1 Equipment

5.1.1 Beakers, 250 mL.

5.1.2 Filter flask and Gelman magnetic filter funnel with vacuum source.

5.1.3 Analytical balance.

5.1.4 Filter forceps.

5.1.5 Graduated cylinders.

5.1.6 Desiccator.

5.1.7 Drying oven, 180°C.

5.1.8 Glass fiber filters, 7.0 cm diameter

5.2 Filter preparation

5.2.1 Place stacks of approximately 10 filters in the funnel and rinse with three successive 20 mL portions of deionized water.

5.2.2 Separate filters, place in an aluminum pan and dry for at least one hour at 103°C. Handle only with forceps.

5.2.3 Desiccate filters until needed.

5.3 Beaker Preparation

5.3.1 Label clean 250 mL beakers and heat in oven at 180°C for one hour. Handle only with tongs or gloves.

5.3.2 Desiccate until needed, at least one hour.

5.4 Analysis

5.4.1 Weigh beakers needed on an analytical balance and record weight.

5.4.2 Place filter in filtering apparatus, turn on vacuum and rinse with a small amount of deionized water (to create good filter-funnel seal).

5.4.3 Measure 100 mL of well mixed sample (or appropriate smaller aliquot) and pour it through a filter. Rinse with three successive 10 mL portions of deionized water. Continue applying vacuum until all standing water has passed through the filter.

5.4.4 Turn off the vacuum source and transfer filtrate to the tared beaker. Rinse flask into beaker. Place beaker in 98°C oven overnight to evaporate without spattering, then raise temperature to 180°C for at least one hour.

5.4.5 If suspended solids are to be determined also, the filter can now be transferred (see SOP CW-012-00, Total Suspended Solids (TSS)).

5.4.6 Desiccate beakers for at least one hour and then weigh.

5.4.7 If the total residue in the beaker exceeds 0.5 g repeat the analysis using a smaller aliquot.

5.5 Calculations

$$\text{TDS, mg/L} = \frac{(A-B) \times 1000}{C}$$

Where:

A = weight of beaker + residue, mg

B = weight of beaker, mg

C = mL of sample filtered

6.0 Quality Control

6.1 A minimum of one SRS must be run with each sample set.

6.2 Duplicates must be analyzed at a frequency of 10%.

ROSS	SOP Standard Operating Procedure	SOP No: CW-012-00 Inital Date: 2/1/91 Page 1 of 3 Date Revised:

Title:

Total Suspended Solids (TSS)

Prepared by:	Approved by:	Date:	QC Approval:
		6/3/91	

1.0 PURPOSE

To describe the procedure used for the determination of total suspended solids (non-filterable residue).

2.0 APPLICATION

This procedure applies to any aqueous sample which can be filtered through a glass fiber filter.

3.0 REFERENCES

3.1 EPA 600, Revision 1983, Method 160.2.

3.2 Standard Method for the Examination of Water and Wastewater, 17th edition, Method 2540D.

4.0 ASSOCIATED SOPs

4.1 SOP No. CW-011-00, Total Dissolved Solids (TDS).

4.2 SOP No. CW-013-00, Total Solids (TS).

5.0 PROCEDURE

5.1 Equipment

5.1.1 Glass fiber filter, 4.25 cm diameter, Whatman 934-AH or equivalent.

5.1.2 Filter flask and Gelman magnetic filter funnel with vacuum source.

5.1.3 Analytical balance.

5.1.4 Aluminum weighing pans.

5.1.5 Graduated cylinders.

5.1.6 Desiccator.

5.1.7 Filter forceps.

5.1.8 Drying oven, 103-105°C.

5.2 Filter Preparation

5.2.1 Place stacks of approximately 10 filters in the funnel and rinse with three successive 20 mL portions of deionized water.

5.2.2 Separate filters, place in aluminum pan and dry for at least one hour at 103-105°C. Handle filters with forceps only.

5.2.3 Desiccate filters until needed.

5.3 Analysis

5.3.1 Weigh filters needed on an analytical balance and record weight. Place in individually labeled aluminum pans.

5.3.2 For each sample, place a filter in the filtering apparatus, turn on the vacuum and rinse with a small amount of deionized water (to create good filter-funnel seal).

5.3.3 Measure 100 mL of well-mixed sample (or appropriate smaller aliquot) and pour it through the filter. Rinse with three successive 10 mL portions of deionized water and rinse any materials from the walls of the funnel. Continue applying vacuum until all standing water has passed through the filter.

5.3.4 Carefully remove filter from funnel base (suction need not be turned off), and place in its labeled pan. Dry at 103-105°C for at least one hour.

5.3.5 If dissolved solids are to be determined also, the filtrate can now be recovered (see SOP No. CW-011-00, Total Dissolved Solids (TDS)).

5.3.6 Desiccate filters for at least 30 minutes and then weigh.

5.3.7 If the total residue on the filter exceeds 0.5 g, repeat the analysis using a smaller aliquot.

5.5 Calculations

$$\text{TSS, mg/L} = \frac{(A-B) \times 1000}{C}$$

Where:

A = weight of filter + residue, mg

B = weight of filter, mg

C = mL of sample filtered

6.0 Quality Control

6.1 A minimum of one SRS must be run with each sample set.

6.2 Duplicate must be analyzed at a frequency of 10%.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: GPC Operating Procedure							
Prepared by:	BES	Approved by:	CAR	Date:	6/8/95	QA Approval:	

1.0 PURPOSE

The purpose of this procedure is to describe the operation of the Gel Permeation Chromatography Autoprep models 1002B and 1000.

2.0 APPLICATION

This procedure is mandatory for all CLP soil extracts. It also applies to all soil or water extracts which have one or more of the following characteristics: unusually strong odor, significant color, noticeably higher viscosity than methylene chloride, or extracts which contain higher molecular weight contaminants. GPC is used to remove oils, fats, and other large compounds from extracts that interfere with the analysis of targets analytes.

3.0 REFERENCES

3.1 CLP Statement of Work OLMO3.X

3.2 GPC Operational Manual for Autoprep 1000 and 1002B

4.0 ASSOCIATED SOPs

4.1 OP-042, CLP Semivolatile Organic Extraction of Water Samples by OLMO3.X

4.2 OP-043, CLP Pesticide Extraction of Soil Samples by OLM03.X

4.3 OP-044, CLP Pesticide Extraction of Water Samples by OLMO3.X

4.4 OP-037, Solvent Extract Waste Disposal

4.5 OP-048, CLP Semivolatile Organic Extraction of Soil Samples by OLMO3.X

5.0 APPARATUS/EQUIPMENT/REAGENTS

5.1 Apparatus/Equipment

5.1.1 Methylene chloride, pesticide grade

5.1.2 Biobeads (S-X3) 200-400 mesh

5.1.3 GPC calibration solution - Prepare a solution in methylene chloride that contains the following analytes in the concentrations below:

Analyte	mg/mL
Corn oil	25
Bis(2-ethylhexyl) phthalate	0.5
Methoxychlor	0.1
Perylene	0.02
Sulfur	0.08

Note: Sulfur is not very soluble in methylene chloride. Weigh out the corn oil, warm it and transfer the weighed amount of sulfur into the warm corn oil. Then transfer the corn oil to the 500 mL volumetric flask and add the rest of the analytes.

5.1.4 n-Butyl chloride -make a solution 10% butyl chloride 90% methylene chloride

5.2 Equipment

5.2.1 GPC Autoprep Model 1002B and GPC Autoprep Model 1000 High pressure column 350 mm x 21.2 mm.

5.2.2 Ultraviolet detector fixed wavelength (250 nm) with a Semiprep Flow-thru Cell

5.2.3 Strip chart recorder

5.2.4 Syringe 10 mL with Luerlock fitting

5.2.5 Acrodisc syringe filters (PTFE or nylon) 0.5 um and 0.1 um

5.2.6 Erlenmeyer flasks, 500 mL

5.3 Calibrating the GPC

5.3.1 Activate the GPC pump by indexing on the controller console until the standby light goes out and the pump light comes on.

5.3.2 Let the pump run for at least 30 minutes. If high pressure column is being used, select high pressure limit when unit is first turned on.

5.3.3 Turn on the UV detector and the chart recorder. Set the range at 0.5 auf, the rise time at 1.0 sec., and the chart speed at 1 cm/min. Ensure that all valve switches and tubing are configured for flow through UV detector.

5.3.4 Zero the detector and chart recorder.

5.3.5 Check the flow rate exiting the main column or UV recorder; it should read between 4.5 - 5.5 mL/minute. Adjust to as near to 5.0 mL/minute as possible.

5.3.6 Using a Luer lock syringe and Acrodisc filter, filter 10 mL of the GPC calibration solution into a GPC tube. For correct chromatogram scaling, the calibration solution will need a 1:3 dilution with methylene chloride prior to run. Place the tube into position 1 on the GPC and make sure it is hand tight.

5.3.7 Set the dump time at 60:00 minutes and the collect time at 00:00 minutes. No wash time is necessary. High Pressure units should have dump set to 40 minutes. Index through the program to check all parameters. If all parameters are correct then press run.

Ultrasonic sensor pre-load time = 05 sec.

Debounce time = 0.05 sec.

Maximum load time = 25.00 sec.

Start with sample 01, Terminal sample 1

5.3.8 The chart recording should be initiated as soon as the sample is injected onto the column.

NOTE: Ensure waste containers are empty, clean solvent supply is available, nitrogen gas is on, and rinse pressure is at least 10.

5.3.9 When the run is complete turn off the detector and chart recorder and index until the pump is activated.

5.3.10 The first double peak is corn oil, then phthalate, methoxychlor, perylene, and finally sulfur.

5.3.11 Measure from the start of deflection to the end of the corn oil peak in centimeters. That is the dump time for BNAs.

5.3.12 Measure from the start deflection to the end of the phthalate peak in centimeters. That is the dump time for pesticides/PCBs.

5.3.13 The distance from the dump time to the beginning of the sulfur peak is the collect time.

5.3.14 Label all peaks on the strip chart, record date, title, column ID, operator, flow rate, dump and collect for BNA and Pest methods onto the chart. Attach chart into corresponding GPC logbook.

5.3.15 Chart recording must exhibit good chromatography (separation of peaks) and resolution relative to baseline. See: CLP SOW Technical Acceptable Criteria for GPC calibration.

5.4 Sample Extract Cleanup

5.4.1 Highly viscous sample extracts must be diluted and loaded into several GPC tubes or use an aliquot of the sample diluted to 10 mL.

5.4.2 All sample extracts must be filtered through ≤5 um filter disc before GPC cleanup.

5.4.3 Rinse off all port tubing with methylene chloride. Rinse out GPC tubes and syringes with acetone, then methylene chloride.

5.4.4 Set the dump and collect times as previously determined for the analysis to be performed on the extracts. Set the wash time at 10 minutes for relatively clean samples and up to 15 minutes for very dirty samples.

5.4.5 Put a methylene chloride "junk" sample onto the first position. Samples should be loaded in order of darkness, from light to dark, to avoid contamination.

5.4.6 Make sure all GPC tubes are hand tight. Ensure that pink label tape is affixed to the extract to indicate the extract is being or has been run through the GPC.

5.4.7 Put aluminum foil capped Erlenmeyer flask under the collect lines and make sure all line numbers match labels and logbook designation.

5.4.8 Index through the program making sure all parameters are correct.

5.4.9 Check the flow rate, it should not vary greater than 2.5% from calibration run for high pressure units and not greater than 5% for normal pressure units. Column pressure typically falls within 7 - 11 psi for normal pressure units and 170 - 240 psi for high pressure units.

5.4.10 If above criteria is met, push run button.

5.4.11 Ensure all sample runs are recorded into GPC logbook.

6.0 QUALITY CONTROL

6.1 At least once every 7 days, the calibration of the GPC must be verified with two check mixtures. Concentrate 2.0 mL of pesticide matrix spike solution to less than 1.0 mL under nitrogen and adjust the final volume to 10.0 mL with methylene chloride.

6.2 The second mixture is prepared with 2 ug of Aroclor 1016 and 2 ug of Aroclor 1260 in a final volume of 10.0 mL methylene chloride.

6.3 Put the solutions on GPC using previously determined pesticide dump and collect times.

6.4 Concentrate the extracts on the steambath and switch to hexane. Bring to a final volume of 10 mL.

6.5 Analyze by GC/EC. The pattern of the Aroclor quantitation peaks must be the same as that of previously run standards and the recovery of each of the single component peaks must be between 80 and 110 percent. If this is not the case then the column must be replaced and recalibrated according to Section 5.3.

6.6 Run a SV blank check. (10 mL of methylene chloride run through the GPC and taken to 0.5 mL FV)

6.7 Run a Pest BLK check. (10 mL of methylene chloride run through the GPC and exchanged to hexane and adjusted to a 5 mL FV)

Note: It is a good idea to get all GPC checks to the appropriate analytical department as soon as possible after running a calibration. GPC may not be used for CLP samples until all applicable check parameters pass.

7.0 TROUBLESHOOTING

7.1 Occasionally SX-3 gel columns will become visually stained following dirty sample runs. Run 10% n-butyl chloride solution all 23 positions as a washing program. This can be done on the Envirosep columns as well if you suspect they are dirty.

7.2 Methylene chloride extracts injected onto column must contain at least 90% methylene chloride. Extracts containing acetone should be nitrogen blown to > 1 mL then diluted up to 10 mL in methylene chloride prior to GPC.

7.3 Record all maintenance activities pertaining to GPC into corresponding GPC maintenance logbooks.

7.4 Dirty collect lines should be purged before attempting to run extracts.

7.5 Each GPC should be backwashed every 6 months or if clogged or if sample load time is > 30 seconds.

7.6 Read troubleshooting section of GPC manual if any symptom of trouble exists.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Extraction Solvent Suitability Check							
Prepared by:	BES	Approved by:	CBK	Date:	6/8/95	QA Approval:	

1.0 PURPOSE

This procedure describes the method used to determine the suitability of extraction solvents.

2.0 APPLICATION AND SCOPE

This method applies to all solvents used to extract and concentrate samples for organic analysis.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 APPARATUS/REAGENTS

5.1 Kuderna-Danish (KD) concentrator, 10 mL receiver, and 3 ball Snyder column

5.2 Hot water bath

5..3 Nitrogen evaporation device - N-EVAP or equivalent

5.4 Pasteur pipets, 9" length

5.5 Autosampler vial calibrated to 1.0 mL

5.6 Ether without preservatives

5.7 Acetone - pesticide grade or equivalent

5.8 Methylene chloride - pesticide grade or equivalent

5.9 Hexane - pesticide grade or equivalent

6.0 PROCEDURE

6.1 Concentration - This step applies to all solvents used for organic extractions except for Freon.

Note: Methylene chloride will have two lot checks concentrated and analyzed by GC and an additional lot check concentrated for GC/MS. For GC, one lot check will get hexane exchanged and the other one be concentrated directly down to 1 mL.

6.1.1 Assemble a very clean solvent washed KD concentrator.

6.1.2 Pour 500 mL of the extraction solvent to be analyzed directly into the KD. (Remember, do not pour solvent through a sodium sulfate filled funnel.)

6.1.3 Add a few boiling chips to KD and concentrate to about 4 mL on a hot water bath. Allow to cool 10 minutes.

6.1.4 If methylene chloride is being checked, hexane exchange one of the KD flasks using about 25 mL of hexane added through the Snyder column.

6.1.5 Remove from bath when apparent volume is about 3 mL.

6.1.6 Remove Snyder column, do not rinse lower joint into the concentrator.

6.1.7 Concentrate to 1.0 mL on the N-Evap at 30°C - 35°C.

6.1.8 Transfer the solvent to an autosampler vial calibrated to 1.0 mL.

6.1.9 Send methylene chloride checks to both GC and GC/MS for analysis. All other solvent checks will be analyzed by GC.

7.0 QUALITY CONTROL

7.1 For a solvent to be acceptable, no compounds of interest may be detected in the extraction solvent at a concentration greater than one third the EQL.

7.2 Hard copies of all solvent analyses will be filed in the extraction lab.

8.0 INTERFERENCES

Avoid contact of latex gloves on the inside of extraction glassware and on tips of Pasteur pipets.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Extraction of Acid Herbicides for Water Samples Using Method 8151 (Modified)			
Prepared by: BES	Approved by:	Date: 5/7/96	QA Approval:

1.0 PURPOSE

This method describes the procedure used to extract herbicides from water samples.

2.0 APPLICATION

This method applies to all water samples which are to be analyzed for acidic herbicides. Ross Analytical Services, Inc. (RASI), has modified the sample preparation process documented in Method 8151. RASI uses continuous extractors versus separatory funnels to perform sample extractions. Method 8151 vaguely refers to employing the use of a technique other than separatory funnel extraction whenever emulsions form. RASI has found that the continuous extraction process produces efficiencies comparable to separatory funnel extraction for most samples; therefore, we have adopted the use of this technique for all water samples.

Section 9.0 describes potential interferences.

3.0 REFERENCES

"Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization: Capillary Column Technique", SW-846, Method 8151, September 1994, Revision 0

4.0 ASSOCIATED SOPs

GX-001, Organic Preparation of General Solutions

5.0 REAGENTS

5.1 Reagent water

5.2 Sulfuric acid

1:1 (vol/vol) - Slowly add 50 mL concentrated H_2SO_4 to 50 mL reagent water.

5.3 Hydrochloric acid

1:1 (vol/vol) - Add one volume of concentrated HCl to one volume of reagent water.

5.4 Acidified sodium sulfate - Bake at 400 °C for 4 hours. Add enough diethyl ether to make a slurry with 100 g sodium sulfate. Add 0.1 mL of concentrated H_2SO_4 and mix thoroughly. Check the pH by mixing a 1 g portion of the resulting solid with 5 mL of reagent water; the pH should be less than 4. Discard this portion used to check the pH. Dry the mixture under a hood on a hot plate set at 1. When all the ether has evaporated, oven dry the mixture at 105°C for 4 hours.

5.5 Solvents - Acetone, methanol, ethanol, methylene chloride, hexane, and diethyl ether (pesticide grade or equivalent)

5.6 Anhydrous sodium sulfate - Bake at 400 °C for 4 hours.

5.7 N-methyl-N-nitroso-p-toluenesulfonamide (Diazald)

5.8 Silicic acid

5.9 Surrogate standard solution - This solution is prepared by the Gas Chromatography Department at a concentration of 100 ug/mL.

5.10 Spiking solution for MS, MSD, and LCS - This solution is prepared by the Gas Chromatography Department at a concentration of 5 ug/mL.

5.11 Acid ether - Add 10 drops of concentrated sulfuric acid to 300 mL of diethyl ether.

5.12 Potassium hydroxide solution (37%) - Dissolve 37 g of KOH in reagent water and dilute to 100 mL.

5.13 Wide range pH paper

5.14 12 N Sulfuric acid - Slowly add 335 mL concentrated sulfuric acid to approximate 600 mL reagent water. Bring to volume in a 1 liter volumetric.

5.15 6 N Sodium hydroxide - Dissolve 216 g NaOH in 900 mL reagent water. Bring to volume in a 1 liter volumetric.

6.0 EQUIPMENT

6.1 Erlenmeyer flasks - 250 mL, 500 mL, 2000 mL

6.2 Diazomethane generator

6.3 Culture tubes - 10 mL calibrated to 5 mL

6.4 Continuous extractors

6.5 10 cm long stem funnels

6.6 Kuderna-Danish (KD) concentrators

6.7 Boiling chips - Solvent washed

6.8 Hot water bath

6.9 Beakers - 400 mL, 600 mL, 1000 mL

6.10 Heating mantel

6.11 Balance - capable of weighing +/- 0.01 g

6.12 Syringe - 1 mL, 2.5 mL, 5 mL, 0.5 mL

6.13 Glass rod

6.14 Pasteur pipets

6.15 Graduated cylinder - 500 mL, 1000 mL

7.0 PROCEDURE

7.1 Extraction Procedure

Rinse the continuous extractor and flask with dilute hydrochloric acid solution. Rinse the residual acid from the glassware with one rinse of deionized water.

7.1.1 Add 250 mL of methylene chloride to the bottom of the continuous extractor.

7.1.2 Check and record the pH of the sample with wide range pH paper.

7.1.3 Mark the sample volume on the side of the sample container.

7.1.4 Based on color, suspended material, and odor of the sample, determine if clean-up is required.

7.1.5 If sample DOES NOT require clean-up, proceed with 7.1.11. If sample DOES require clean-up, proceed with 7.1.6.

7.1.6 Add 10 mL of 6 N NaOH directly into each liquid-liquid extractor.

7.1.7 Add 1 liter of sample to extractor using an acid-rinsed graduated cylinder.

7.1.8 Add 0.5 mL of surrogate (100 ug/mL of 2,4-DB) to all extractions including all QC samples prepared with these samples.

7.1.9 Add 1 mL of spike solution (5 ug/mL) to two portions of a sample chosen for the MS and MSD, then add 1 mL of spike to one portion of reagent water for an LCS.

7.1.10 Mix the sample with a glass rod and check the pH. Add additional 6 N NaOH if pH is less than 12. Allow the sample to stand one half an hour with occasional mixing. Extract the sample for 16 - 18 hours. Check the pH to ensure that it is still at a pH of 12 or greater following the clean-up extraction. If the pH is less than 12, **DO NOT DISCARD THE EXTRACT**. If the pH is 12 or greater, discard the extract. Add a clean, acid-rinsed flask containing 150 mL of methylene chloride and proceed to 7.1.15.

7.1.11 Add 1 liter of sample to the extractor using an acid-rinsed graduated cylinder.

7.1.12 Add 0.5 mL of surrogate (100 ug/mL of 2,4-DB) to all extractions including all QC samples.

7.1.13 Add 1 mL of spike solution (5 ug/mL) to two portions of a sample chosen for the MS and MSD, then add 1 mL of spike to one portion of reagent water for an LCS.

7.1.14 Adjust the pH of the sample to less than 2 with 17 mL of 12N sulfuric acid. Check the pH and adjust as needed with additional acid.

7.1.15 Extract the sample for 18-24 hours. Check the extraction pH to ensure that it was at least 2 or lower following extraction. If the pH was greater than 2, adjust the pH to 2 or less and continue extracting the sample.

7.1.16 Following completion of the extraction process, add 5 g of acidified anhydrous sodium sulfate into the ball flask. Let the extract sit for 1 hour prior to concentrating it using a KD.

7.2 Concentration

Rinse the KD assembly and the funnel, glasswool, and sodium sulfate with acid ether solution. Add Na_2SO_4 to funnel, then rinse entire assembly several times with methylene chloride.

7.2.1 Pour the extract through an acid rinsed and solvent rinsed sodium sulfate into a KD flask.

7.2.2 Pre-wet a 3 ball Snyder column with about 1 mL of methylene chloride, then evaporate the extract down to about 5 mL on a hot water bath. At the proper rate of distillation the balls of the column will actively chatter, but the chamber will not flood. Allow to cool for 10 minutes.

7.2.3 Remove the Snyder column and rinse the lower joint into the KD flask.

7.2.4 Rinse the flask with about 1 mL methylene chloride into the receiver.

7.2.5 Remove the receiver and rinse the lower joint of the flask into the receiver.

7.2.6 If the extract is not to be esterified immediately, cap and store extract.

7.2.7 Immediately prior to esterification, blow the extract to 0.5 mL at **ROOM TEMPERATURE** with nitrogen. Add one drop of methanol to extract to aid in water removal.

7.3 Esterification by diazomethane

Diazomethane is very explosive and carcinogenic!

Before proceeding, read all the appropriate Material Safety Data Sheets (MSDSs) of all reagents used in this method. Keep all reagents under a hood and wear gloves. Keep the diazomethane generator behind a safety shield in a hood free of all reagents and equipment not required in the preparation. Avoid using boiling chips, metallic implements, unpolished glass parts and scratched glassware. **This procedure should not be performed if no one else is in the laboratory.**

7.3.1 Fill the condenser on the diazomethane generator with dry ice and add acetone until the finger is about 1/3 full.

7.3.2 Add 10 mL of 95% ethanol and 5 g of potassium hydroxide in 8 mL of reagent water to the reaction flask.

7.3.3 Fill the separatory funnel with 5 g of Diazald in 45 mL diethyl ether.

NOTE: Vent tube should be submerged into a test tube containing ether.

7.3.4 Warm the reaction flask to 65 °C and slowly add the Diazald solution into it through the separatory funnel over a 20 minute period.

7.3.5 When the Diazald solution is used up, slowly add 10 mL of diethyl ether and continue to distill until the distillate is colorless.

7.3.6 The solution of diazomethane can be stored for up to 72 hours at 0 °C or until the brilliant yellow color begins to fade.

7.3.7 Add 2 mL of the diazomethane solution to the receiver with the dried sample extract. Seal the extract with a Teflon stopper and store at room temperature for 30 minutes.

7.3.8 Destroy the unreacted diazomethane with 0.2 g of silicic acid.

7.3.9 Allow the extract to stand until the evolution of nitrogen gas has stopped, then rinse the walls of receiver tube with 500 uL hexane.

7.3.10 Transfer the extract to a culture tube calibrated to 25 mL with three 2 mL rinses of hexane. Pull a 0.5 mL aliquot and dilute to 1 mL in an autosampler vial. This will equal a 50 mL final volume.

7.3.11 Methylated extracts must be analyzed immediately to minimize trans-esterification and other reactions that may occur.

8.0 QUALITY CONTROL

8.1 Extract two blanks with each sample batch, or two blanks for every 20 samples extracted, whichever is more frequent. Once a batch has been completed, retain one blank; send only one blank with each batch to the Gas Chromatography Department. The second blank is analyzed only if the first is damaged or lost. The second blank is not used to replace the first blank if the first blank fails QC criteria.

8.2 Extract a matrix spike/matrix spike duplicate pair for every 20 samples, or batch size 10 or more.

8.3 Extract an LCS for every batch of samples at a frequency of at least 1 per 20 samples.

9.0 INTERFERENCES

Glassware must be scrupulously cleaned and acid-rinsed to prevent interferences from arising.

10.0 TABLE(S)

Table 1
Extractable Target Compounds Using Diazomethane

COMPOUND
2,4-D
Silvex (2,4,5-TP)
2,3,5-T
Dicamba
2,4-DB
Dalapon
MCPP
MCPA
Dichloroprop
Dinoseb
Pentachlorophenol

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: OP-014-01
Initial Date: 2/1/91
Page 1 of 12
Date Revised: 6/7/95

Title: Organic Preparation of General Solutions.			
Prepared by: BES	Approved by:	Date: 6/8/95	QA Approval:

1.0 PURPOSE

This SOP describes the preparation of reagents used in the organic preparation laboratory.

2.0 APPLICATION

This procedure is applicable to all organic preparation lab extractions.

3.0 REFERENCES

3.1 EPA Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, 1982

3.2 CLP Statement of Work OLMO3.X

3.3 EPA SW-846, 3rd Edition, Final Update II

4.0 ASSOCIATED SOPs

None

5.0 REAGENTS

5.1 Sodium sulfate, granular

5.2 Sulfuric acid, concentrated

5.3 Reagent water

5.4 Hydrochloric acid, concentrated

5.5 Potassium hydroxide, reagent grade

5.6 Sodium hydroxide, reagent grade

5.7 Acetone, pesticide grade or equivalent

5.8 Methylene chloride, pesticide grade or equivalent

5.9 Hexane, pesticide grade or equivalent

6.0 PROCEDURE

6.1 Anhydrous sodium sulfate (powdered and granular): Sodium sulfate for drying sample extracts and samples is prepared by heating 2.5 Kg of sodium sulfate in a Pyrex dish at 400°C for 4 hours. Place the sodium sulfate in the muffle furnace while cool and heat to 400°C. After 4 hours at 400°C, turn the oven off and allow to cool to about 110°C before removing the Pyrex dish from the furnace. Place the sodium sulfate in a sealed glass jar labeled, "baked sodium sulfate".

6.2 Acidified sodium sulfate: Sodium sulfate for drying acid herbicides is prepared by making a slurry (syrup consistency) of 1 Kg granular sodium sulfate (which has been treated at 400°C as described in Section 6.1) with diethyl ether. To the slurry add 1.0 mL of concentrated sulfuric acid and mix well with a glass rod. Place the acidified slurry in a Pyrex dish and evaporate off the ether under a hood on a hot plate set at its lowest setting. Allow to dry with occasional mixing to ensure all the ether is gone before placing into the oven to dry at 200°C over night. Keep the sodium sulfate in a sealed glass jar, labeled : "Acid sodium sulfate, use for herbicides only".

6.3 1:1 Sulfuric acid for adjusting the pH of water samples: Slowly add 500 mL of concentrated sulfuric acid to 500 mL of reagent water in a 2 liter Erlenmeyer flask. Stopper the sink and use it as a cool water bath during mixing as this process will generate heat. Allow the solution to cool and store in a bottle with a Teflon-lined lid. Shelf life of the reagent is 1 year. Label the reagent: "1:1 H2SO4 - sulfuric acid".

6.4 1:3 Sulfuric acid solution to adjust the pH of extracts: Slowly add 25 mL concentrated sulfuric acid to 75 mL reagent water in a beaker and mix well. Allow the solution to cool and store in a bottle with a Teflon-lined lid. Cool to 4°C. Prepare just prior to use. Shelf life of the reagent is 1 year.

6.5 1:9 Hydrochloric acid solution to adjust the pH of solid samples for herbicide extraction: Add 20 mL of hydrochloric acid to 180 mL of reagent water. Keep solution in a bottle with a Teflon-lined lid. Shelf life of the reagent is 1 year. Label using blue tape.

6.6 37% Potassium hydroxide solution for hydrolysis of acid herbicides: Dissolve 37 g of potassium hydroxide in 50 mL reagent water. Allow the solution to cool and dilute to 100 mL with reagent water. Store the solution in a sealed Teflon bottle. Shelf life of the reagent is 1 year. Label using blue tape.

6.7 10 N Sodium hydroxide solution to adjust the pH of water samples: Dissolve 400 g of sodium hydroxide in 1000 mL reagent water in a large Erlenmeyer flask. Allow the solution to cool and dilute to 1000 mL with reagent water. Store the solution in a sealed Teflon bottle. Shelf life of the reagent is 1 year.

6.8 5% Sodium sulfate solution for acid herbicide preparations: Dissolve 10 g of sodium sulfate (treated at 400°C) in 100 mL reagent water and dilute to 200 mL. Store reagent in a glass bottle with a Teflon lined lid. Shelf life of the reagent is 1 year.

6.9 1:1 Acetone/methylene chloride extraction solvent: Add 2 liters of acetone to 2 liters of methylene chloride in a 4 liter glass reagent bottle with a Teflon lid. Shelf life of the reagent is 1 year.

6.10 10:90 Acetone/hexane elution solvent: Accurately measure 100 mL of acetone into about 30 mL of hexane in a 1000 mL volumetric flask and dilute to volume with hexane. Store the solution in an amber glass bottle with a Teflon-lined lid. Shelf life of the reagent is 1 year. Do not shake when making mixture!

7.0 Quality Control

7.1 Each reagent container will have a tag with the name of the reagent, concentration, preparation date, expiration date, ID number and initials of the preparer.

7.2 Since reagents are used up so rapidly expiration dates never come into consideration. Therefore we do not track in a reagent preparation logbook.

ROSS	SOP Standard Operating Procedure	SOP No: OP-015-00 Inital Date: 2/1/91 Page 1 of 4 Date Revised:

Title:

Preventive Maintenance for Organic Preparation Laboratory Equipment.

Prepared by:	Approved by:	Date:	QC Approval:
Gary E Olson	C H Caldwell	6/3/91	

1.0 PURPOSE

This procedure is intended for the routine upkeep of instrumentation to prevent damage and minimize down time.

2.0 APPLICATION

This procedure applies to all instruments in the organic preparation laboratory.

3.0 REFERENCES

The manuals for all the organic preparation instruments are kept in the lab for easy reference. The manuals should be consulted for details on performing all maintenance.

4.0 ASSOCIATED SOPs

None.

5.0 PROCEDURE

5.1 Sonicator

5.1.1 Clean exterior surfaces once a month with a damp cloth.

5.1.2 Wipe out the inside of the sound box after each use with a damp cloth.

5.1.3 Remove residue from the probe with a damp cloth and rinse with acetone.

5.1.4 Inspect the probe extender for erosion. Existing erosion will increase the rate of future erosion. After every 5 hours of use the tip should be polished with emery cloth.

5.2 Zymark TurboVap Evaporator

5.2.1 Every two months drain and clean the bath with a sponge to remove any bacteria. Add fresh reagent water and 15 drops of Clear Bath to inhibit biological growth.

5.2.2 Every two months check all nitrogen gas lines and connectors for leaks with Snoop.

5.2.3 Daily add additional water as needed to replace water lost during use though evaporation.

5.2.4 Clean all exterior surfaces monthly with a damp cloth.

5.3 N-EVAP Evaporator

5.3.1 Every two months drain and clean bath with a sponge to remove any bacteria. Add fresh reagent water and 15 drops of Clear Bath to inhibit biological growth.

5.3.2 Daily add additional water as needed to replace water lost during use through evaporation.

5.3.3 Check and replace broken tubing every two months or sooner if required.

5.3.4 Clean all exterior surfaces monthly with a damp cloth.

5.4 Centrifuge

5.4.1 Clean all exterior surfaces monthly with a damp cloth.

5.4.2 After each use, clean interior surfaces with a damp cloth to prevent corrosion. If required, a mild soap or detergent such as Liqui-Nox may be employed.

5.5 Gel Permeation Chromatograph

5.5.1 Clean all exterior surfaces monthly or before each use to prevent small particals from clogging valves and transfer lines.

5.5.2 Reseat all easily accessable cable connectors at least semiannually.

6.0 Documentation

6.1 All maintenance procedures will be recorded in a maintenance log book which is stored near each instrument. Information in the log book will include the date and procedure performed and the initials of the analyst.

SPARE PARTS LIST

Sonicator, Heat Systems Model XL2020

Fuse	Cat. # GMA10	10A 115V

Zymark TuboVap Evaporator

Fuse	Cat. # 38904	6A 250V Slo-Blo
Sensor	Cat. # 44-191	

N-EVAP Evaporator

1/4" Silicone Tubing	Cat. # 10613
Thermostat	Cat. # 10823

Centrifuge, Mistral 3000E

Fuse	10A 250V 326

GPC

Pen	Cat. # 416-902	
Paper	Cat. # 416-903	
Fuse	Cat. # 367-008	
Fuse	Cat. # 367-220	
Fuse		1A 240V/220V
Fuse		115 VAC-3AG Slo-Blo 0.3a
Fuse		230 VAC-3AG Slo-Blo 0.15a
15ml Vial	Cat. # 236-660	

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date
[illegible]	8/27/92

Terminated by	Date

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Completion of the Extraction Summary Form				
Prepared by: BES	Approved by: CLR	Date: 6/8/95	QA Approval:	

1.0 PURPOSE

This method describes the procedure to complete the organic laboratory extraction summary form and blank logbook.

2.0 APPLICATION

This procedure applies to all organic laboratory extractions.

3.0 REFERENCES

Contract Lab Program, Statement of Work OLMO3.X

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Prior to starting any analysis, read the work order summary carefully to ensure the proper extraction is performed.

5.2 Check the sample container to be sure the client ID is the same as on the work order.

5.3 Transfer the information on the work order to the extraction summary sheet (See Section 7.0, Figure 1, for an example extraction summary sheet) beginning with the client and project ID. All CLP projects will include the Case and SDG along with the work order number.

5.4 Record all the appropriate dates and the sample matrix. Record initials of the (1) staff writing up the extraction summary, (2) staff who spike the samples both for the surrogate and matrix spikes, (3) staff who perform the extraction, and (4) staff who finalize the extracts.

5.5 Check off or circle which of the analytes the extraction applies to, the method of extraction, and clean-up procedure. Include the lot number of each solvent and reagent used in the extraction and clean-up, and column ID of the GPC, if used.

5.6 Record the client ID and lab ID from the work order. Record the sample weight or volume used immediately after the measurement is taken. For all water samples, include the initial pH and adjusted pH. All CLP solid samples require a pH and moisture determination. Record the initial extract concentration

volume and the aliquot volume taken for a clean-up along with the extract final volume following the clean-up.

5.7 For extracts which undergo GPC, fill out the summary sheet following the GPC example sheet hanging in the OP lab.

5.8 Record the matrix spike and surrogate spike IDs and volume used for the sample and check sample.

5.9 In the Note section, record blanks and check samples that apply to other work orders.

5.10 Record the notebook number for all CLP projects.

5.11 Record the GPC check associated with the samples, if applicable, in the GPC check box.

5.12 Blank IDs for CLP and SAS samples are recorded in the CLP Blank Logbook (See Section 7.0, Figure 2 for an example CLP Blank Logbook sheet). The blanks for each SDG are recorded on a separate page and each blank will have a unique ID within an SDG. The prefix for semivolatile organic blanks is "S"; "P" is for Pesticide/PCB blanks. The suffixes are an alpha-numeric progression from A0, A1, A2 to Z8, Z9.

Examples:

Case/SDG # 1	**Case/SDG # 18**
SBLKA0	SBLKZ0
SBLKA1	SBLKZ1
PBLKA2	PBLKZ3
PBLKA3	
SBLKA4	

Case/SDG # 2	**Case/SDG # 19**
SBLKB0	PBLKA0
SBLKB1	SBLKA1

The unused portion of the log book page is "Z'ed" out and initialed. The Case/SDG are noted at the bottom of the log book page. All information under each heading must be completed including surrogate ID and volume used under the "NOTE/SURROGATE" column.

5.13 Entries in the log book for non-CLP blanks and LCS samples are recorded numerically. The prefix to the number is to identify the blank for a specific analysis.

Blank ID	
Herb 2014	Herbicide blank
Pest 2015	Pesticide blank
BNA 2016	BNA blank

The prefix to identify LCS samples is "CS" to prevent any confusion with blanks. The spike solution used for the LCS is identified on the extraction summaries, as well as in the Check Sample Log Book.

Check Sample ID

CS 1045
CS 1046
CS 1047

All information under each heading in both the Organic Blank Log Book and the LCS Log Book must be complete, including spike solution ID's and volumes.

6.0 QUALITY CONTROL

6.1 All summaries must be reviewed, to verify all the necessary information is included on the summary, by an analyst other than the person who completed the summary. After reviewing the summary, the reviewer will initial and date the summary.

6.2 All unused portions of the summary will be "Z'ed" out as required by CLP.

7.0 FIGURES

Figure 1
Example Extraction Summary Form

PESTICIDE ORGANIC EXTRACTION SUMMARY

CASE/SDG:	Date Ext'd:	Date Completed:	Matrix:
Client:	Ext'd By:	Checker:	
Work Order:	Date Rec'd:	Extraction Location:	

Analytes	Extraction Method			Clean-up	Solvent Lot #	
O-Pest _____	3550cons____	3540cssh____	TC-4____	GPC _____	Ch2Cl2_____	Other____
Pest/PCB _____	3520cont____	CLPcont_____	608____	Florisil _____	Acetone _____	
	3510cssf	CLPscns		Sulfur	Hexane	

Client ID	Lab ID	Number	Amount Extracted	pH Int'l	pH Adjusted 1st	pH Adjusted 2nd	Int'l Vol. (mL)	GPC Aliquot (mL)	Vol After GPC		Florisil Aliquot	Final Vol (mL)

Surr Std	Amount	MS Std	MSD Std	Amount	LCS Std	Amount

GPC Column ID	Pest/PCB GPC Check

Notebook #

Ross Analytical Notes:

PAGE #

Figure 2
Example CLP Blank Logbook Sheet

BLANK ID	MATRIX	EXTRACTION METHOD	ANALYSIS	SURROGATE ADDED	START DATE	INITIAL

ROSS ANALTICAL SERVIVES INC.

LOGBK #

PAGE:

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Microdistillation of Alcohols from Solid and Liquid Samples			
Prepared by:	Approved by:	Date: 2/10/97	QA Approval:

1.0 PURPOSE

This procedure describes the method used to distill alcohol from solid and liquid samples.

2.0 APPLICATION

This method applies to all alcohol samples.

3.0 REFERENCES

None

4.0 ASSOCIATED SOP

GV-001, Analysis for Alcohols

5.0 REAGENTS/EQUIPMENT

5.1 Reagents

5.1.1 Reagent water, Labconco or equivalent

5.1.2 Alcohol surrogate solution, isopropanol at 785.05 ug/mL

5.1.3 Alcohol standard spike solution

5.1.4 Internal standard solution, n-propanol at 805.3 ug/mL

5.2 Equipment

5.2.1 Round bottom flask, threaded neck, 100 mL

5.2.2 Boiling chips

5.2.3 Distillation column, 14/20 upper joint, straight bottom

5.2.4 Glass beads

5.2.5 Adapter tube to fit hose, fitting, glass, 14/20 x ¼ inch

Figure 2
Example CLP Blank Logbook Sheet

BLANK ID	MATRIX	EXTRACTION METHOD	ANALYSIS	SURROGATE ADDED	START DATE	INITIAL

ROSS ANALTICAL SERVIVES INC.

LOGBK #

[redacted]

PAGE:

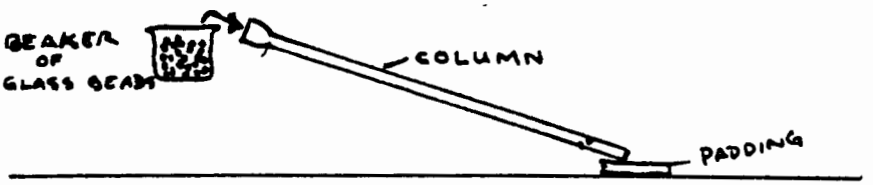
5.2.6 Teflon transfer line with ¼ inch SS or brass fitting

5.2.7 Calibrated vial, 1.0 mL

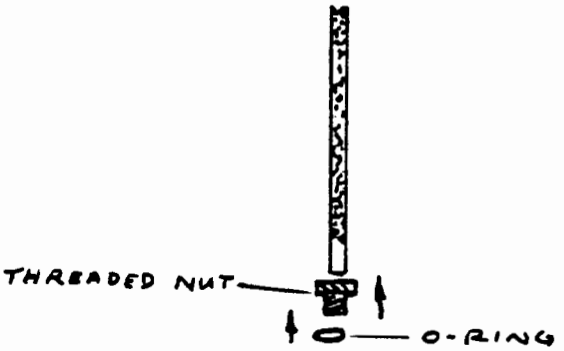
6.0 PROCEDURE

6.3 Distillation Procedure

6.3.1 Fill column with glass beads to bottom of ground glass joint. Pour beads at an angle of about 30 degrees when filling column to prevent breakage of the bead retainers.



6.3.2 Place filled column in holder in vertical position. Lift column and place threaded nut over column. Wet an O-ring (PTFE) with water and slide over lower part of column about ¼ inch from bottom.



6.3.3 Weigh sample, 30 g, or measure 40 mL liquid sample into 100 mL flask. Use 40 mL of reagent water for blank check and CCV (continuing calibration verification - identical to LCS).

6.3.4 Add boiling chips to all flasks.

6.3.5 Add appropriate amounts of surrogate, internal standard, and spike to the proper samples.

6.3.6 Assemble column and flask using a small amount of water to allow the O-ring to seat. Tighten threaded nut to contact O-ring and turn 1/8 turn more after contact.

6.3.7 Calibrate vials with 1.0 mL water.

6.3.8 Assemble glass adapter to fitting and tighten to finger tightness. Wet ground glass portion of adapter (dip adapter into a small beaker of water) and drop into place. Press in firmly, but do not twist more than 1/16 turn. Add retainer clip.

6.3.9 Place insulation in place on columns.

6.3.10 Turn on heat to maximum amount and bring to a vigorous boil.

6.3.11 Make sure tubing is in the vial.

6.3.12 **Stop process as soon as steam breaks through the tubing**. Approximately 0.25 - 0.50 mL will be in the vial.

6.3.13 Bring vial(s) to 1.0 mL with reagent water.

NOTE: GROUND GLASS EQUIPMENT MAY BE USED FOR LIQUIDS ONLY.

7.0 Quality Control

7.1 Extract a blank with each set of samples distilled or every 20 samples, whichever is more frequent.

7.2 Analyze a check sample and CCV every set of samples distilled or every 20 samples, whichever is more frequent.

7.3 Analyze an MS/MSD pair every set of samples distilled or every 20 samples, whichever is more frequent.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Solvent Extract Waste Disposal in the Organic Preparation Lab			
Prepared by: BES	Approved by: CAR	Date: 9/26/94	QA Approval:

1.0 PURPOSE

This procedure is to ensure that extract wastes generated in the Organic Preparation (OP) lab are properly disposed.

2.0 APPLICATION

These wastes take on 4 forms, solids - as in PCB screens, neutral solvents - as in pesticide/PCB extracts, acid solvents - as in PCB extracts, and PCB wastes over 50 ppm.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

OP-001 B/N/A Extraction of Water
OP-002 Pesticide and PCB Water Extraction
OP-005 CLP Semivolatile Organic Extractions
OP-007 Extraction of Soils and Wastes for Pesticides and PCBs for Non-CLP
OP-008 CLP Pesticides/PCB Extraction
OP-009 Extraction of Acid Herbicides from Solid Samples
OP-010 PCB Oil and Liquid Waste Extraction
OP-011 Extraction of BNA's in Soils and Sludges
OP-013 Extraction of Acid Herbicides from Water Samples
OP-018 Extraction of Organophosphorus Pesticides from Water Samples
OP-019 Extraction of Organophosphorus Pesticides from Soil Samples
OP-024 PCB Solid Waste Extraction
OP-025 BNA Liquid Waste Dilution
OP-026 PUF Sample Extraction
OP-032 Extraction of PCBs from Wipes or Swabs

5.0 EQUIPMENT

5.1 Lab jacket

5.2 Latex gloves

5.3 Solvent waste bottles

5.4 Short stem, wide mouth funnel

5.5 Deionized water in a squirt bottle

5.6 Wastewater container

5.7 Non-hazardous waste container

5.8 Aluminum drying pan

5.9 Fume hood

5.10 Spill control equipment

6.0 SOLUTIONS

6.1 Hexane - non-Spectro grade or equivalent

6.2 Methylene chloride - pesticide grade or equivalent

6.3 Acetone - pesticide grade or equivalent

6.4 Deionized water

6.5 Concentrated hydrochloric acid

6.6 Ether - herbicide grade or equivalent

6.7 Cyclohexane - pesticide grade or equivalent

7.0 PROCEDURE

NOTE: Perform this procedure under a well ventilated fume hood.

7.1 Time Frame for Disposal

7.1.1 Solvent extracts - Dispose of all solvent extracts which are beyond three weeks past extraction for commercial clients, retain newer extracts. CLP extracts are held for 6 months. Never discard an extract before analysis.

7.1.2 Acid waste - Dispose of acid waste which are 48 hours beyond the time when the work order has been reported.

7.1.3 Solid screen extracts - Dispose of solid screen extracts which are 48 hours beyond the time when the work order has been reported.

7.2 Separate all wastes according to the time frames and categories above. Also, separate any samples which contain PCBs above 50 ppm into a fourth grouping (See 7.11).

7.3 Dispose of all sulfuric acid wastes by rinsing the test tubes three times with deionized water and dumping the deionized water into a wastewater container. Place empty test tubes into an empty solvent bottle.

7.4 Neutralize wastewater by following SOP No.: OP-039.

7.5 Dispose of all solvent extracts by dumping them through the funnel into the appropriate solvent waste bottles e.g., B/N/As into halogenated wastes, less than or equal to 50 ppm PCB extracts into non-halogenated wastes. Separate any samples which contain PCBs above 50 ppm into a fourth grouping. Place test tubes into an empty solvent bottle.

7.6 Dispose of solid screen extracts by draining solvent into solvent waste bottle and then putting solids to dry into an aluminum drying pan placed in a fume hood. Put glass jars and lids into a non-hazardous waste container.

7.7 After solids have dried, place them into a non-hazardous waste container.

7.8 Take all glassware out into the warehouse in order for the glass to be ground.

7.9 Place all non-hazardous waste into the non-hazardous bucket.

7.10 Take full waste solvent bottles into the used solvent cabinets.

7.11 Dispose of all PCB extracts with greater than 50 ppm of Aroclor into the PCB waste canister in the used solvent cabinet. Put test tubes in the PCB waste container in the Organic Preparation lab.

8.0 SAFETY

8.1 Wear your personal protective equipment which includes lab coats, latex gloves, face shields, and apron.

8.2 Work under a fume hood.

8.3 Make sure there is someone else present in the room while disposing of wastes.

8.4 Know where and how to use all spill equipment in case of a spill.

8.5 Waste containers must be closed at all times except when adding or removing wastes.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Wastewater Disposal in the Organic Preparation Lab			
Prepared by: BES	Approved by: CAH	Date: 9/26/94	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to provide instructions on how to dispose of wastewater generated in the Organic Preparation (OP) lab.

2.0 APPLICATION

This procedure applies to aqueous wastes that are generated as a result of sample preparation and analysis. This is a laboratory procedure that is incorporated as a part of all other standard operating procedures (SOPs) that generate aqueous wastes.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

OP-001 B/N/A Extraction of Water
OP-002 Pesticide and PCB Water Extraction
OP-013 Extraction of Acid Herbicides from Water
OP-018 Extraction of Organophosphorus Pesticides from Water Samples
OP-021 Extraction of Water Samples for Petroleum Hydrocarbons
OP-031 Microdistillation of Alcohols from Solid and Liquid Samples
OP-037 Solvent Extract Waste Disposal in Organic Preparation Lab

5.0 PROCEDURE

5.1 Equipment and Apparatus

5.1.1 Goggles

5.1.2 Gloves

5.1.3 Lab jacket

5.1.4 5 Gallon work-place accumulation container (plastic) with screw top.

5.1.5 Mixing stick

5.1.6 Spill kit

5.1.7 Well-ventilated fume hood.

5.2 Reagents

5.2.1 Sodium hydroxide (granular pellets)

5.2.2 pH Paper

5.2.3 Concentrated hydrochloric acid

5.3 Procedure

5.3.1 Wear a minimum of lab jacket, gloves and goggles before handling low pH materials.

5.3.2 Place the 5 gallon accumulation container near or in a hood before opening. Screw open the top.

5.3.3 Mix the solution with the mixing stick and check pH with pH paper.

5.3.4 If the pH is less than pH 5, add a small quantity of sodium hydroxide while mixing. Continue to mix until the sodium hydroxide is dissolved. Re-check pH.

5.3.5 Continue this procedure until the solution has a pH in the range of 5 - 8.

5.3.6 If too much sodium hydroxide is added and the pH is too high, add small quantities of hydrochloric acid while mixing. Check pH until solution is in the proper range.

5.3.7 Continually monitor the temperature by feeling the side of the pail. If the mixture becomes hot, allow to cool before making additional pH adjustments.

5.3.8 If pH is greater than pH 8, add a small quantity of concentrated hydrochloric acid while mixing. Re-check pH.

5.3.9 Continue this procedure until the solution has a pH in the range of 5-8.

5.3.10 If too much hydrochloric acid is added, add small quantities of sodium hydroxide while mixing, then check pH until solution is in the proper range.

5.3.11 Continually monitor the temperature by feeling the side of the pail. If the mixture becomes hot, allow to cool before making additional pH adjustments.

5.3.12 After adjusting the pH, the closed satellite accumulation container must be taken to the waste accumulation area in a high walled cart. The SHE group will pour the wastes from the satellite container into waste drums.

CAUTION: Do not over-fill drum.

6.0 SAFETY

6.1 Always wear the personnel protection equipment listed in Section 5.1 when performing pH adjustments.

6.2 Perform all operations in well-ventilated fume hood.

6.3 This operation should only be done when others are present in the same room.

6.4 Keep spill control equipment near this operation.

6.5 Waste containers must be kept closed except when adding or removing waste.

Ross Analytical Services, Inc.

STANDARD OPERATING PROCEDURE

SOP No.: CW-004-02
Initial Date: 2/1/91

Date Revised: 05/16/96

Title: Total Cyanide			
Prepared by:	Approved by:	Date: 5/28/96	QA Approval:

1.0 PURPOSE

To describe the distillation procedure and manual colorimetric analysis of samples for total cyanide by EPA Methods 9010A and 335.2.

2.0 APPLICATION

This procedure is applicable to the determination of total cyanide in waters, wastewaters, sludges, soils, and wastes. Both the conventional maxi-scale and automated midi-scale distillations are described.

Note that there are several different procedures for sulfide removal described in various methods. Although all agree that the most satisfactory procedure is screening and pretreatment at the time of collection, this is not likely to occur in most cases; consequently, sulfide removal must take place at the time of analysis. To simplify the procedure and allow consolidation of two very similar methods, 9010A and 335.2, RASI has decided on a single method of sulfide removal based on its effectiveness and ease of use. Bismuth nitrate is added to samples containing sulfide during the distillation as described in Method 9010A. This is a minor departure from Method 335.2, which uses an impinger of lead acetate scrubbing solution to remove sulfide.

3.0 REFERENCES

"Cyanide: Titrimetric Method", Standard Methods for the Examination of Water and Wastewater, 19th Edition, Method 4500-CN^- D, Approved 1990

"Cyanide, Total", Methods for the Chemical Analysis of Water and Wastes (EPA-600), Revision 1983, Method 335.2, Revision 1980

"Total and Amenable Cyanide", Test Methods for Evaluating Solid Waste (SW-846), Method 9010A. Revision July 1992

AP1214 Automated Cyanide Preparation System User's Manual, Revision E, Leeman Labs, Inc.

4.0 ASSOCIATED SOPs

CW-005, Cyanides Amenable to Chlorination

CW-006, Use of the UV/Vis Spectrophotometer

CW-065, Total Cyanide by CLP Protocol

5.0 EQUIPMENT

5.1 Distillation apparatus, one of the following:

5.1.1 Maxi- scale reflux distillation apparatus as shown in Section 10.0, Figure 1

5.1.2 Leeman Labs AP1214 Automated Cyanide Preparation System as shown in Section 10.0, Figure 2

5.2 Hitachi U-2000 UV/Vis spectrophotometer, set at 578 nm

5.4 Nessler tubes or polypropylene centrifuge tubes, 50 mL, with caps

5.5 Volumetric flasks, 250 mL

5.6 Lead acetate test paper

5.7 KI-starch test paper

6.0 REAGENTS

6.1 NaOH solution, 1.25 N - Dissolve 50 g NaOH pellets in deionized water and dilute to one liter. Store in a plastic container at room temperature. This solution is good for one year.

6.2 Sulfuric acid, 1:1 - Slowly add 500 mL of concentrated H_2SO_4 to 500 mL deionized water. Let cool and store in glass container at room temperature. This solution is good for one year.

6.3 Sulfamic acid (0.4 N) - Dissolve 40 g H_2NSO_3H in one liter of deionized water. Store in plastic container at room temperature. This solution is good for one year.

6.4 Magnesium chloride solution - Dissolve 510 g of $MgCl_2 \cdot 6H_2O$ in about 700 mL of deionized water and dilute to 1 L. Store in a glass container at room temperature. This solution is good for one year.

6.5 Sodium dihydrogen phosphate, 1M - Dissolve 138 g of $NaH_2PO_4 \cdot H_2O$ in deionized water and dilute to one liter. Keep refrigerated in a plastic container. This solution is good for one year.

6.6 Chloramine-T solution - Dissolve 1 g of chloramine-T in 100 mL deionized water and refrigerate until ready for use. Prepare fresh weekly.

6.7 Pyridine-barbituric acid color reagent - Prepare this reagent in a hood. Place 15 g of barbituric acid in a 250 mL volumetric flask and add just enough deionized water to wash the sides of the flask and wet the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of concentrated HCl, mix and cool to room temperature. Dilute to volume and mix. This reagent can be used for six months or until precipitate forms. Keep solution refrigerated in an amber glass container.

6.8 Stock cyanide solution, ~1000 mg/L - Dissolve 2.51 g KCN and 2 g KOH (1.6 g NaOH may be substituted) in 900 mL deionized water. Dilute to one liter and store in an amber glass container. Standardize against 0.020 N $AgNO_3$: Place 10.0 mL stock solution and 20 mL 1.25 N NaOH solution in a 500-mL erlenmeyer flask and dilute to 100 mL with deionized water. Add 10-12 drops of rhodanine indicator and titrate with silver nitrate to the first color change from canary yellow to a salmon hue. The titration must be performed slowly to allow the solution to reach equilibrium after each addition and avoid overshooting the endpoint. As a blank, titrate 100 mL of 0.25 N NaOH to the same endpoint; subtract the volume of titrant for the blank from the volume needed for the stock solution before using in the following calculation:

$$\text{mg/L CN, stock solution} = \frac{(\text{mL } AgNO_3)(\text{N } AgNO_3)(52.02)(1000)}{\text{mL cyanide stock}}$$

Perform the standardization in triplicate and average the three values. Standardize biweekly and record in Standard Logbook.

6.9 Stock cyanide LCS solution, ~1000 mg/L - Dissolve 1.88 g NaCN and 1.6 g NaOH pellets in 900 mL deionized water. Dilute to one liter and store in an amber glass container at room temperature. Standardize biweekly as above in §6.8. Record in Standard Logbook.

6.10 Working cyanide standard solution, 10 mg/L - Using the stock concentration determined above (§6.8), calculate the volume of stock which must be diluted to 100 mL to make a working concentration of 10 mg/L:

$$\text{mL stock required} = \frac{1000}{\text{concentration of stock, mg/L}}$$

Pipet the amount calculated into a 100 mL volumetric flask, add 20 mL 1.25 N NaOH, and bring up to volume with deionized water. *Prepare fresh daily.*

6.11 Working cyanide LCS solution, 10 mg/L - Using the LCS stock concentration determined above (§6.9), calculate the volume of stock which must be diluted to 100 mL to make a working concentration of 10 mg/L:

$$\text{mL stock required} = \frac{1000}{\text{concentration of stock, mg/L}}$$

Pipet the amount calculated into a 100 mL volumetric flask, add 20 mL 1.25 N NaOH, and bring up to volume with deionized water. *Prepare fresh daily.*

6.12 Standard silver nitrate solution, 0.020 N - Commercially purchased, certified NIST traceable.

6.13 Rhodanine indicator - Dissolve 20 mg of p-dimethyl-amino-benzalrhodanine in 100 mL acetone. Commercially purchased indicator may also be used.

6.14 NaOH solution, 0.25 N - Dissolve 10 g NaOH pellets in deionized water and dilute to one liter. Store in plastic container at room temperature. This solution is good for one year.

6.15 Ascorbic acid, reagent grade

6.16 Bismuth nitrate solution (0.062M) - Dissolve 30 g $Bi(NO_3)_3 \cdot 5H_2O$ in 100 mL of deionized water. While stirring, add 250 mL of glacial acetic acid, CH_3COOH. Stir until dissolved and dilute to 1 liter with deionized water.

6.17 Acetate buffer, pH 4 - commercially purchased.

7.0 PROCEDURE

7.1 Preliminary determinations

7.1.1 Test for oxidizing agents in aqueous samples by placing a drop or two of sample on KI-starch paper. A blue color indicates the presence of an oxidizer. Record result on the bench sheet.

7.1.2 Test for sulfides in aqueous samples by placing a drop of sample on lead acetate paper pre-moistened with acetate buffer, pH 4. A black color indicates the presence of sulfides. Record result on the bench sheet.

7.1.3 Test for sulfides in non-aqueous samples as follows: Place 5 grams of sample in a 50 mL beaker, add approximately 5 mL water and slurry briefly. Attach a piece of lead acetate paper to the bottom of a watch glass with a little water. Add several drops of concentrated HCl to the slurry in the beaker, then quickly cover with the watch glass. Allow to sit undisturbed for several minutes. If sulfide is present, the paper will darken. Record result on the bench sheet.

7.2 Distillation with conventional maxi-scale units

NOTE: If sulfides are present in the samples, the calibration standards must be distilled in the same manner. See §7.4.1.1.

7.2.1 Place 500 mL of an aqueous sample or an aliquot diluted to 500 mL into the boiling flask. For soils or non-aqueous liquids use 5 g of sample and add 500 mL deionized water to the flask. Add boiling chips as necessary.

7.2.2 If an oxidizing agent was found to be present in §7.1.1, add ascorbic acid to the flask, a little at a time, until sample produces no color on the indicator paper. Alternately, the entire sample may be treated by adding the ascorbic acid to the sample bottle in the same manner, prior to measuring out an aliquot for analysis. When the sample no longer tests positive, add an additional 0.6 grams per liter of sample. A slight excess is not a problem but avoid a large excess.

7.2.3 Place 50 mL of 1.25 N NaOH in the absorber.

7.2.4 Connect apparatus as in Figure 1.

7.2.5 If using stills with two scrubber/absorbers in series, place 25 mL of deionized water in the first scrubber and insert between the boiling flask and NaOH absorber.

7.2.6 Turn on the vacuum pump and the condenser water. Adjust the vacuum so that approximately three bubbles of air per second enter the boiling flask through the air inlet tube.

7.2.7 If sulfides were found to be present in §7.1.2, add 50 mL bismuth nitrate solution through the air inlet tube. Allow to mix for 3 minutes.

7.2.8 Add 50 mL sulfamic acid through the air inlet tube. Mix for 3 minutes. This reagent removes interferences from nitrates and nitrites.

7.2.9 *Slowly* add 50 mL 1:1 H_2SO_4 (or 25 mL concentrated) through the air inlet tube. Rinse the tube with small portions of deionized water and allow to mix for approximately 3 minutes.

7.2.10 Pour 20 mL of $MgCl_2$ solution into the air inlet tube, rinse with small portions of deionized water and allow to mix.

7.2.11 Heat the solution to boiling and allow to reflux for one hour. Turn heat off and continue air flow for 15 minutes to cool the flask. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

7.2.12 Pour the solution from the absorber into a 250 mL volumetric flask. Rinse the absorber with deionized water and add to the flask. Pour the contents of the scrubber containing deionized water (if used) into the flask as well, with rinsing. Dilute to volume with deionized water.

7.2.13 Turn off condenser water.

7.3 Distillation from midi-scale unit

NOTE: If sulfides are present in the samples, the calibration standards must be distilled in the same manner. See §7.4.1.1.

7.3.1 Place 50 mL of an aqueous sample or an aliquot diluted to 50 mL into the distillation flask. For solids or non-aqueous liquids use 1 g of sample and add 50 mL deionized water to the flask. Add boiling chips as necessary.

7.3.2 If an oxidizing agent was found to be present in §7.1.1, add ascorbic acid to the flask, a little at a time, until sample produces no color on the indicator paper. Alternately, the entire sample may be treated by adding the ascorbic acid to the sample bottle in the same manner, prior to measuring out an aliquot for analysis. When the sample no longer tests positive, add an additional 0.6 grams per liter of sample. A slight excess is not a problem but avoid a large excess.

7.3.3 Add 50 mL of 0.25 N NaOH to the absorber flask.

7.3.4 Drain vacuum trap bottle and fill with 300 mL 1.25 N NaOH.

7.3.5 Ensure that all glassware and tubing connections are assembled as in Figure 2 and are gas tight.

7.3.6 Turn on condenser water and vacuum pump.

7.3.7 Mass Flow Meter Calibration: Turn on the Main Unit. Adjust FLOW knob to display the total number of channels active for sample preparation (default = 14 channels). Press the CALIBRATE button on the main unit front panel. With the CALIBRATE light steady on, observe the sample flask in the channel indicated on the display. Adjust the FLOW knob to show three bubbles/second in the flask. When the three bubbles/second rate is achieved for the channel, press the CALIBRATE button again. If the flow rate is not correct at this point, the instrument will beep and "BAD" will appear on the display. Continue adjusting the flow until the proper rate is achieved. Repeat this process until all necessary channels are calibrated. Press HALT when calibration is complete.

7.3.8 If sulfides were found to be present in §7.1.2, add 5.0 mL bismuth nitrate solution through the air inlet tube of the distillation head, and allow to mix for 3 minutes.

7.3.9 Add 5 mL of sulfamic acid through the air inlet tube, and allow to mix for 3 minutes.

7.3.10 *Slowly* add 5 mL of 1:1 H_2SO_4 (or 2.5 mL of concentrated) through the air inlet tube, rinse down with a little deionized water, and allow to mix for 3 minutes.

7.3.11 Add 2 mL of magnesium chloride solution through the air inlet tube and rinse down with a little deionized water.

7.3.12 Press the DIGEST button on the main unit front panel. The DIGEST button will begin to blink. The display will count down five minutes of vacuum flow, allowing the samples and reagents to mix thoroughly. At the end of five minutes, a low beep sounds, the DIGEST light stops blinking and "ACd" appears on the display.

7.3.13 After the five minute mixing period, press DIGEST again. The DIGEST light will begin to blink and the heating unit turns on.

7.3.14 Sample digestion begins and a 130 minute countdown is displayed, which includes warm-up, digestion and cool-down.

7.3.15 After a high "beep" sounds at the end of the cool-down period, ensure that the sample flasks are cool enough to handle, then turn off the vacuum pump.

7.3.16 Remove the microcondensor assembly from each gas absorbing flask and pour the flask's contents into a 50 mL plastic container. Do not rinse or dilute.

7.3.17 Turn off condenser water.

7.4 Analysis

7.4.1 Preparation of standards

7.4.1.1 For samples containing sulfide, standards must be distilled in the same manner as the samples (see §7.2 or 7.3), including the addition of bismuth nitrate. Standards distilled in this way will give a linear calibration curve, but as the concentration increases the recovery decreases. Distill four standards and a blank. Pipet the volumes below into the distillation flasks to achieve the concentrations noted.

Conventional Maxi Scale Distillation:

Volume of 10 mg/L Standard	Final Conc. of Distilled Standard
0.00 mL	0.000 mg/L
0.25	0.010
1.00	0.040
5.00	0.200
20.00	0.800

Midi Scale Distillation:

Volume of 10 mg/L Standard	Final Conc. of Distilled Standard
0 uL	0.000 mg/L
50	0.010
200	0.040
1000	0.200
4000	0.800

The distilled standards are stable for up to 14 days if kept refrigerated. The calibration may be performed using previously distilled standards which are less than 14 days old.

7.4.1.2 For samples not containing sulfide, use a digital pipettor to prepare the following series of undistilled standards. For each standard, pipet the given volume into a 50 mL Nessler tube. Add 25 mL of 0.25 N NaOH to each tube. NOTE: For calculations sake, volume analyzed is 25 mL.

Volume of 10 mg/L Standard	Concentration of Standard
0 uL	0.000 mg/L
25	0.010
100	0.040
250	0.100
500	0.200
1000	0.400
2000	0.800

Color the standards as in §7.4.3 to 7.4.6 below.

7.4.2 Place 25 mL of each sample and standard distillate (or an aliquot diluted to 25 mL with 0.25 N NaOH solution) into a Nessler tube.

7.4.3 Add 7.5 mL sodium dihydrogen phosphate solution. Cap tubes and mix.

7.4.4 Add 1 mL chloramine T solution. Cap tubes and mix. Wait one minute and test with KI-starch paper. If the test is negative, add 0.5 mL additional chloramine-T and wait another minute. Continue to add chloramine-T in 0.5 mL increments until an excess is maintained.

7.4.5 Add 2.5 mL pyridine-barbituric acid color reagent.

7.4.6 Dilute to volume (50 mL) with deionized water. Cap tubes and mix.

7.4.7 Allow 8 minutes for color development, then calibrate the spectrophotometer at 578 nm, using the standards prepared in §7.4.1. Refer to SOP CW-006 for use of the spectrophotometer.

7.4.8 Analyze the samples within 15 minutes of adding the pyridine-barbituric acid reagent. The run order is as follows:

Calibration Standards
ICV -- reread the 0.20 mg/L standard
ICB -- reread the blank standard
Distilled Check Standard (DCS) -- only if calibration standards were not distilled with samples
Prep Blank -- only if calibration standards were not distilled with samples
LCS -- either aqueous or solid
7 to 9 Samples -- count up to 10 analyses since ICB (MS/MSDs count as samples)
CCV -- reread the 0.20 mg/L standard
CCB -- reread the blank standard
Up to 10 samples
CCV and CCB -- after every 10 samples and at end of run

7.4.9 If a sample concentration is over the range of the calibration, dilute an aliquot of sample with 0.25 N NaOH and reanalyze. If the concentration is far beyond the calibration range (e.g., the sample turns nearly black and/or a precipitate forms upon addition of color reagents, or a 1:250 dilution of the distillate is still overrange), redistill the sample using a smaller initial aliquot.

7.5 Calculations

7.5.1 Concentrations from the instrument are read directly in mg/L.

7.5.2 For aqueous samples distilled with the midi-scale apparatus using a 50 mL aliquot require no further calculation. Diluted sample results should be multiplied by any dilution factor used.

7.5.3 Aqueous samples distilled from the maxi-scale apparatus using a 500 mL aliquot undergo a twofold concentration. Multiply the concentration readings from the instrument by 0.5. Multiply by any dilution factor used.

7.5.4 Calculate results for non-aqueous samples using this equation:

$$\text{Total CN, mg/Kg} = \frac{\text{mg/L reading} \times \text{final volume in mL} \times \text{dilution factor}}{\text{g sample}}$$

8.0 QUALITY CONTROL

8.1 The holding time for all cyanide samples is 14 days from collection, except for samples analyzed by CLP protocol, which have a holding time of 12 days from receipt (see SOP CW-065). All samples should be preserved with NaOH to achieve a pH of greater than 12.

8.2 Distill and analyze at least one reagent blank per analytical batch or 20 samples, whichever is more frequent. If standards are being distilled in the same batch with the samples, the blank standard serves as the prep blank for that batch. The blank result must be <EQL.

8.3 If standards are not being distilled in the same batch with the samples, distill a 0.200 mg/L standard as described in §7.4.1.1, using the same 10 mg/L working standard as for the calibration standards. For samples which do not contain sulfide, leave out the bismuth nitrate. This distilled standard must agree with the calibration standard of the same concentration (0.200 mg/L) within 10%, otherwise the problem must be determined and corrected before proceeding. All associated samples may need to be redistilled, or the calibration standards may have to be remade.

8.4 Distill and analyze an aqueous LCS with each batch of aqueous samples. Use 2.00 mL of the working LCS standard solution for a maxi scale distillation, or 200 uL for a midi scale distillation (true value = 0.040 mg/L). Acceptable LCS recoveries are between 85% - 115%. If the LCS fails, all associated samples must be redistilled and reanalyzed.

8.5 Distill and analyze a soil LCS (commercially purchased) with every batch of non-aqueous samples. Use the vendor's acceptance limits.

8.6 Distill and analyze a matrix spike/matrix spike duplicate pair on at least 5% of samples of each matrix. Spike the distillation flasks with working LCS standard solution in the same amount as the aqueous LCS (see §8.4). Acceptable MS/MSD recoveries for water and soils are between 80% - 120% with a RPD less than 20%. If these limits are not met, qualify the results in the report.

8.7 Reread the 0.20 mg/L standard and the calibration blank immediately following the calibration, after every ten samples, and at the end of the batch. The blank must be <EQL. The standard must be within ±10% of the expected value. If either of these conditions is not met, reanalyze all samples run since the previous compliant calibration check.

9.0 INTERFERENCES

9.1 Sulfides adversely affect the colorimetric procedure. Bismuth nitrate is used to remove the interferences.

9.2 Chlorine is an oxidizing agent that will decompose most cyanides. Chlorine should be eliminated when the sample is collected. Ascorbic acid is added to any sample which tests positive for oxidizing agents at the time of analysis.

9.3 To eliminate possible nitrate and nitrite interferences, samples are treated with sulfamic acid prior to distillation.

10.0 FIGURES

Figure 1

Reflux Distillation Apparatus

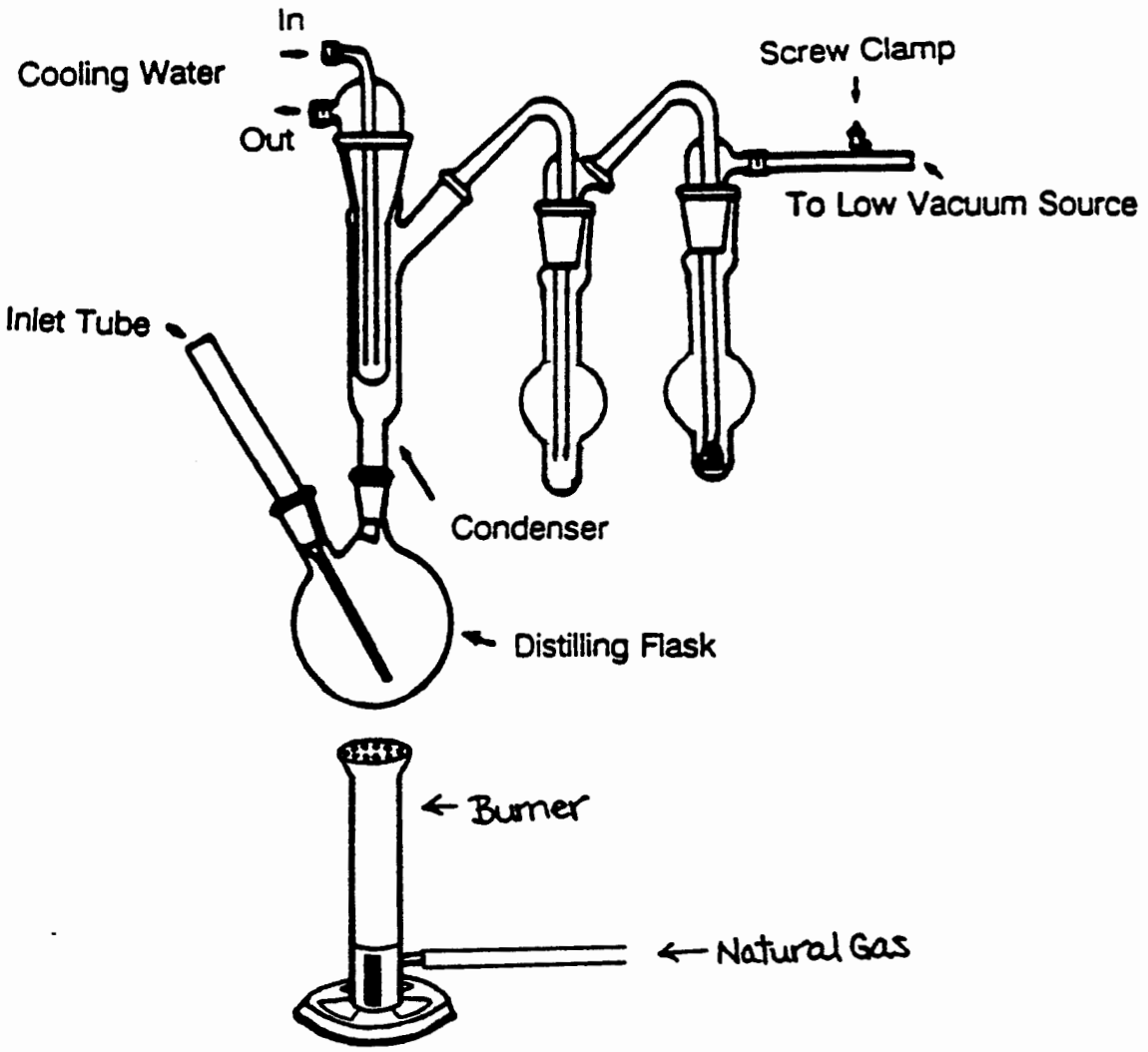
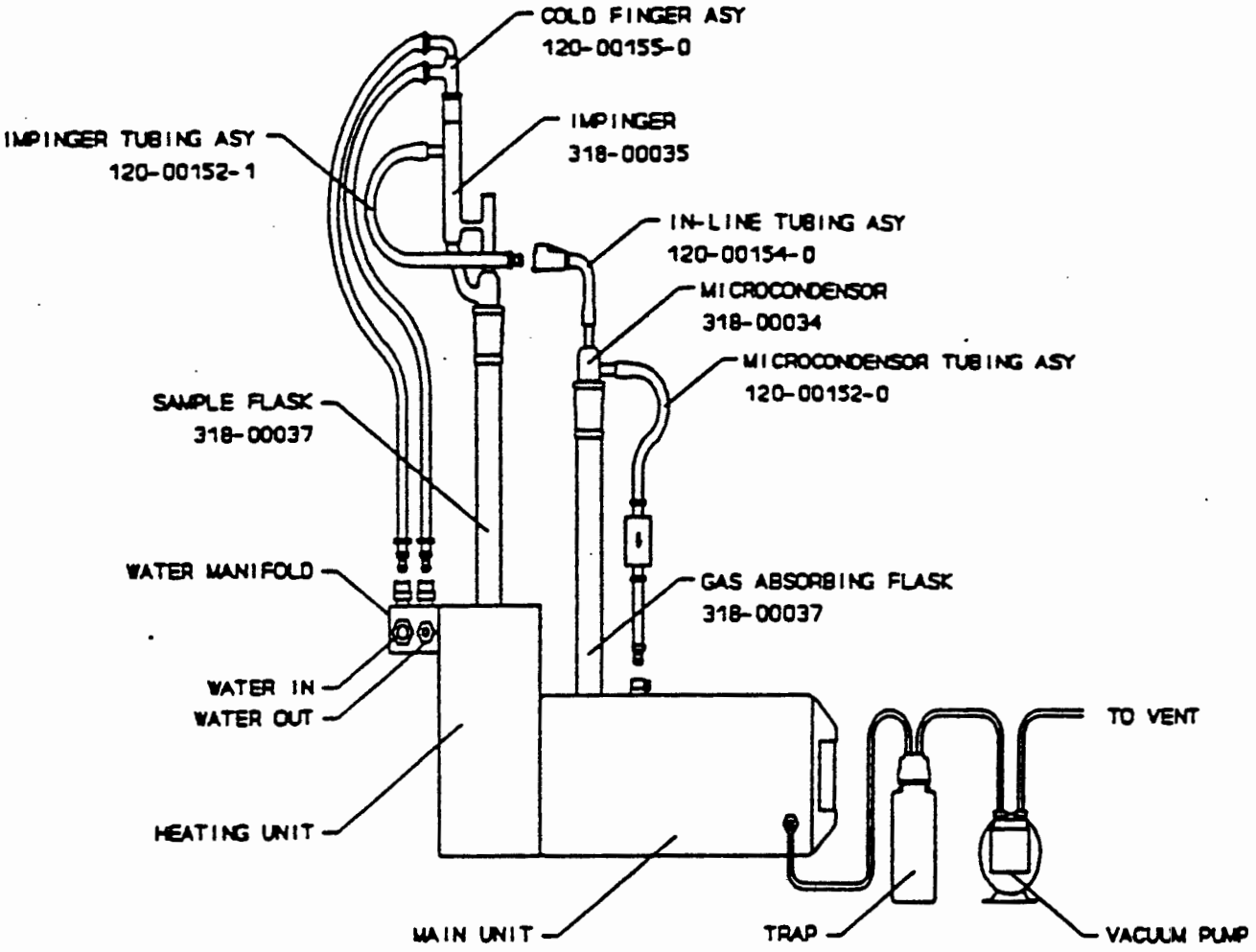


Figure 2

Automated Distillation Apparatus



Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Chloride by Mercuric Nitrate Titration			
Prepared by:	Approved by:	Date: 7/15/96	QA Approval:

1.0 PURPOSE

To describe the procedure for the determination of chloride by titration with mercuric nitrate.

2.0 APPLICATION

This procedure is applicable to waters, wastewaters, groundwaters, and aqueous extracts of solids. Note that there is one indicator for each method. Rather than prepare a different indicator for each of the methods, RASI prepares only the indicator described in Method 325.3. RASI has found that this indicator provides sufficient clarity to obtain the desired accuracy and precision requirements of the method.

3.0 REFERENCES

"Chloride", Methods for Chemical Analysis of Water and Wastes, March 1983, Method 325.3, Revised 1982

"Chloride (Titrimetric, Mercuric Nitrate)", SW-846, Method 9252A, Revision 1, September 1994

"Chloride; Mercuric Nitrate Method", Standard Methods for the Examination of Water and Wastewater, 19th Edition, Method 4500-Cl^- C

4.0 ASSOCIATED SOPs

None

5.0 REAGENTS

5.1 Mercuric nitrate titrant, 0.0141 N; commercially purchased. Standardize weekly against triplicate 5.0 mL aliquots of 1000 mg/L chloride standard (diluted to 50 mL with deionized water) using the procedure in Section 7.2 below. Calculate the actual normality as follows:

N titrant = 0.0141/volume titrant, mL.

Average the three values, and use for calculations.

5.2 Chloride stock standard, 1000 mg/L: commercially purchased, certified NIST traceable. Two standards, one each from separate vendors or different lots from a single vendor, are required. One will be designated for standardizing the mercuric nitrate titrant, and the other for spiking LCSs and MS/MSDs.

5.3 Diphenylcarbazone-bromophenol blue mixed indicator: Dissolve 0.50 g diphenylcarbazone and 0.05 g bromphenol blue in 75 mL of 95% isopropanol and dilute to 100 mL. Store in a brown bottle and discard after 6 months.

5.4 Nitric acid solution (3+997) - Add 3 mL concentrated HNO_3 to 997 mL deionized water and mix.

5.5 Sodium hydroxide (10 g/L) - Dissolve 10 g of NaOH pellets in deionized water and dilute to one liter.

Pretreatment reagents - See Section 9.0, Interferences.

5.6 Alphazurine indicator - Dissolve 0.005 g of alphazurine blue-green dye in isopropanol in a 100 mL Class A volumetric flask and dilute to volume with isopropanol.

5.7 Hydroquinone solution (10 g/L) - Dissolve 1 g of purified hydroquinone in reagent water in a 100 mL Class A volumetric flask and dilute to volume.

5.8 Hydrogen peroxide (H_2O_2) - 30%

6.0 EQUIPMENT

6.1 Buret, 50 mL Class A with 0.01 mL gradations

6.2 Stir plate and stir bars

6.3 Erlenmeyer flasks, 125 mL

7.0 PROCEDURE

7.1 Analysis

7.1.1 Measure 50 mL of sample or an aliquot diluted to 50 mL with deionized water (expected concentration should be less than 100 mg/L) into an Erlenmeyer flask. Stir at low speed on stir plate.

7.1.2 Add 10 drops (0.5 mL) of the indicator and allow to mix. If sample turns blue-violet or red, add HNO_3 solution dropwise just until the color changes to yellow. If the sample turns yellow or orange when indicator is added, add NaOH solution drop-wise until the color changes to blue, then use HNO_3 to restore yellow color as above.

7.1.3 Add 1 mL excess HNO_3 solution.

7.1.4 Titrate with 0.0141 N mercuric nitrate to a permanent blue-violet color, and record the volume used. If the endpoint is not reached after 10 mL titrant, start over with a smaller aliquot. Determine the blank correction by titrating 50 mL of chloride-free deionized water the same way.

7.2 Calculation

$$\text{mg/L chloride} = \frac{(A - B) * N * 35{,}450}{\text{mL of sample}}$$

where:

A = mL titrant for sample

B = mL titrant for blank

N = normality mercuric nitrate titrant

8.0 QUALITY CONTROL

8.1 The mercuric nitrate titrant must be standardized against 1000 mg/L chloride standard prior to the first analyses in any given week.

8.2 A minimum of 1 reagent blank and 1 LCS must be analyzed with each sample batch. For the LCS, spike 50 mL of deionized water with 2 mL of the 1000 mg/L chloride spiking standard (§5.2). LCS recovery must be within 90-110%.

8.3 A minimum of 1 MS/MSD pair must be analyzed for every 20 samples. For the MS/MSD, spike 50 mL of sample (or an aliquot diluted to 50 mL) with 1 mL of the 1000 mg/L chloride spiking standard (§5.2). If recoveries are outside 80-120% or the RPD is >20%, qualify the data in the report.

8.4 The holding time for chloride analysis is 28 days.

9.0 INTERFERENCES

9.1 Bromide and iodide are titrated with mercuric nitrate in the same manner as chloride.

Pretreatment

9.2 If chromate is present at <100 mg/L and iron is not present, add 5 - 10 drops of alphazurine indicator solution and acidify to a pH of 3 (indicating paper). The end point will then be an olive-purple color.

If chromate is present at >100 mg/L and iron is not present, add 2 mL of fresh hydroquinone solution.

9.3 If ferric ion is present, use a volume containing no more than 2.5 mg of ferric ion or ferric ion plus chromate ion. Add 2 mL of fresh hydroquinone solution.

9.4 If sulfite ion is present, add 0.5 mL of H_2O_2 solution to a 50 mL sample and mix for 1 minute.

ROSS	SOP Standard Operating Procedure	SOP No: CW-001-00 Inital Date: 2/1/91 Page 1 of 2 Date Revised:

Title:

Total Alkalinity

Prepared by:	Approved by:	Date:	QC Approval:
Peggy J Bro	CH Caldwell	5/23/91	CH

1.0 PURPOSE

To describe the procedure for the determination of total alkalinity by electrometric titration to pH 4.5.

2.0 APPLICATION

This procedure is applicable to drinking water, natural waters and wastewaters at any concentration range. Appropriate aliquots should be used to avoid a titration volume greater than 30 ml.

3.0 REFERENCES

3.1 EPA-600, Rev. 1983. Method 310.1.

3.2 Standard Methods for the Examination of Water and Wastewater, 17th edition. Method 2320B.

4.0 ASSOCIATED SOPS

None.

5.0 PROCEDURE

5.1 REAGENTS/EQUIPMENT

5.1.1 Standard sulfuric acid, ~0.0357 N: dilute 1.0 ml conc. H_2SO_4 to 1 liter with deionized water. Standardize in triplicate against 40 mg portions of THAM dissolved in 50 ml deionized water, using methyl orange to give a light orange endpoint.

$$\text{Normality } H_2SO_4 = \frac{\text{mg THAM}}{\text{ml } H_2SO_4 * 121.14}$$

5.1.2 THAM, Tris(hydroxymethyl)aminomethane.

5.1.3 Methyl orange indicator: dissolve 50 mg methyl orange powder in deionized water and dilute to 100 ml.

5.1.4 Buret, 50 ml class A.

5.1.5 Stir plate and stir bars.

5.1.6 250 ml Erlenmeyer flasks.

5.1.7 pH meter, calibrated between 4 and 7.

5.2 ANALYSIS

5.2.1 Measure 50 ml of sample or an aliquot diluted to 50 ml with deionized water (expected concentration should be less than 500 mg/l) into an Erlenmeyer flask, avoiding undue agitation. Begin stirring very gently on the stir plate.

5.2.2 Place electrode into sample and leave it there throughout the titration.

5.2.3 Titrate with ~0.0357 N H_2SO_4 to a pH of exactly 4.5. As the endpoint approaches, allow enough time between additions to let pH stabilize. Record the volume used.

5.3 CALCULATIONS

$$\text{Alkalinity, mg/l } CaCO_3 = \frac{A * N * 50{,}000}{\text{ml sample}}$$

where:

A = ml acid used

N = normality of acid

6.0 QUALITY CONTROL

6.1 A minimum of 1 SRS must be analyzed with each sample batch.

6.2 A minimum of 1 MS/MSD pair must be analyzed for every 20 samples.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Digestion Procedure for Metals in Aqueous Samples Using SW-846, Method 3020A (modified)			
Prepared by: BES	Approved by:	Date: 7/13/95	QA Approval:

1.0 PURPOSE

This procedure describes the preparation of aqueous samples for GFAA metals analysis by SW-846, Method 3020A modified. The procedure incorporates the use of hydrogen peroxide to aid in attainment of proper oxidation states for the analysis of arsenic and selenium. Method 3050A includes hydrogen peroxide in the digestion mix and is used for all of the elements that would be prepared for analysis by the modified 3020 procedure. Methods 7060A and 7740 as written for arsenic and selenium incorporate use of nickel nitrate as the primary modifier; both allow other modifiers. Current GFAA analysis utilizing stabilized temperature platform technology incorporates a magnesium/palladium modifier added immediately before analysis.

2.0 APPLICATION

This procedure is applicable to aqueous samples for analysis by graphite furnace atomic absorption (GFAA) for As, Be, Cd, Cr, Co, Cu, Pb, Mo, Se, Tl, V and Zn using SW-846, 7000 Series Methods. Possible interferences are discussed in Section 8.0, and in SOPs MT-007 and MT-013 which describe GFAA analysis.

3.0 REFERENCES

3.1 EPA SW-846, 3rd Edition, Methods 7060A and 7740

3.1 EPA SW-846, 3rd Edition, Methods 3010A and 3020A

4.0 ASSOCIATED SOPs

4.1 IP-012, Wastewater Disposal in the Inorganic Preparation Lab

4.2 IP-013, Acid Bath Preparation

4.3 MT-008, Analysis of Metals Using the Varian SpectrAA 400Z by EPA, SW-846, 7000 Series Methods

4.4 MT-013, Analysis of Metals Using the Varian SpectrAA 20 by EPA, SW-846, 7000 Series Methods

5.0 REAGENTS/EQUIPMENT

5.1 Reagents

5.1.1 Reagent water

5.1.2 Concentrated nitric acid, Mallinckrodt AR Select or equivalent

5.1.3 Hydrogen peroxide, H_2O_2, 30%

5.1.4 Solutions Plus QC 31 Dilution Spike prepared by the Metals Department

5.2 Equipment

5.2.1 Beakers, 150 or 250 mL, acid washed

5.2.2 Volumetric flasks, 50 mL, acid washed

5.2.3 Electric hot plate, with heat control

5.2.4 Red capped polypropylene centrifuge tubes

5.2.5 Qualitative filter paper

5.2.6 Rainin Pipetman, 1000 uL

6.0 PROCEDURE

6.1 Measure a 50 mL aliquot of the well-mixed sample (or an aliquot diluted to 50 mL) into a labeled beaker. If a smaller aliquot is required due to matrix interferences, then record the volume used, dilute it to 50 mL with reagent water and continue with the procedure.

6.2 For each batch of 20 or fewer samples, measure out two 50 mL portions of reagent water into labeled beakers for a LCS and a blank. Likewise, measure out two 50 mL portions of sample to be used for the MS/MSD pair into labeled beakers.

6.3 Spike the LCS and the MS/MSD samples with 500 uL of the GFAA 31 Dilution Spike.

6.4 Add 1 mL of 30% H_2O_2 and 3.0 mL concentration HNO_3 to all beakers and mix.

6.5 Place beaker on the hot plate and evaporate at 95°C to 10-15 mL, making certain that the sample does not boil. If the sample goes to dryness discard and re-prepare it.

6.6 Cover with a watch glass and allow to reflux gently, adding additional acid if necessary until solution is light in color or has no further change in appearance.

6.7 Remove from hot plate and allow to cool.

6.8 Rinse the watch glass into the beaker and transfer digestate to a 50 mL, graduated conical centrifuge tube with at least 3 rinses. Filter if necessary.

6.7 Dilute to volume with reagent water, then cap the tube with a red cap and invert several times to mix.

6.8 If the sample does not contain particulate matter filtration is not necessary. However, if any sample in the set requires filtration all samples must also be filtered.

7.0 QUALITY CONTROL

7.1 Prepare one method blank and one LCS with each batch.

7.2 Each batch must contain no more than 20 samples excluding MS/MSD pairs.

7.3 A minimum of one MS/MSD pair must be prepared for every 20 samples.

8.0 INTERFERENCES

To prevent glassware contamination, all glassware must be acid washed.

9.0 TROUBLESHOOTING

Read all work orders thoroughly in order not to miss any special QC requirements, such as performing a MS/MSD pair on a specific sample.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: IP-013-00
Initial Date: 12/9/94

Date Revised:

Title: Acid Bath Preparation			
Prepared by: SES	Approved by:	Date: 1/10/95	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to prepare and use an acid bath to clean glassware.

2.0 APPLICATION

This procedure applies to all glassware that is used for metals digestions and related sample preparation in the Inorganic Preparation Laboratory.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

SOP No. IP-012, Wastewater Disposal for the Inorganic Preparation Lab

5.0 REAGENTS/EQUIPMENT

5.1 Reagents

5.1.1 High purity reagent water

5.1.2 Concentrated nitric acid, Malinkrodt AR Select

5.2 Equipment

5.2.1 Large or multiple smaller polypropylene bath(s) capable of containing enough beakers typically used in one shift.

5.2.2 Rubber gloves

5.2.3 Fume hood or canopy hood capable of venting fumes out of the laboratory

5.2.4 Face shield

5.2.5 Splash apron

6.0 PROCEDURE

6.1 Bath Preparation and Use

6.1.1 Add equal amounts of nitric acid and reagent water in bath so that the bath is about 1/3 full. Note: Add acid to water.

6.1.2 Put glassware into bath, making sure that each piece is submerged in the acid. Fill entire base of the bath(s) with glassware.

6.1.3 Put lid onto acid bath(s) and soak glassware 8 - 12 hours.

6.1.4 Pour acid out of glassware and back into the acid bath(s).

6.1.5 Remove glassware from bath(s) and rinse thoroughly with deionized water, invert and dry on a rack.

7.0 QUALITY CONTROL

7.1 Once a month take 10 mL of the acid from each bath and digest according to SW-846, Method 3010. Send samples to the Metals Lab for analysis.

7.1.1 If contamination is present in the acid bath sample(s) at a level greater than a normal blank, then the bath must be changed; else, change the bath every three months according to Section 5.3. The Metals Lab will determine if the bath is contaminated and notify the Inorganic Preparation Group to change the bath(s).

7.1.2 Dispose of acid wastes as described in SOP No. IP-012, Wastewater Disposal for the Inorganic Preparation Lab.

8.0 INTERFERENCES

Carryover from glassware improperly washed before soaking in the acid bath can cause contamination. Be certain that all glassware is washed with soap and water before soaking in bath.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: IP-012-00
Initial Date: 8/22/94

Date Revised:

Title: Wastewater Disposal in the Inorganic Preparation Lab			
Prepared by:	Approved by: BES	Date: 9-26-94	QA Approval:

1.0 PURPOSE

To provide a safe, environmentally sound approach for handling, treatment and disposal of laboratory wastewater generated in the Inorganic Preparation (IP) laboratory at Ross Analytical Services, Inc. (RASI).

2.0 APPLICATION

This standard operation procedure (SOP) is to be used to disposed of analyzed raw samples, sample residues, sample extracts, and sample digestates. This SOP has been prepared to provide direction for RASI employees for the disposal of these laboratory wastes in a safe and environmentally sound manner.

3.0 REFERENCES

Prudent Practices for Handling Hazardous Chemicals in Laboratories

4.0 ASSOCIATED SOPs

All IP SOPs

5.0 PROCEDURE

5.1 Reagents and Equipment

5.1.1 Apron

5.1.2 Faceshield

5.1.3 Safety glasses

5.1.4 Rubber gloves

5.1.5 Lab jacket

5.1.6 Operating fume hood

5.1.7 Funnel (Size ???)

5.1.8 Mixing stick

5.1.9 Sodium hydroxide, granular

5.1.10 Concentrated hydrochloric acid

5.1.11 pH Paper

5.1.12 5 Gallon accumulation container with top

5.1.13 Spill control equipment

5.2 Neutralization

5.2.1 After obtaining all safety equipment place the 5 gallon accumulation container in the fume hood before opening. Open the lid carefully; the container should be no more than 3/4 full.

5.2.2 Using the pH paper, check the pH of the solution to be disposed.

5.2.3 If caustic, carefully add concentrated hydrochloric acid until the solution becomes neutral. If the container becomes hot to the touch, dicontinue adding the acid until it has cooled. Periodically, check the pH of the solution to see if it has become neutral. GOTO step 5.1.

5.2.4 If acidic, carefully add the sodium hydroxide solution. If the container becomes hot to the touch, discontinue adding the caustic solution until it has cooled. Periodically check the pH of the solution to see it has become neutral.

5.2.5 Allow the solution to cool completely, then re-check the pH. If solution is not neutral, proceed to either of the steps above.

5.2.6 Tightly seal the container then contact the SHE Department for transporting it to the waste containment building.

NOTE: All laboratory employees are responsible for the disposal of laboratory wastewaters in accordance with the procedure established herein.

6.0 SAFETY

6.1 Always wear personal protection equipment as listed under Section 5.1, e.g., gloves, faceshield, apron, safety glasses, and lab jacket when performing pH adjustments.

6.2 Perform all operations in a well-ventilated fume hood.

6.3 This operation should only be performed when others are present in the same room.

6.4 Keep spill control equipment near this operation.

6.5 Waste containers must be kept closed except when adding or removing waste.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Digestion Procedure for Metals in Aqueous Samples Using SW-846, Methods 3010A, 3020A			
Prepared by: BES/SLS	Approved by:	Date: 1/25/95	QA Approval:

1.0 PURPOSE

This procedure describes the preparation of aqueous samples for metals analysis by SW-846, Methods 3010A, 3020A.

2.0 APPLICATION

This procedure is applicable to water, wastewater and leachate samples for analysis by inductively coupled plasma (ICP), Method 6010A for the elements listed below:

Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Tl, V, Zn

This procedure is also applicable for analysis by graphite furnace atomic absorption (GFAA) by 7000 Series Methods elements listed below:

Be, Cd, Cr, Co, Pb, Mo, Tl, V

Possible interferences are discussed in Section 8.0.

3.0 REFERENCES

EPA SW-846, 3rd Edition, Methods 3010A and 3020A

4.0 ASSOCIATED SOPs

IP-012, Wastewater Disposal for the Inorganic Preparation Laboratory

IP-013, Acid Bath Preparation

5.0 REAGENTS/EQUIPMENT

5.1 Reagents

5.1.1 High purity reagent water (HPRW)

5.1.2 Concentrated nitric acid, Mallinkrodt AR Select or equivalent

5.1.3 Concentrated hydrochloric acid, Mallinkrodt AR Select or equivalent

5.1.4 1:1 HCl - add desired volume of concentrated HCl to an equal volume of HPRW and mix

5.1.5 Solutions Plus QC 31 Spike for ICP (see Section 10.0, Table 1)

5.1.6 Solutions Plus QC 31 Dilution Spike for GFAA prepared by the MT lab. The dilution spike is a 1:20 dilution of the Solutions Plus QC 31 Spike.

5.1.7 Solutions Plus TCLP Spike (see Section 10.0, Table 2)

5.2 Equipment

5.2.1 1000 uL Rainin Pipetman

5.2.2 250 mL Beakers, acid washed

5.2.3 Watch glasses, acid washed

5.2.4 Electric hot plate with temperature control

5.2.5 Qualitative filter paper

6.0 PROCEDURE

6.1 Measure a 50 mL aliquot of the well-mixed sample (or an aliquot diluted to 50 mL with HPRW) into a labeled beaker. Record the aliquot used.

NOTE: Use 10 mL for all TCLP leachates and scrubber waters.

6.2 For each batch of 20 or fewer samples, measure out two 50 mL portions of HPRW into labeled beakers for a LCS and a blank.

6.3 Spike samples in beakers selected for a LCS and MS/MSD pairs with standard solutions. The ICP MS/MSD pair is spiked with 500 uL of the Solutions Plus QC 31 standard. The GFAA MS/MSD pair is spiked with 500 uL of the QC 31 Dilution Spike. Prepare a LCS using 500 uL of the same spike standard used for the MS/MSD pairs for ICP or GFAA, depending on which analysis is required. TCLP MS/MSD pairs are spiked with 500 uL of Solutions Plus TCLP Spike.

6.4 Add 1.5 mL of concentrated nitric acid.

6.5 Place beaker on the hot plate and evaporate to between 5 and 10 mL, making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. If sample does go to dryness, discard and re-prepare.

6.6 Cool beaker, add 1.5 mL of concentrated nitric acid, cover with a watch glass, and return to the hot plate.

6.7 Allow to reflux gently, adding additional acid if necessary, until the solution is light in color or has no further changes in appearance. Remove from hot plate and allow to cool.

6.8 If the sample is being prepared for GFAA analysis, add an additional 5 mL HPRW and return to the hot plate. If it is for ICP analysis, add 5 mL of 1:1 HCl and return to hot plate.

6.9 Heat for an additional 10 to 15 minutes to help dissolve any precipitate.

6.10 Remove from hot plate and cool.

6.11 Rinse watch glass into beaker and transfer sample to a 50 mL volumetric flask with at least 3 rinses. Filter if necessary.

6.12 Dilute sample to final volume then transfer to a polypropylene screw-cap tube for analysis. Use a blue capped tube for ICP samples and a red capped tube for GFAA samples.

7.0 QUALITY CONTROL

7.1 Prepare one preparation blank using 50 mL of HPRW per batch. Each batch must be no greater than 20 samples, excluding MS/MSD pairs.

7.2 Prepare one LCS using 50 mL of HPRW and spiking with 500 uL of ICP or GFAA spike depending upon the analysis requested.

7.3 Digest an MS/MSD pair per batch.

7.4 Holding time for aqueous samples preserved with nitric acid is 6 months.

8.0 INTERFERENCES

Carryover from previous samples and residual soap on glassware can cause contamination. All glassware must be washed with soap and water, tap water rinsed, soaked in a 1:1 nitric acid:water for 8 to 12 hours then rinsed several times with deionized water. Final rinse should be with HPRW.

9.0 TROUBLESHOOTING

Read work orders thoroughly to ensure that no MS/MSD pairs nor special metals requests are missed.

10.0 TABLE(S)

Table 1
Solutions Plus QC 31 Spiking Solution

Element	Concentration	
Aluminum	2500	mg/L
Antimony	100	mg/L
Arsenic	100	mg/L
Barium	100	mg/L
Beryllium	100	mg/L
Bismuth	100	mg/L
Boron	100	mg/L
Cadmium	100	mg/L
Calcium	2500	mg/L
Chromium	100	mg/L
Cobalt	100	mg/L
Copper	100	mg/L
Iron	2000	mg/L
Lead	100	mg/L
Lithium	100	mg/L
Magnesium	2000	mg/L
Manganese	100	mg/L
Molybdenum	100	mg/L
Nickel	100	mg/L
Potassium	2000	mg/L
Selenium	100	mg/L
Silicon	100	mg/L
Silver	100	mg/L
Sodium	2500	mg/L
Strontium	100	mg/L
Thallium	100	mg/L
Tin	100	mg/L
Titanium	100	mg/L
Vanadium	100	mg/L
Zinc	100	mg/L
Zirconium	100	mg/L

Table 2
Solutions Plus TCLP Spike

Element	Concentration	
Arsenic	500	mg/L
Barium	2500	mg/L
Cadmium	100	mg/L
Chromium	500	mg/L
Lead	500	mg/L
Selenium	100	mg/L
Silver	500	mg/L

ROSS	SOP Standard Operating Procedure	SOP No: IP-001-00 Inital Date: 2/1/91 Page 1 of 3 Date Revised:

Title:

Digestion Procedure for Mercury in Aqueous Samples

Prepared by:	Approved by:	Date:	QC Approval:
[illegible]	C H Caldwell	5/23/91	[illegible]

1.0 PURPOSE

This procedure describes the preparation of aqueous samples for mercury analysis.

2.0 APPLICATION

This procedure is applicable to water, wastewater and leachate samples for mercury analysis by the cold vapor method.

3.0 REFERENCES

3.1 EPA SW-846, 3rd edition, Method 7470.

3.2 EPA-600, Rev. 1983, Method 245.1.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 REAGENTS/EQUIPMENT

5.1.1 ASTM Type I water.

5.1.2 Concentrated nitric acid, Mallinckrodt AR Select or equivalent.

5.1.3 Concentrated sulfuric acid, Mallinckrodt AR or equivalent.

5.1.4 Potassium permanganate, 5% w/v: dissolve 50 g potassium permanganate in Type I water and dilute to one liter.

5.1.5 Potassium persulfate, 5% w/v: dissolve 50 g potassium persulfate in Type I water and dilute to one liter.

5.1.6 Stock mercury standard, 1000 mg/l: commercially purchased.

5.1.7 Intermediate mercury standard, 10 mg/l: into a 100/ml volumetric flask approximately half full of Type I water, pipet 500 ul conc. HNO_3 and 1000 ul stock mercury standard (use Eppendorf pipettor). Dilute to volume with Type I water. Shelf life 3 months.

5.1.8 Working mercury standard, 100 ug/l: into a 100/ml volumetric flask approximately half full of Type I water, pipet 500 ul conc. HNO_3 and 1000 ul intermediate mercury standard (use Eppendorf pipettor). Dilute to volume with Type I water. Make fresh daily.

5.1.9 BOD bottles and stoppers, acid washed.

5.1.10 Water bath with lid, 95°C. Preheat water to boiling while setting up samples.

5.2 SAMPLE PREPARATION

5.2.1 STANDARD CURVE: Prepare a complete curve with each batch of samples. Transfer into BOD bottles 0.0, 0.5, 1.0, 2.0, 5.0. and 10.0 mls of the working standard and add sufficient Type I water to total 100 ml. This curve has 0, 0.05, 0.1, 0.2, 0.5, and 1.0 ug/Hg.

5.2.2 SAMPLES: Measure a 100-ml aliquot of the well-mixed sample (or an aliquot diluted to 100 ml) into a labeled BOD bottle.

NOTE: Use a 1/10 dilution for all TCLP leachates and scrubber waters. Spike bottles selected for MS/MSDs with 1.0 ml working standard.

5.2.3 Add 5 ml conc. H_2SO_4 to all bottles and mix.

5.2.4 Add 2.5 ml conc. HNO_3 to all bottles and mix.

5.2.5 Add 15 ml potassium permanganate solution to all bottles and mix. Allow to stand for 15 minutes. If color fades, add more permanganate until color persists for 15 minutes.

Adjust all other bottles in the batch with Type I water so volumes are the same. [If more than an additional 15 ml of permanganate is required, start over with a smaller aliquot]

5.2.6 Add 8 ml potassium persulfate to all bottles and mix.

5.2.7 Stopper bottles very tightly and cover with plastic caps.

5.2.8 Place bottles in boiling water bath and replace lid. The temperature will drop immediately to around 85°C. Keep burners on moderately high until temperature reaches 95°C, then turn down to low. Cook samples for 2 hours.

NOTE: Check water temperature and level periodically. If level is low, add hot water slowly to the bath. Keep temperature at 95°C by adjusting burners.

5.2.9 When digestion is complete, turn off heat, remove bottles from bath, and let cool. Samples are now ready for analysis.

5.3 CALCULATIONS

None.

6.0 QUALITY CONTROL

6.1 All glassware must be washed with soap and water, tap water rinsed, acid-washed in 1:1 HNO_3, then rinsed several times with deionized water. Final rinse should be with Type I water.

6.2 A minimum of 1 blank (in addition to the calibration blank) and 1 SRS must be prepared with each batch of samples.

6.3 A minimum of 1 MS/MSD pair must be prepared for every 20 samples.

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: Digestion Procedure for Mercury in Aqueous Samples

SOP Number: IP-001-01

Date change implemented : 12/19/91

Change initiated by: [signature]

Operational approval: [signature]

QA Approval: [signature]

==========

Section to be changed: See below

Change(s): 3.3 Add "USEPA Statement of work ILM02.0, Method 245.2 CLP-M."

5.1.7 Change "500µl conc. HNO_3" to "1.5mL conc. HNO_3". Change "Shelf life 3 months" to "Make fresh daily."

5.1.8 Change "500µl conc. HNO_3" to "1.5mL conc. HNO_3".

5.2.1 Follow existing with "For CLP analyses, the 0.2µg standard should be omitted."

5.2.7 Delete "and cover with plastic caps."

5.2.5 Replace "Type I water" with "additional permanganate."

6.2 Add "For CLP analyses, the SR3 is labeled 'ICV/CCV' and must be at a mid-r concentration different from the calibration standards."

6.3 Insert after "pair": "(or 1 duplicate/spike pair for CLP analyses)".

6.4 Add "with every batch for CLP analyses, prepare a CRA standard at 0.02µg including all reagents, but do not digest."

ROSS	SOP Standard Operating Procedure	SOP No: CW-017-00 Inital Date: 2/1/91 Page 1 of 4 Date Revised:

Title:

Total and Ortho Phosphorus

Prepared by:	Approved by:	Date:	QC Approval:
		6/3/91	

1.0 PURPOSE

To describe the procedure used for the determination of total and ortho phosphorus. Samples are analyzed directly for ortho phosphorus and must undergo a preliminary digestion for total phosphorus analysis.

2.0 APPLICATION

This procedure is applicable to the analysis of waters, wastewaters, soils and wastes.

3.0 REFERENCES

3.1 EPA 600, Revision 1983, Method 365.2.

4.0 ASSOCIATED SOPs

4.1 SOP No. CW-006-00, Use of the UV/VIS Spectrophotometer.

5.0 PROCEDURE

5.1 Reagents/Equipment

<u>Ortho Phosphorus Reagents</u>

5.1.1 H_2SO_4 solution, 5N: Dissolve 70 mL concentrated H_2SO_4 to 500 mL with deionized water.

5.1.2 Antimony potassium tartrate solution: Weigh 1.3715 g $K(SbO)C_4H_4C_6 \cdot 11\ H_2O$ in 400 mL deionized water and dilute to 500 mL. Store at 4°C in a dark glass stoppered bottle.

5.1.3 Ammonium molybdate solution: Dissolve 20 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 500 mL deionized water. Store at 4°C in a plastic bottle.

5.1.4 Ascorbic acid solution, 0.1 M: Dissolve 1.76 g ascorbic acid in 100 mL deionized water. Make fresh daily.

5.1.5 Combined reagent: Mix the following reagents in the given order for 100 mL of combined reagent:

50 mL 5N H_2SO_4
5 mL antimony potassium tartrate
15 mL ammonium molybdate
30 mL ascorbic acid

Mix after addition of each reagent. Due to limited stability, prepare fresh daily.

5.1.6 Stock phosphorus solution, 50 mg/L P: Dissolve 0.2197 g dried potassium dihydrogen phosphate, KH_2PO_4, in deionized water and dilute to one liter.

5.1.7 Standard phosphorus solution, 0.5 mg/L P: Dilute 10.0 mL of stock phosphorus solution to 1000 mL with deionized water.

5.1.8 NaOH solution, 1N: Dissolve 40 g NaOH in 600 mL deionized water, cool and dilute to one liter.

5.1.9 Phenolphthalein indicator.

<u>Total Phosphorus Reagents</u> - The above reagents with the addition of those below:

5.1.10 H_2SO_4 solution, 11 N: Slowly add 310 mL concentrated H_2SO_4 to 600 mL deionized water. Cool and dilute to one liter.

5.1.11 Ammonium persulfate (peroxydisulfate) crystals.

<u>Equipment</u>

5.1.12 Spectrophotometer for use at 880 nm.

5.2 Total Phosphorus Digestion

5.2.1 Add 1 mL 11N H_2SO_4 to 50 mL sample, or an aliquot diluted to 50 mL, in a 150 mL beaker.

5.2.2 Add 0.4 g ammonium persulfate.

5.2.3 Boil gently on a preheated hot plate until a final volume of approximately 10 mL is reached. Do not allow sample to go to dryness. Remove from hot plate and allow to cool.

5.2.4 Dilute the sample to approximately 30 mL with deionized water and add 1-2 drops of phenolophalein. Add 1N NaOH until sample turns light pink. (If sample is not clear at this point, add 2-3 drops to 11N H_2SO_4 and filter.) Dilute to 50 mL. The sample is now ready for color analysis as described below or for automated analysis in the Inorganic Instrumentation Laboratory. (Digestates may be refrigerated prior to analysis.)

5.3 Ortho Phosphorus/Total Phosphorus Color Analysis

5.3.1 Prepare a series of standards using the 0.5 mg/L standard phosphorus solution (P):

mL standard solution	µP
0	0
1	0.5
3	1.5
5	2.5
10	5
20	10
30	15
40	20
50	25

Dilute all standards to 50 mL with deionized water.

5.3.2 Adjust the pH of the samples to 7, if necessary.

5.3.3 Add 8.0 mL of combined reagent to each standard and sample then mix thoroughly.

5.3.4 After a minimum of 10 minutes but no longer than 30 minutes, calibrate the spectrophometer with standards at 880 nm. Use the 0 standard to initially set the spectrophometer to zero.

5.3.5 Read the concentration of the samples directly from the instrument (in µg P).

5.5 Calculations

$$\text{mg/L P(Ortho or total)} = \frac{\mu\text{g P from curve}}{\text{sample aliquot}}$$

6.0 Quality Control

6.1 Rinse all glassware with hot 1:1 HCl followed by a thorough rinsing with deionized water before use.

6.2 For ortho phosphorus: Analyze a SRS a minimum of once every set. Analyze a matrix spike/matrix spike duplicate (MS/MSD) pair every 20 samples.

6.3 For total phosphorus: Carry 1.5 µg and 15 µg standards, a blank and a SRS through each digestion procedure. Analyze a MS/MSD pair every 20 samples. If samples are to be analyzed on the Lachet, carry all standards through the digestion procedure.

6.4 Refer to control charts for QC acceptance limits.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: pH Meter Calibration and pH Analysis			
Prepared by:	Approved by:	Date: 11/28/94	QC Approval:

1.0 PURPOSE

To describe the procedures for calibrating the Orion model 720A pH meter and for determining sample pH.

2.0 APPLICATION

This procedure is to be used for the calibration of the pH meter and the pH analysis of waters, soils, and wastes.

3.0 REFERENCES

3.1 Orion pH meter instruction manual.

3.2 Methods for Chemical Analysis of Waters and Wastes (EPA-600/4-79-020), Method 150.1.

3.3 Test Methods for Evaluating Solid Waste (SW-846) 3rd edition, Methods 9040A and 9045B.

4.0 ASSOCIATED SOP's

None. Note, however, that this SOP combines the previous SOP-CW-022 (pH Meter Calibration) and SOP-CW-023 (pH Analysis).

5.0 EQUIPMENT

5.1 pH meter, Orion model 720A

5.2 Combination pH electrode

5.3 Beakers, 50 mL

5.4 Thermometer

5.5 Analytical balance, capable of weighing to 0.1 g

5.6 Magnetic stirrer and Teflon coated stir bar

6.0 STANDARDS AND SOLUTIONS

6.1 ASTM type II reagent water

6.2 pH buffer, pH = 4, NIST traceable. Commercially purchased from Baxter Scientific, catalog number H7590 - 4 or equivalent.

6.3 pH buffer, pH = 7, NIST traceable. Commercially purchased from Baxter Scientific, catalog number H7590 - 7 or equivalent.

6.4 pH buffer, pH - 10, NIST traceable. Commercially purchased from Baxter Scientific, catalog number H7590- 10 or equivalent.

7.0 PROCEDURE

7.1 pH Meter Calibration

7.1.1 The pH meter should be in the "STANDBY" mode when not in use. Press any key on the keypad to turn the meter on.

7.1.2 Press the "MODE" key until the pH mode indicator is displayed. Press the "RESOLUTION" key until the display has two decimal places.

7.1.3 Allow the buffers to come to room temperature. Place approximately 30 mL of each buffer into 50 mL beakers.

7.1.4 Measure the temperature of a buffer. Press the "2nd" key, followed by the " SET TEMP" key. Enter the temperature of the buffer at the prompt and press "YES" to enter the value into memory.

7.1.5 Press the "CALIBRATE" key. The time and date of the last calibration will be displayed. The display will read "ENTER NO. BUFFERS", press "3" for the number of buffers to be used, followed by "YES".

7.1.6 Rinse the electrode with reagent water and blot dry. Place the electrode in the pH 4 buffer, making sure that it does not touch the bottom or sides of the beaker. Add a magnetic stir bar, put on the stirrer, and begin slowly mixing the buffer. Make sure to mix the buffers and samples at the same speed. When the electrode signal has stabilized the value for the buffer is displayed. Press "YES" to enter. If performing a manual calibration use the numeric keys to enter the true value of the buffer, then press "YES".

7.1.7 Repeat step 7.1.6 for the pH 7 and pH 10 buffers. The actual electrode slope is displayed after all buffers have been measured. Enter this value into the pH meter calibration log kept near the pH meter. The slope value should be greater than 92%. If it is not, then the meter must be recalibrated.

7.1.8 Rinse the electrode with reagent water and blot dry. Place the beaker containing the pH 4 buffer on the stir plate and stir at the same rate as when calibrating. Place the electrode in the pH 4 buffer. Press the "MEASURE" key. When the display reads "READY" record the pH value in the pH calibration log.

7.1.9 Repeat step 7.1.8 for the pH 7 and pH 10 buffers. The results should be ±0.05 of the true values. If not the meter must be recalibrated.

7.2 pH Analysis of Waters and of Aqueous Wastes (An aqueous sample is one in which the aqueous phase is at least 20% of the total sample volume.)

7.2.1 Calibrate the pH meter as in section 7.1.

7.2.2 Allow the samples to come to room temperature.

7.2.3 Place approximately 30 mL of sample into a 50 mL beaker. Add a stir bar and place the beaker on the stir plate. Stir the sample at the same rate that was used for the calibration buffers.

7.2.4 Rinse the electrode with reagent water and place it in the sample. Make sure the electrode does not touch the bottom or side of the beaker.

7.2.5 Press the "MEASURE" key. When the display reads "READY" record the pH.

7.2.6 Repeat steps 7.2.3 through 7.2.5 on fresh aliquots of sample until two successive values differ by < 0.1 pH units. Rinse the electrode between each pair of readings.

7.2.7 Rinse the electrode with reagent water and place in a beaker of pH 7 buffer for storage. Press the "STANDBY" key to put in standby mode while not in use.

7.3 pH Analysis of Soils

7.3.1 Calibrate the pH meter as in section 7.1.

7.3.2 To 20 g of soil or other sample in a 50 mL beaker, add 20 mL of reagent water. Stir the suspension several times during the next 30 minutes.

7.3.3 Let the soil suspension stand for about 1 hour to allow most of the suspended matter to settle.

7.3.4 Rinse the electrode with reagent water and lower it into the beaker until the tip is just below the interface between the suspension and the supernatant.

7.3.5 Press the "MEASURE" key. When the display reads "READY" record the pH.

7.3.6 Rinse the electrode with reagent water and place in a beaker of pH 7 buffer for storage. Press the "STANDBY" key to put in standby mode while not in use.

7.4 pH Analysis of Solid or Liquid Non-aqueous Waste Samples.

7.4.1 Calibrate the pH meter as in section 7.1.

7.4.2 To 20 g of soil or other sample in a 50 mL beaker, add 20 mL of reagent water. Stir the suspension several times during the next 30 minutes.

7.4.3 Let the soil suspension stand for about 15 minutes to allow most of the suspended matter to settle, or filter off the aqueous phase. If the waste absorbs all of the water, begin again at step 7.4.2 adding 40 mL of water to 20 g of sample. If the sample creates two liquid phases, decant or pipette off the aqueous phase for measurement.

7.4.4 Rinse the electrode with reagent water and lower it into the beaker until the tip is just below the interface between the suspension and the supernatant, if the phases have not been separated..

7.4.5 Press the "MEASURE" key. When the display reads "READY" record the pH.

7.4.6 Rinse the electrode with reagent water and place in a beaker of pH 7 buffer for storage. Press the "STANDBY" key to put in standby mode while not in use.

8.0 QUALITY CONTROL

8.1 Samples should be analyzed as soon as possible after receipt.

8.2 Analyze in duplicate 10% of non-aqueous samples or one per batch, whichever is more frequent. (100 % of aqueous samples are analyzed in duplicate.)

8.3 Reanalyze the pH buffers every 20 samples or at the end of the batch, whichever is more frequent. These results should be within ±0.05 pH units of the true values. If not, recalibrate and reanalyze all of the samples that were analyzed since the last acceptable buffer readings.

8.4 Electrodes must be thoroughly rinsed between samples.

9.0 INTERFERENCES

9.1 Samples with very high or very low pH may give incorrect readings. For samples with a true pH >10, the measured pH may be incorrectly low. Samples with a true pH <1 may give incorrectly high measurements. Any samples for which the corrosivity characteristic is being determined whose pH readings are below 4 or above 10 must be reanalyzed after recalibration with a pH 2 or 12 buffer, respectively.

9.2 Sodium error at pH levels > 10 can be reduced or eliminated by using a low-sodium error electrode.

9.3 Temperature fluctuations may cause measurement errors.

9.4 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or by washing the electrode with a detergent, followed by rinsing with reagent water. If this does not remove the coating, the electrode can be cleaned with an ultrasonic bath or soaked in a solution of 1:10 HCl, then thoroughly rinsed with water.

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original version of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: pH Meter Calibration and pH Analysis

SOP Number: CW-022-02

Date change implemented: 4/18/95

Change initiated by: Craig Caldwell

Operational approval: CAC

QA Approval: [illegible]

Section to be changed: 3.3 and 7.2

Change(s): Addition of newly-published requirement to Method 9040 for high-pH samples

Item: In section 3.3, change refererence to Method 9040A to 9040B, and 9045B to 9045C.

Renumber existing section 7.2.7 to be 7.2.8.

Insert new section:

7.2.7 If the measured pH is above 12.0 and the corrosivity characteristic is being determined, the meter must be recalibrated using pH 10 and 12 buffers held at 25±1° C and the sample reanalyzed at that temperature. If the purpose of the measurement is not known, assume it is for the corrosivity characteristic.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Waste Disposal Procedure for the Wet Chemistry Lab			
Prepared by: D. Buist	Approved by:	Date: 10/5/94	QA Approval:

1.0 PURPOSE

To adjust pH of analytical by-products for safe handling and waste disposal, and to place waste materials in the proper containers.

2.0 APPLICATION

This procedure applies to all aqueous materials that are generated as a result of sample preparation and analysis.

3.0 REFERENCES

Prudent Practices for Handling Hazardous Chemicals in Laboratories

4.0 ASSOCIATED SOPs

All CW SOP's

5.0 PROCEDURE

5.1 Apparatus

5.1.1 Face shield*

5.1.2 Gloves

5.1.3 Apron*

5.1.4 Plastic container, 5 gallon work place accumulation container*

5.1.5 Mixing device, paint mixer or equivalent*

5.1.6 Spill kit

5.1.7 Fume hood

5.1.8 Cart

* Obtain from the SHE group if needed.

5.2 Reagents

5.2.1 Sodium hydroxide*

5.2.2 pH paper*

5.2.3 Hydrochloric acid

* Obtain from the SHE group if needed.

5.3 Procedure for placing waste materials in waste containers. Note that different colored containers denote different handling.

5.3.1 Yellow container. Place the acidic extraction water from the oil and grease extraction in this container. This is nonhazardous waste. The pH of this container should be adjusted as in Section 5.4.

5.3.2 Green container. This is for wastes with pH >12 or wastes that contain cyanide. This includes the NaOH waste from the total cyanide, reactive cyanide, and amenable cyanide distillations, and the colorimetric procedures for these tests and for phenol. THE pH OF THIS CONTAINER SHOULD NEVER BE ADJUSTED!

5.3.3 Red container. This is for hazardous waste with pH <2 that contain hazardous chemicals such as mercury or acetone or high levels of lead. This includes wastes from chloride titrations, CODs, TKNs, the colorimetric procedure for hexavalent chromium, and the lead acetate from cyanide distillations. The pH of this container should be adjusted as in section 5.4.

5.3.4 Blue container. This is for nonhazardous waste with a pH <2. This includes waste from most other tests. The pH of this container should be adjusted as in section 5.4.

NOTE: IF THERE IS ANY QUESTION AS TO WHAT CONTAINER TO PUT A WASTE IN CHECK WITH THE WET CHEMISTRY GROUP LEADER OR WITH A REPRESENTATIVE OF THE SHE DEPARTMENT!!!

5.4 Procedure for adjusting the pH of low pH aqueous materials.

5.4.1 Wear protective apron, face shield, and gloves before handling low pH materials.

5.4.2 Place the 5 gallon accumulation container in a fume hood before opening. Open the lid carefully, the container should be no more than 3/4 full.

5.4.3 Mix the solution and check the pH with pH paper.

5.4.4 If the pH is < 7, add a small quantity of sodium hydroxide while mixing. Continue to mix until the sodium hydroxide is dissolved and check the pH.

5.4.5 Continue this procedure until the solution has a pH in the range of 6 - 7.

5.4.6 If too much sodium hydroxide is added and the pH is now too high, add small quantities of hydrochloric acid while mixing. Check the pH until it is in the proper range.

5.4.7 Always monitor the temperature by feeling the side of the container. If the container becomes warm, allow to cool before making additional pH adjustments.

5.4.8 After the container has been adjusted to a pH between 6 and 7, allow the container to cool and put on the lid securely. Transport the container to the waste storage area on a cart to be transferred to a drum by the SHE department.

5.4.9 The work place accumulation containers marked HAZARDOUS WASTE should be placed in secondary containment for hazardous waste.

5.4.10 The work place accumulation containers marked NONHAZARDOUS WASTE should be placed in secondary containment for nonhazardous waste.

6.0 SAFETY

6.1 Always wear personal protection equipment when performing pH adjustments.

6.2 Perform all operations in a hood.

6.3 This operation should be performed when others are present in the same room.

6.4 Keep spill control equipment near the operation.

6.5 Waste containers must be kept closed except when adding or removing waste.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Bromide				
Prepared by: Shawn Jefferson	Approved by:	Date: 7/17/96	QA Approval:	

1.0 PURPOSE

To describe the procedure for the analysis of bromide by iodometric titration.

2.0 APPLICATION

This procedure is applicable to drinking, surface and saline waters, domestic and industrial waste effluents.

3.0 ASSOCIATED SOPs

SOP No. CW-022, pH Meter Calibration and pH Analysis

SOP No. CW-035, Iodide

4.0 REFERENCES

"Bromide", Methods for Chemical Analysis of Water and Wastes (EPA 600), Method 320.1, Issued 1974

5.0 EQUIPMENT

5.1 Buret, 50 mL, with support stand and clamp

5.2 Erlenmeyer flasks, 250 mL

5.3 Magnetic stir plate and stir bars

5.4 Volumetric flasks, 100, 250, and 1000 mL

5.5 Filter paper, Whatman 541

5.6 pH meter

5.7 Hot plate

5.8 Pipet, 50 mL

5.9 Beaker or flask, 500 mL

6.0 REAGENTS

6.1 Acetic acid solution (1:8): Mix 100 mL of glacial acetic acid with 800 mL of reagent water.

6.2 Bromine water: In a fume hood, add 0.2 mL of bromine to 500 mL reagent water. Stir with a magnetic stirrer and stir bar for several hours or until the bromine is dissolved. Store in an amber bottle.

CAUTION: Bromine is extremely corrosive and irritating. Open only in a hood.

6.3 Calcium carbonate ($CaCO_3$), powdered

6.4 Calcium hypochlorite solution: Add 35 g of calcium hypochlorite, $Ca(OCl)_2$, to approximately 800 mL of reagent water in a 1 L volumetric flask. Stir on a magnetic stirrer for about 30 minutes. Dilute to 1 L and filter. Store in an amber bottle.

6.5 Calcium oxide (CaO), anhydrous, powdered

6.6 Hydrochloric acid solution (1:4). Mix 100 mL of HCl with 400 mL reagent water.

6.7 Potassium iodide (KI) crystals, ACS reagent grade

6.8 Sodium acetate solution: Dissolve 275 g of sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3H_2O$) in reagent water. Dilute to 1 L.

6.9 Sodium chloride (NaCl) crystals, ACS reagent grade

6.10 Sodium formate solution: Dissolve 50 g sodium formate ($NaCHO_2$) in HOT reagent water. Dilute to 100 mL.

6.11 Sodium molybdate solution: Dissolve 1 g sodium molybdate ($Na_2MoO_4 \cdot 2H_2O$) in reagent water and dilute to 100 mL.

6.12 Sulfuric acid solution (1:4): SLOWLY add 200 mL H_2SO_4 to 800 mL reagent water and mix well

6.13 Phenylarsine oxide solution (PAO) 0.0375 N: Commercially purchased. Ricca brand or equivalent.

6.14 PAO working solution 0.0075 N. Pipet 50 mL of 0.0375 N PAO solution into a 250 mL volumetric flask. Bring up to volume with reagent water. *Prepare fresh daily.*

6.15 Starch indicator: Commercially purchased

6.16 Potassium fluoride ($KF \cdot 2H_2O$), reagent grade

7.0 PROCEDURE

7.1 Pretreatment

7.1.1 Add a visible excess of CaO (§6.5) to 400 mL of sample in a 500 mL beaker or flask.

7.1.2 Shake or stir vigorously for approximately 5 minutes.

7.1.3 Filter through a dry Whatman 541 filter paper, discarding the first 75 mL.

7.2 Bromide plus iodide determination

7.2.1 Calibrate pH meter according to CW-022.

7.2.2 Place 100 mL of pretreated sample (§7.1), or an aliquot diluted to 100 mL, in a 250 mL Erlenmeyer flask. Add approximately 5 g NaCl and stir to dissolve. Using a pH meter, adjust to pH 7 or slightly less by dropwise addition of HCl solution (§6.6).

7.2.3 Add 20 mL of calcium hypochlorite solution (§6.4). Add 1 mL HCl solution (§6.6) and approximately 0.2 g calcium carbonate (§6.3).

7.2.4 Heat to boiling on hotplate and boil for 8 minutes. While samples are heating begin Section 7.3.

7.2.5 Remove from hotplate. Allow to cool for 2-3 minutes. Carefully add 4 mL of sodium formate solution (§6.10), slowly enough to prevent foaming. Wash down sides with reagent water.

7.2.6 Return to hotplate and boil for an additional 8 minutes. Occasionally wash sides of flask with reagent water.

7.2.7 Remove from hotplate. Wash sides with reagent water and allow to cool.

7.2.8 If a precipitate forms (iron), add approximately 0.5 g sodium fluoride.

7.2.9 Add 3 drops sodium molybdate solution (§6.11).

7.2.10 Titrate as in §7.4.

7.3 Iodide determination

7.3.1 Place 100 mL of pretreated sample (§7.1) or a portion diluted to 100 mL into a 250 mL Erlenmeyer flask. Using a pH meter, adjust the pH to 7 or slightly less by dropwise addition of H_2SO_4 solution (§6.12).

Perform Steps 7.3.2 - 7.3.5 in a fume hood:

7.3.2 Add 15 mL sodium acetate solution (§6.8.) and 5 mL acetic acid solution (§6.1.). Mix well. Add 40 mL bromine water (§6.2) and mix well. Wait 5 minutes.

7.3.3 Add 2 mL sodium formate solution (§6.10) and mix well. Wait 5 minutes.

7.3.4 Purge the space above the sample with a stream of nitrogen gas to remove bromine fumes.

7.3.5 If a precipitate forms, add 0.5 g potassium fluoride (§6.16).

7.3.6 Titrate as in § 7.4.

7.4 Titration procedure

7.4.1 Dissolve approximately 1 g potassium iodide (§6.7) in the sample from §7.2 or 7.3. Add 10 mL of sulfuric acid solution (§6.12). Allow the sample to sit in the dark for 5 minutes.

7.4.2 Titrate with 0.0075 N PAO working solution (§6.14), until the sample is a pale straw color. Add 1 mL starch indicator and titrate to a colorless solution (disappearance of blue). Disregard returning color.

8.0 CALCULATIONS

8.1 For bromide concentration:

$$\text{mg/L } Br^- = 13{,}320 \left[\left(\frac{A \times B}{C} \right) - \left(\frac{D \times E}{F} \right) \right]$$

Where:

A = Volume (mL) of PAO needed to titrate the sample for bromide plus iodide (minus the volume of PAO needed to titrate the blank)

B = Normality of the PAO used for bromide plus iodide titration

C = Volume (mL) of sample titrated for bromide plus iodide

D = Volume (mL) of PAO needed to titrate the sample for iodide (minus the volume of PAO needed to titrate the blank)

E = Normality of the PAO used for iodide titration

F = Volume (mL) of sample titrated for iodide

8.2 For iodide concentration, if needed:

$$\text{mg/L } I^- = 21{,}150 \times \frac{D \times E}{F}$$

Where:

D = Volume (mL) of PAO needed to titrate the sample for iodide (minus the volume of PAO needed to titrate the blank)

E = Normality of PAO used for iodide titration

F = Volume (mL) of sample titrated for iodine

8.0 QUALITY CONTROL

8.1 A method blank must be run with each batch of samples (20 or less). The amount of titrant used for the blank is subtracted from the amount of titrant used for the samples. Blanks may contain iodide, iodate, bromide, or bromate from reagents.

8.2 An LCS for bromide must be run with each batch of samples. If iodide is being determined at the same time, the LCS may be spiked with both. Regardless of the spike, the LCS must be carried through the entire procedure along with the samples, including both titrations. Recovery must be 90-110%. If the recovery does not fall within this range, check preparation of reagents and reanalyze LCS, blank and all samples.

8.3 A matrix spike/matrix spike duplicate (MS/MSD) OR a duplicate/matrix spike (DUP/MS) pair must be prepared and analyzed on every 20th sample or as required by the client, whichever is more frequent. The recovery limits for the spike are 85-115%, with a duplicate RPD of 20% or less.

8.4 Samples should be stored at 4°C and analyzed within 28 days of collection. If the iodide result will be reported as well as the bromide, the sample must be analyzed within 24 hours of collection. If an iodide determination alone is performed on the day of receipt in order to meet the holding time, and bromide is to be analyzed at a later date, *the iodide determination must be repeated on the same day as the bromide to produce valid results.*

9.0 INTERFERENCES

9.1 Iron, manganese, and organic matter may interfere. The pretreatment with calcium oxide removes or reduces these to insignificant concentrations.

9.2 Color interferes with the observation of indicator and bromine water color changes. This interference can be eliminated by the use of a pH meter instead of a pH indicator, and the use of standardized amounts of oxidant and oxidant quencher.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Iodide			
Prepared by: DMC	Approved by: CAK	Date: 4/19/95	QA Approval: CCL

1.0 PURPOSE

To describe the procedure for the analysis of iodide by iodometric titration.

2.0 APPLICATION

This procedure is applicable to drinking, surface and saline waters, domestic and industrial waste effluents.

3.0 ASSOCIATED SOPs

CW-022, pH Meter Use and Calibration

4.0 REFERENCES

EPA 600, Method 345.1

5.0 EQUIPMENT

5.1 Buret, 50 mL

5.2 Buret stand and clamp

5.3 Erlenmeyer flasks, 250 mL

5.4 Magnetic stir plate

5.5 Stir bars

5.6 Volumetric flasks, 1 L

5.7 Filter paper, Whatman 541

5.8 pH meter

5.9 Hot plate

5.10 Volumetric flasks, 100 mL

5.11 Volumetric flask, 250 mL

5.12 Pipet, 50 mL

5.13 Beaker or flask, 500 mL

6.0 REAGENTS

6.1 Acetic acid solution (1:8). Mix 100 mL of glacial acetic acid with 800 mL of deionized water.

6.2 Bromine water. In a fume hood, add 0.2 mL of bromine to 500 mL deionized water. Stir with a magnetic stirrer and stir bar for several hours or until the bromine is dissolved. Store in an amber bottle.

CAUTION: Bromide is extremely corrosive and irritating. Open only in a hood.

6.3 Calcium oxide (CaO), anhydrous, powdered

6.4 Potassium iodide (KI) crystals, ACS reagent grade

6.5 Sodium chloride (NaCl) crystals, ACS reagent grade

6.6 Sodium formate solution (500 g/L). Dissolve 50 g sodium formate ($NaCHO_2$) in HOT deionized water. Dilute to 100 mL.

6.7 Sodium acetate solution (275 g/L). Dissolve 275 g of sodium acetate trihydrate ($NaC_2H_3O_2 \bullet 3H_2O$) in deionized water. Dilute to 1 L.

6.8 Sulfuric acid solution (1:4). SLOWLY add 200 mL H_2SO_4 to 800 mL deionized water

6.9 Phenylarsine oxide solution (PAO) 0.0375 N, commercially purchased. Ricca brand or equivalent.

6.10 PAO working solution 0.0075 N. Pipet 50 mL of 0.0375 N PAO solution to a 250 mL volumetric flask. Bring up to volume with deionized water. Prepare fresh daily.

6.11 Starch indicator, commercially purchased

6.12 Potassium fluoride ($KF \bullet 2H_2O$), reagent grade

7.0 PROCEDURE

7.1 Pretreatment

7.1.1 Add a visible excess of CaO (Section 6.3) to 400 mL of sample.

7.1.2 Shake or stir vigorously for approximately 5 minutes.

7.1.3 Filter through a dry filter paper, discarding first 75 mL.

7.2 Iodide determination

Perform Section 7.2.2 through 7.2.5 in a hood.

7.2.1 Place 100 mL of pretreated sample (Section 7.1) or a portion diluted to 100 mL into a 250 mL Erlenmeyer flask. Adjust the pH to approximately 7 or slightly less with pH meter by dropwise addition of H_2SO_4 solution (Section 6.8).

7.2.2 Add 15 mL sodium acetate solution (Section 6.7.) and 5 mL acetic acid solution (Section 6.1.). Mix well. Add 40 mL bromine water (Section 6.2), mix well. Wait 5 minutes.

7.2.3 Add 2 mL sodium formate solution (Section 6.6), mix well. Wait 5 minutes.

7.2.4 Purge space above sample with a stream of nitrogen gas to remove bromine fumes.

7.2.5 If a precipitate forms add 0.5 g sodium fluoride.

7.2.6 Titrate as in Section 7.3.

7.3 Titration procedure

7.3.1 Dissolve approximately 1 g potassium iodide in samples from Section 7.2. Add 10 mL of sulfuric acid solution.

7.3.2 Titrate with PAO working solution (Section 6.10) 0.0075 N, until the sample is a pale straw color. Add 1 mL starch indicator and titrate to a colorless solution (disappearance of blue). Disregard returning color.

7.4 Calculations

For iodide determination, if needed:

$$I\ mg/L = 21{,}150\ [(Vpao \bullet N)/V]$$

Where:

Vpao = Volume (mL) of PAO needed to titrate the sample minus the volume of PAO needed to titrate the blank

N = Normality of PAO used to titrate the sample

V = Volume of sample titrated

8.0 QUALITY CONTROL

8.1 A blank for both the bromide plus iodide and the iodide determinations must be run with each batch of samples. The amount of titrant used for the blank is subtracted from the amount of titrant used for the samples. The blank will often have a positive result because of iodine in the reagents.

8.2 An LCS must be run with each batch of samples. LCS recovery must be 90-110%. If the recovery does not fall within this range, check preparation of reagents and reanalyze LCS, blank and all samples.

8.3 Samples must be stored at 4°C and analyzed within 24 hours of collection.

9.0 INTERFERENCES

9.1 Iron, manganese, and organic matter can interfere. The pretreatment with calcium oxide removes or reduces these to insignificant concentrations.

9.2 Color interferes with the observation of indicator and bromine water color changes. This interference can be eliminated by the use of a pH meter instead of a pH indicator, and the use of standardized amounts of bromine water and sodium formate, instead of observing the light color changes.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Dissolved Reactive Silica by Ammonium Molybdate Colorimetry			
Prepared by:	Approved by:	Date: 6/16/95	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to discribe the colorimetric determination of dissolved silica.

2.0 APPLICATION

This procedure is applicable to aqueous samples.

3.0 REFERENCES

U.S. EPA Methods for the Chemical Analysis of Water and Wastes, Method 370.1

4.0 ASSOCIATED SOPs

4.1 CW-006, Use of the UV-VIS Spectrophotometer

5.0 EQUIPMENT

5.1 UV-Vis spectrophotometer, Hitachi

5.2 Nessler tubes, 50 mL & stoppers

5.3 0.45u membrane filters

5.4 Vacuum filter apparatus

5.5 150 mL beakers

5.6 pH meter

6.0 Reagents

6.1 Hydrochloric acid, HCl, 1:1. Add 50 mL of concentrated HCl to 50 mL of reagent water. Store in plastic.

6.2 Ammonuim molybdate reagent: Place 10g $(NH_4)_6Mo_7O_{24} \bullet 4H_2O$ in 75 mL deionized water in a 150mL beaker. Dissolve by stirring and gentle warming. Transfer to a 100 mL volumetric flask and dilute to volume. Adjust to pH 7-8 with silica free ammonium hydroxide or sodium hydroxide solution. Store in plastic.

6.3 Oxalic acid solution: Dissolve 10g $H_2C_2O_4 \bullet 2H_2O$ in deionized water in a 100 mL volumetric flask. Dilute to the mark. Store in plastic. This solution may also be commercially purchased from Ricca or another vendor.

6.4 Stock silica solution; commercially purchased, Ricca or equivalent. 1000 mg/L.

6.5 Stock silica solution for calibration verification commercially purchased from a different source than 6.8. 1000 mg/L.

7.0 Calibraton

7.1 Prepare a calibration curve using six standards and a blank.

7.2 Calibration standards: Pipet 50, 100, 250, 500, 1000, and 1500 µl of 1000 mg/L silica standard (6.8) into separate 50 mL Nessler tubes to prepare standards with concentrations of 1, 2, 5, 10, 20, and 30 mg/L. Dilute each standard to 50 mL with deionized water.

7.3 Blank: Add 50 mL of deionized water to Nessler tube.

7.4 ICV (10 mg/L): Prepare ICV by pipeting 500 µL of secondary silica standard (6.9) into a Nessler tube and diluting to 50 mL.

7.5 Turn on UV-Vis spectrophotometer and allow to warm up for 15 to 20 minutes. Load program for Silica as in SOP CW-006.

7.6 Add rapidly 1.0 mL of 1:1 HCl (6.3) and 2.0 mL of ammonium molybdlate reagent (6.4) to each Nessler tube.

7.7 Stopper the tubes and mix by inverting at least 6 times.

7.8 Let stand 5 to 10 minutes.

7.9 Add 1.5 mL oxalic acid solution (6.7) and mix thoroughly.

7.10 Using calibration blank zero the instrument read each standard as instructed in CW-006, after 2 minutes but before 15 minutes from addition of oxalic acid..

7.11 Read the ICV. It must be within 90-110% of the true value.

8.0 Analysis

8.1 Vacuum filter sample through a 0.45µ membrane.

8.2 Place 50 mL of sample in a Nessler tube.

8.3 Add rapidly 1.0 mL of 1:1 HCl (6.3) and 2.0 mL ammonium molybdate reagent (6.4).

8.4 Stopper tubes and mix by inverting at least 6 times.

8.5 Let stand 5 to 10 minutes.

8.6 Add 1.5 mL oxalic acid solution (6.7) and mix thoroughly.

8.7 Read color spectrophotometrically after 2 minutes but before 15 minutes from the addition of the oxalic acid.

8.8 If samples read higher than the highest standard, reanalyze at a dilution calculated to be within the working range of the calibration curve.

8.9 Analyze a CCV and CCB after every 10 samples and at the end of the run. Prepare the CCV & CCB like the ICV and ICB in 7.4 and develop color as in 8.3 throught 8.6.

9.0 Calculations

Samples results are read directly in concentration (mg/L). If a dilution was made, multiply the concentration from the spectrometer by the dilution factor.

10.0 Quality Control

10.1 Analyses must be performed within 28 days of sample collection.

10.2 The correlation coefficient of the calibration curve must be ≥0.995. If not, check preparation of standards and recalibrate.

10.3 The ICV recovery must be 90-110%. If not recalibrate and rerun.

10.5 Analyze the CCV after every tenth sample and at the end of the run; recovery must be 90-110%. If not, reanlyze all samples since the last passing CCV. Recalibrate if necessary.

10.6 Analyze the reagent blank after every tenth sample and at the end of the run; its reading must be <EQL. If not, reanalyze all samples since the last passing CCB. Recalibrate if necessary.

10.7 Analyze an MS/MSD pair at least every 20 samples.

11.0 Interferences

11.1 Excessive color and/or turbidity interfere. Correct by running sample blanks prepared without the addition of the ammonium molybdate solution. (Add 2 mL of reagent water instead to match volumes.)

11.2 Tannin interference may be eliminated and phosphate interferences may be decreased with oxalic acid.

11.3 Large amounts of iron and sulfide interfere.

11.4 Contact with glass should be minimized by storing samples and reagents in plastic containers.

11.5 Silica free reagents should be used as much as possible.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Nitrate Nitrogen by Brucine Colorimetry						
Prepared by: R. Hon	Approved by: C Haldwell	Date: 10/4/95	QA Approval: CH			

1.0 PURPOSE

To describe the preparation and colorimetric determination of nitrate nitrogen.

2.0 APPLICATION

This method is applicable to the analysis of aqueous samples and aqueous extracts of non-aqueous samples. Modifications can be made to correct for turbidity, color, salinity, or dissolved organic compounds in the sample.

3.0 REFERENCES

3.1 U.S. EPA, Methods for Chemical Analysis of Waters and Wastes, Method 352.1.

3.2 U.S. EPA, Test Methods for Evaluating Solid Waste, Method 9200.

4.0 ASSOCIATED SOP's

4.1 CW-006, Use of the UV/VIS Spectrophotometer.

5.0 EQUIPMENT

5.1 Hitachi U2000 UV/VIS Spectrophotometer with sample sipper.

5.2 Sufficient number of 40 to 50 mL glass sample tubes for reagent blanks, standards and samples.

5.3 Neoprene- coated wire racks to hold sample tubes.

5.4 Water bath suitable for use at 100° C. The bath should be of sufficient capacity to accept the required number of tubes without a significant drop in temperature when the tubes are immersed.

5.5 Water bath suitable for use at 10 - 15° C.

5.6 pH Indicator paper.

5.7 50 mL beakers.

5.8 KI-Starch test papers.

5.9 Filter apparatus and 0.45μ filters.

6.0 REAGENTS AND STANDARDS

6.1 Sodium Chloride solution (30%): Dissolve 300 g NaCl in reagent water and dilute to 1 L .

6.2 Sulfuric acid solution: Carefully add 500 concentrated H_2SO_4 to 125 mL reagent water. Store tightly capped to prevent absorption of atmospheric moisture.

6.3 Brucine-sulfanilic acid reagent: Dissolve 1 g brucine sulfate $[(C_{23}H_{26}N_2O_4)_2 \cdot H_2SO_4 \cdot 7H_2O]$ and 0.1 g sulfanilic acid $[NH_2C_6H_4SO_3H \cdot H_2O)]$ in 70 mL hot reagent water. Add 3 mL concentrated HCl cool, mix, and dilute to 100 mL with reagent water. Store in a dark bottle at 5° C. This solution is stable for 3 months. The pink that slowly develops does not affect its usefulness. Mark bottle with warning, **"CAUTION: Brucine sulfate is toxic; do not ingest."**

6.4 Potassium nitrate stock solution (100 mg/L): Oven dry potassium nitrate at 105 C for 24 hours. Dissolve 0.7218 g in reagent water and dilute to 1 L. Preserve with 2 mL chloroform per liter. This solution is stable for at least 6 months. This solution may be commercially purchased.

6.5 Potassium nitrate working standard (10 mg/L): Dilute 10.0 mL of the stock solution (6.4) to 100 mL. Prepare fresh weekly.

6.6 LCS (100 mg/L): Dissolve 0.6071 g anhydrous sodium nitrate in reagent and dilute to 1 L. This solution may also be commercially purchased.

6.7 Acetic acid (1+3): Add 100 mL glacial acetic acid (CH_3COOH) to 300 mL reagent water.

6.8 Sodium hydroxide (1 N): Dissolve 40 g of NaOH in reagent water. Cool and dilute to 1L. Store in plastic.

6.9 Sodium arsenite, 10% solution. Dissolve 10 g of $NaAsO_2$ in reagent water and dilute to 100 mL.

6.10 Potassium permanganate, 10% solution. Dissolve 10 g of KMnO4 in reagent water and dilute to 100 mL.

6.10 Hydrogen peroxide, 30% solution.

7.0 PROCEDURE

7.1 Test for the presence of oxidizing agents with KI-Starch test paper. Pour a small aliquot of sample into a 50 mL beaker. Acidify with acetic acid. Dip the test paper the sample. If the test paper turns blue, oxidizing agents are present. Add small amounts of sodium arsenite solution, mix the sample and test with the KI-starch test paper again. Repeat this until the test paper remains white after dipping it in the sample.

7.2 Test for the presence of reducing agents with potassium permanganate. Add a few drops of 10% solution to a few mL of sample. The disappearance of the purple color (other than by dilution) indicates reducing agents. Add small amounts of hydrogen peroxide until an aliquot of the sample tests negative.

7.3 Adjust the pH of the samples to approximately 7 with acetic acid or sodium hydroxide solutions. If necessary, filter to remove turbidity.

7.4 Set up the required number of tubes in the rack to handle the reagent blank, standards and samples. Space tubes evenly throughout the rack to allow for even flow of bath water between the tubes.

7.5 This test uses a wavelength in the yellow region of the spectrum. Dissolved organic matter will often turn yellow upon addition of the sulfuric acid in step 7.9 If it is necessary to correct for native color or color produced from dissolved organic matter, analyze a second aliquot of the affected samples with all of the reagents except the brucine- sulfanilic acid. These are "sample blanks".

7.6 Prepare standards by pipetting 0.1, 0.25, 0.5, 1.0, 2.0, and 2.5 mL of the 10 mg/L working nitrate standard into sample tubes. Dilute to a final volume of 10 mL. This yields a curve of 0.1 to 2.5 mg/L. Prepare a reagent blank by pipetting 10 mL of reagent water into a sample tube.

7.7 Pipet 10 mL of samples or an aliquot diluted to 10 mL into sample tubes.

7.8 Add 2 mL of the 30% NaCl solution to the reagent blank, standards and samples. Mix contents of tubes by swirling or on vortex mixer. Place rack in a cold water bath (0 - 10° C).

7.9 Pipet 10 mL of the sulfuric acid solution into each tube. Mix by inverting at least three times; more mixing may be necessary. Allow the tubes to come to thermal equilibrium in the cold bath. Be sure that temperatures have equilibrated in all tubes before continuing.

7.10 Add 0.5 mL of the brucine-sulfanilic acid reagent to all tubes except the "sample blank" tubes. Add 0.5 mL of reagent water to them. Carefully mix by inverting at least times.

7.11 Carefully place the rack of tubes in the 100° C water bath for exactly 25 minutes.

CAUTION: Immersion of the rack into the bath should not decrease the temperature of the bath by more than 1-2 C. In order to keep this temperature decrease to a minimum, flow between the tubes should not be restricted by crowding too many samples in the rack. If color development in the standards reveals discrepancies in the procedure, the analysis should be repeated after review of the temperature control steps.

7.12 After the 25 minutes, remove the rack of tubes from the hot water bath, immerse in the cold water bath, and allow the samples to reach thermal equilibrium (20 - 25° C)

7.13 Calibrate the UV/VIS spectrophotometer at 410 nm, as in SOP CW- 006. (Note: The curve for this test is second order.) After calibration, analyze the LCS and samples on spectrophotometer.

8.0 CALCULATION

8.1 The readings from the spectrophotometer are in mg/L. If 10 mL of sample was analyzed no further calculation is necessary. If less than 10 mL was analyzed, multiply result by the appropriate dilution factor.

8.2 If a sample blank was analyzed to correct a sample for color or turbidity, subtract the concentration found in the sample blank from that of the sample.

9.0 QUALITY CONTROL

9.1 Prepare a set of standards with each batch of samples. Reread the midpoint standard and the calibration blank after every ten samples and after the last sample of the batch. The blank must be <EQL. The standard must read within ±10% of its true value. If these criteria are not met, reanalyze all samples run since the last compliant blank and calibration check.

9.2 Samples may be diluted after color development if they are not far above the top of the calibration curve. Otherwise, reanalyze with a smaller aliquot.

9.3 Analyze an LCS with every batch of samples. Add 100 µL of the 100 mg/L LCS solution to 10 mL of deionized water. The result obtained must be within 0.90-1.10 mg/L.

9.4 Analyze one MS/MSD pair for every 10 samples. Spike two additional aliquots of the sample with 100 µL of the LCS solution. If necessary, use a 1:2 dilution of the sample so the total volume in the tube can remain 10 mL. If the recoveries are outside the range of 75-125% or have >20% RPD, qualify the results in the report.

10.0 INTERFERENCES

10.1 Interferences from organic matter or other substances which cause a yellow color are addressed in Section 7.5.

10.2 Interferences from strong oxidizing and reducing agents are addressed in Sections 7.1 and 7.2.

10.3 Ferrous and ferric ion and quadrivalent manganese give slight positive interferences, but in concentrations less than 1 mg/L these are negligible.

10.4 Uneven heating of the samples and standards during the reaction time will result in erratic values.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Specific Conductance (Conductivity)			
Prepared by: Sharon Jefferson	Approved by:	Date: 10/1/96	QA Approval:

1.0 PURPOSE

To describe the procedure for determining the specific conductance of a sample.

2.0 SCOPE AND APPLICATION

This method is applicable only to aqueous samples.

3.0 REFERENCES

3.1 "Conductance", Methods for Chemical Analysis of Water and Wastes, Method 120.1, Issued 1971, Revised 1982

3.2 "Specific Conductance", Test Methods for Evaluating Solid Waste (SW-846), Method 9050A, Revision 1, November 1990

3.3 "Conductivity", Standard Methods for the Examination of Water and Wastewater, 19th Edition, Method 2510 B, Approved 1991

3.4 Model YSI 35 Conductivity Meter Operator's Manual

4.0 ASSOCIATED SOPs

None

5.0 EQUIPMENT AND REAGENTS

5.1 Equipment

5.1.1 YSI 35 Conductivity Meter

5.1.2 Dipping type conductivity cell, cell constant 10.0 cm^{-1} (YSI #3403 or equivalent)

5.1.3 Thermometer which can be read to 0.1 °C in the range of 23 - 27 °C

5.1.4 Nessler tubes, 50 mL

5.2 Reagents

5.2.1 Standard potassium chloride solution (1000 µmhos/cm) - commercially purchased

5.2.2 Laboratory Control Sample (LCS) - the same solution as in Section 5.2.1; however, purchased as a separate lot or from a different vendor

6.0 PROCEDURE

6.1 Cell Constant Determination

6.1.1 Rinse the conductivity cell with at least three portions of standard KCl solution.

6.1.2 Measure the temperature of the standard to the nearest 0.1 °C.

6.1.3 Measure the conductance (conductance, the inverse of resistance, is the measurement performed by the YSI 35 Conductivity Meter) of the standard, using the procedure in §6.2 below.

6.1.4 Compute the cell constant, C:

$$C = \frac{\text{(true conductivity of the std)} \times [1 + 0.0191\,(t - 25)]}{\text{measured conductance of the std, µmhos/cm}}$$

where:

t = temperature in °C

6.2 Sample Analysis

6.2.1 Allow samples to come to room temperature (23 - 27 °C) before analyzing. Pour 50 mL of sample into a Nessler tube. Measure the temperature of the sample to the nearest 0.1 °C.

6.2.2 Rinse the cell with the sample, then insert the cell into the Nessler tube. Make sure that no bubbles are trapped inside the cell. Change the range, if necessary, to get a valid reading on the meter, then record the meter range and displayed reading. If necessary, convert results to micromhos (µmhos).

7.0 CALCULATIONS

$$\text{Specific Conductance (conductivity), µmhos/cm at 25° C} = \frac{K \times C}{1 + 0.0191\,(t - 25)}$$

where:

K = Measured conductance of sample in µmhos/cm
C = Cell constant determined in §6.1
t = temperature in °C

8.0 QUALITY CONTROL

8.1 The holding time for conductivity measurement is 28 days from sample collection, however analysis as soon as possible is recommended, to avoid changes in dissolved constituents.

8.2 Analyze an LCS with every batch of 20 or fewer samples. The acceptable range is 90 - 110%.

8.3 Perform duplicate analyses at a frequency of 10%. The paired results should have $\leq$20% RPD.

8.4 Conductivity measurement should be performed as close to 25 °C as possible. Although the sample results are corrected for temperature deviations in the calculation, the correction factor is that of standard KCl solution. The actual correction factor for a given water sample is approximately the same as the KCl solution. So, the greater the temperature deviates from 25 °C, the greater the uncertainty in the corrected result.

9.0 INTERFERENCES

9.1 Platinum electrodes can degrade and cause erratic results. When this happens, as evidenced by erratic results or flaking off of the platinum black, the electrode should be replatinized.

9.2 The specific conductance cell may become coated with oil and other materials. It is essential that the cell be thoroughly rinsed and, if necessary, cleaned between samples.

CONTROL COPY NO. ____

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: CW-059-00
Initial Date: 10/4/95

Date Revised:

Title: Sulfate by Turbidimetry				
Prepared by: K. Hon	Approved by: [illegible]	Date: 10/4/95	QA Approval: [illegible]	

1.0 PURPOSE

The purpose of this procedure is to describe the turbidimetric determination of sulfate.

2.0 APPLICATION

This procedure is applicable to the determination of sulfate in aqueous samples or aqueous extracts of non-aqueous samples. Note: This SOP describes the Hach Chemical Co. modification to EPA Method 375.4, which is an EPA-approved alternate method.

3.0 REFERENCES

3.1 U.S. EPA, Methods for the Chemical Analysis of Water and Wastes, Method 375.4

3.2 U.S. EPA, Test Methods for Evaluating Solid Waste, Method 9038

3.3 HACH Water Analysis Handbook, 1989.

4.0 ASSOCIATED SOPs

4.1 CW-006, Use of the UV/VIS Spectrophotometer

5.0 EQUIPMENT AND REAGENTS

5.1 Equipment

5.1.1 Spectrophotometer for use at 420 nm.

5.1.2 50 mL centrifuge tubes

5.1.3 Vacuum filtration apparatus

5.1.4 0.45 µm cellulose ester membrane filters

5.2 Reagents

5.2.1 Sulfate standard, 2500 mg/L. Commercially purchased.

5.2.2 Secondary sulfate standard, 1000 mg/L. Commercially purchased.

5.2.3 Hach Sulfate Reagent Powder Pillows

6.0 ANALYSIS

6.1 Prepare a calibration curve by using an Eppendorf pipettor to deliver the volumes of the 1000 mg/L standard listed below into 50 mL centrifuge tubes. Add deionized water to a final volume of 25 mL.

Standards

µL working std	mg/L Sulfate
0	0
250	10
500	20
750	30
1000	40
1500	60
2000	80

6.2 Add the contents of one Sulfate Reagent Powder Pillow to each centrifuge tube, cap and shake to dissolve.

6.3 Allow 5 minutes for turbidity development and then calibrate the spectrophotometer at 420 nm within the next 10 minutes.

6.4 For sample analysis, add the contents of one Sulfate Reagent Powder Pillow to 25 mL of sample or an aliquot diluted to 25 mL. Allow 5 minutes for turbidity development and read on the spectrophotometer.

6.5 If sample does not fall within the above range, repeat the analysis using a smaller aliquot diluted to 25 mL. Do not dilute the sample after adding the powder pillow..

6.6 For colored or turbid samples, filter through a 0.45 µm filter before adding the Powder Pillow reagent. Record that this was done. If the sample is still colored or turbid after filtration, analyze a sample blank using the above procedure without the addition of the Powder Pillow reagent.

7.0 CALCULATIONS

7.1 For waters:

mg/L Sulfate = (Cs - Csb) x dilution factor

Where

Cs is the measured sample concentration in mg/L, and

Csb is the measured sample blank concentration in mg/L, if used

7.2 For solids:

$$\text{mg/Kg Sulfate} = \frac{(Cs - Csb) \times \text{dilution factor} \times \text{Final Volume}}{\text{Sample Weight}}$$

Where

Final Volume = volume of water used to extract the sample in mL, and

Sample Weight = weight of sample extracted, in g

8.0 QUALITY CONTROL

8.1 Analyses must be performed within 28 days of sample collection.

8.2 Analyze one Laboratory Control Sample (LCS) with every 20 samples in each batch. Dilute 250 µL of the 2500 mg/L stock to 25 mL. Acceptable recoveries are between 90% - 110%. If the LCS fails, recalibrate and reanalyze the associated samples.

8.3 Analyze a matrix spike/matrix duplicate pair for every 20 samples of each matrix. Add 500 µL of the 1000 mg/L stock to 25 mL of sample. This yields a true value of 19.6 mg/L. Acceptable matrix spike/matrix duplicate recoveries fall between 80% - 120% with an RPD less than 20%. If results exceed either of these criteria, qualify the data in the report.

8.5 Reread the calibration blank and the 30 mg/L standard after every tenth sample and at the end of the run. the blank is >EQL or if the standard result is not within 10% of the expected value (27-33 mg/L), reanalyze all samples run since the last compliant continuing calibration.

9.0 INTERFERENCES

9.1 Silica and calcium may interfere at levels above 500 mg/L and 20,000 mg/L, respectively.

9.2 Chloride and magnesium do not interfere at levels up to at least 40,000 and 10,000 mg/L, respectively.

ROSS	SOP Standard Operating Procedure	SOP No: IT-004-00 Inital Date: 2/1/91 Page 1 of 12 Date Revised:

Title:

Total Organic Halides in Waters

Prepared by:	Approved by:	Date:	QC Approval:
Belinda Minter	C H Caldwell	5/23/91	[illegible]

1.0 PURPOSE

This procedure describes the determination of total organic halides (TOX) in drinking water, ground water, and other aqueous sample.

2.0 APPLICATION

This procedure applies to the analysis of aqueous samples for all organic halides containing chlorine, bromine, and iodine using a Dohrmann DX-20 Total Organic Halide Analyzer and AD-3 Adsorption Module System.

Fluorine-containing species are not determined by this procedure. This procedure also does not measure TOX of compounds adsorbed to undissolved solids.

3.0 REFERENCES

EPA SW-846, 3rd edition, Method 9020.
Rosemount/Dohrmann Instruction Manuals for the AD-3 Adsorption Module System and the DX-20 Total Organic Halide Analyzer System.

4.0 ASSOCIATED SOP's

None

5.0 PROCEDURE

5.1 Apparatus and Materials:

5.1.1 Adsorption Columns: Pyrex, 5 cm long x 6 mm x 2mm-I.D.

5.1.2 Granular Activated Carbon (GAC).

5.1.3 Cerefelt and Cerefelt plug cutter.

5.1.4 Column Holders.

** OR CPI Pre-packed Columns instead of 5.1.1 thru 5.1.4.

5.1.5 Volumetric flasks, 100 mL and 50 mL.

5.1.6 Microcoulometric-titration system.

Quartz boats for solids and liquids.
Pyrolysis furnace and tubes.
Titration cell
Set of four assorted electrodes (color-coded).
Assorted septums and o-rings.

5.2 Gases:

5.2.1 Carbon Dioxide (CO_2): 99.9% purity.

5.2.2 Oxygen (O_2): 99.9% purity.

5.2.3 Nitrogen (N_2): prepurified.

5.3 Reagents:

5.3.1 Reagent Water (Type I H_2O) - to decrease contamination and improve baseline, use the water from WaterPro PS.

5.3.2 Sodium Sulfite Or Thiosulfate 0.1 M ($Na_2SO_3/Na_2S_2O_3$) - required for water sample pretreatment to reduce free chlorine to chloride; 12.6 gm Na_2SO_3/L of O_2 free reagent water (bubble with Helium).

5.3.3 Concentrated Nitric Acid (HNO_3) - acidification of samples to pH 2; 1 ml of HNO_3/500 ml of sample.

5.3.4 Potassium Nitrate Solution 5000 ppm (KNO_3) - desorbs inorganic halides from GAC in nitrate wash channel; 8.2 gm of KNO_3/L of reagent water.

5.3.5 Trichlorophenol Stock Solution (1 UL = 10.0 ug Cl^-) - 1.86 gm of 2,4,6-Trichlorophenol/100 ml methanol.

5.3.6 Trichlorophenol Adsorption Efficiency Standard (100 ug Cl^-/L) - to check efficiency of DX-20A instrument; inject 10.0 ul of TOX stock solution/L of reagent water.

5.3.7 Acetic Acid In Water (70%) - Dilute 7 volumes of glacial acetic acid with 3 volumes of Type I water to use as electrolyte in titration cell.

5.3.8 Sodium chloride, 1000 mg/L Cl - Dissolve 1.648 g oven-dried NaCl reagent water and dilute to 1 L.

5.4 Sample Preparation:

5.4.1 Samples should arrive in glass bottles with little or no head space, preserved to pH ≤ 2 with HNO_3 and with sodium sulfite added to reduce residual chlorine. If samples arrive at pH ≤ 2, assume acid and sodium sulfite have already been added. If samples are not already preserved, add HNO_3 to pH ≤ 2 and sodium sulfite (1 mL per L).

5.4.2 Samples containing undissolved solids must be filtered through a water leached glass fiber filter before analysis to remove solids.

5.5 Column Preparation:

5.5.1 If using CPI products, skip this step. If using Dohrmann products, use the Cerefelt plug cutter to cut plugs and insert GAC into one end of the Pyrex column. Fill Pyrex column with approximately 40 mg GAC.

<u>Be sure to keep the GAC free from atmospheric and other contamination; this will greatly affect your results</u>.

Then, plug the open end of column with another Cerefelt plug.

5.6 Instrument Set-Up:

5.6.1 The power should be on. The instrument should be left on to cut down on furnace wear and long warm-up times. The "Function Selector Dial" should be set to DET.

5.6.2 Turn on the O_2 and CO_2 gases, and adjust both to 25 psi. Set the N_2 tank at 30 psi.

5.6.3 Attach titration cell to pyrolysis furnace tube with clamp. Do Not Overtighten.

5.6.4 Put quartz sample boat in place in pyrolysis tube and close.

5.6.5 Set "BIAS Selector Dial" to 250 and set "MINUTES Selector Dial" to 10 (should already be set).

5.6.6 Perform a 'cell flush' by draining the microcoulometric titration cell. Rinse the walls and the upper side arm with new electrolyte, make sure the electrodes are clean and in proper position (see Figure 1), then fill the titration cell with 70% acetic acid up to fill line (halfway between the bulb and reference electrode).

5.6.7 Look at the panel reading; if it is negative, flush the cell again and check for bubbles in the sidearms. If it is reading positive, the baseline should jump up and then stabilize to a value less than 1.0 mV after several minutes.

5.6.8 Turn the "MODE Selector Dial" to TOX and wait for a steady base line reading (must be <1.0 mV). Once a steady baseline is achieved, turn the "FUNCTION Selector Dial" to INT and push the START toggle. This will start a 'boat burn' or dry run pyrolysis sequence to clean the quartz boat. At the end of the pyrolysis sequence there will be a microgram reading on the digital panel meter and the ready light will illuminate. The digital panel meters should read less than 0.10 at end of dry run. Two to four dry runs may be necessary to achieve a reading of less than 0.10 ug.

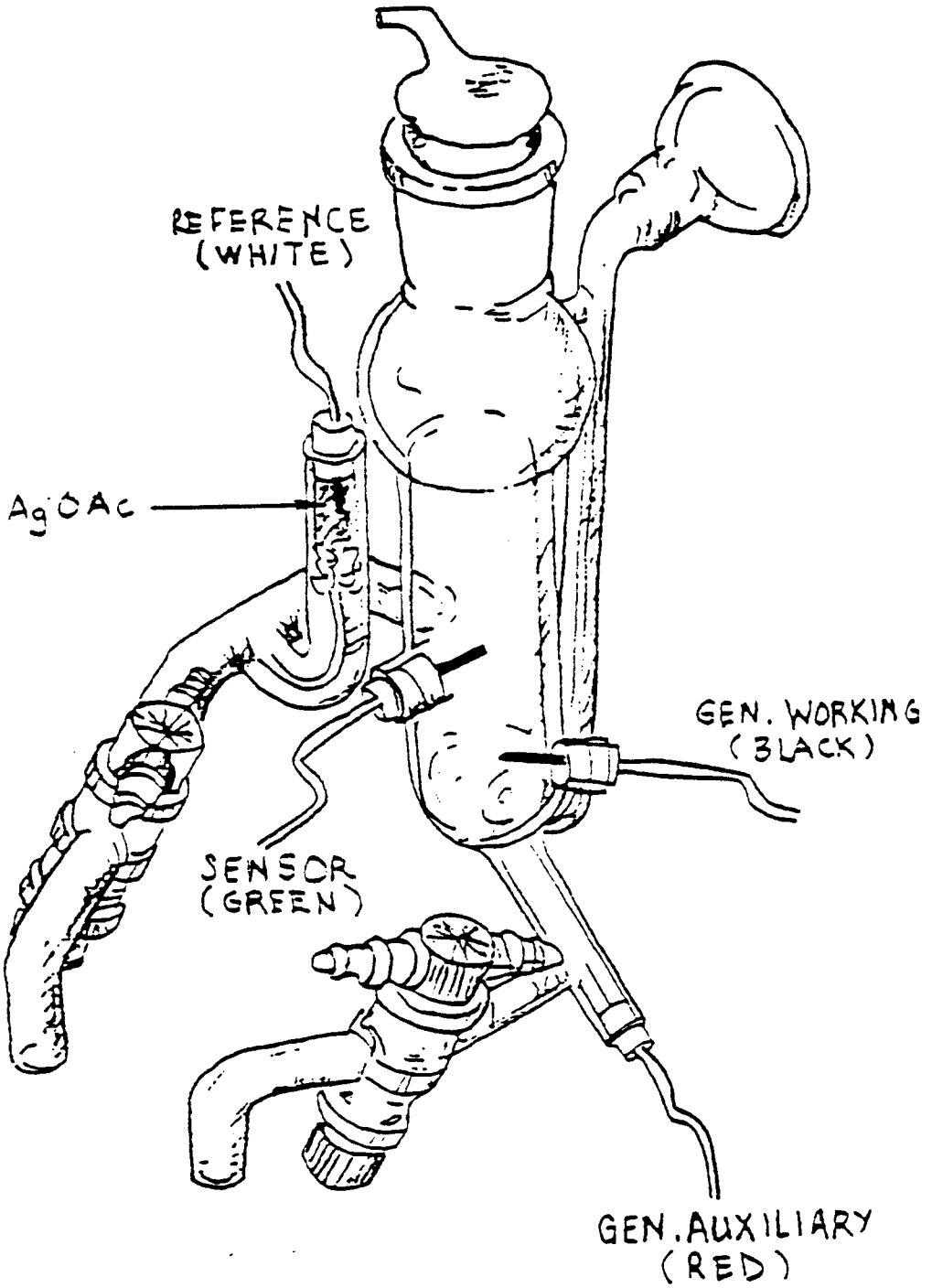
5.7 Daily Start-Up Procedure:

5.7.1 Check the recovery of the MC-3 by putting dry GAC into the boat through the hatchport and press START. Disregard the end result. Turn the "MODE Selector Dial" to INT and inject 2 ul of the 500 ng Cl^-/ul TCP in MeOH check standard onto the GAC <u>ash</u>. The result should be 0.90 ug (90% recovery) or greater. Repeat this analysis at least twice and look for $\pm$2% RSD.

5.7.2 Determine the GAC value for each new batch of carbon columns by pyrolizing approximately 40 mg (1 scoop or 1 column) of carbon. Make at least two determinations, and do not use the GAC if the read-out is > 1.0 ug Cl^-.

Figure 1

T-621
Titration



5.7.3 Determine the 'method blank' by preparing four blanks by adsorbing 100 mls of Type I H_2O and nitrate washing. Each separate nitrate-washed column represents a blank. The values for this method blank should be $\leq$ the GAC value plus 0.2 ug. Repeat until consistent results are achieved. Use the average value for calculations. If you have any blanks left over, use them for future analysis after flushing the titration cell.

5.7.4 Check 'absorption efficiency' by preparing 1 L of the 100 ng Cl^-/mL TCP in MeOH standard and follow all adsorption steps to be used for your unknowns as described in Section 5.8. Use the same volume for standards and unknowns, typically 100 mL. Analyze each column separately. Using the ejector tool, expel the GAC from the column directly onto the sample boat. Close the hatch. The baseline will drop in response to opening the hatch. When it returns to the original value, press START. Recovery should be greater than 90% $\pm$ 2 for two separate adsorption runs. For EPA Method 9020/450.1 compliance, no more than 10% of the total sample TOX should be measured from the bottom column.

5.8 Adsorption Procedure:

5.8.1 Rinse sample reservoirs and column holders with Type I water.

5.8.2 Set the sample channel at 20 psi or 30 psi if using all four. Next set the nitrate channel at 13-14 psi.

5.8.3 Connect two columns in series, each containing 40 mg of 100/200-mesh activated carbon, in their holders to the bottom of the sample reservoir. If using Dohrmann products see Figure 2 (an o-ring must touch each end of the GAC column) and if using CPI products see Figures 3 and 4.

5.8.4 Fill the sample reservoir with sample (5-10% more than you want to collect).

5.8.5 Put 100 mL volumetric flasks in the automatic shutoff holders, adjust the height of the flask to align level sensor with the flask volume line, and force sample through columns with nitrogen.

Figure 2

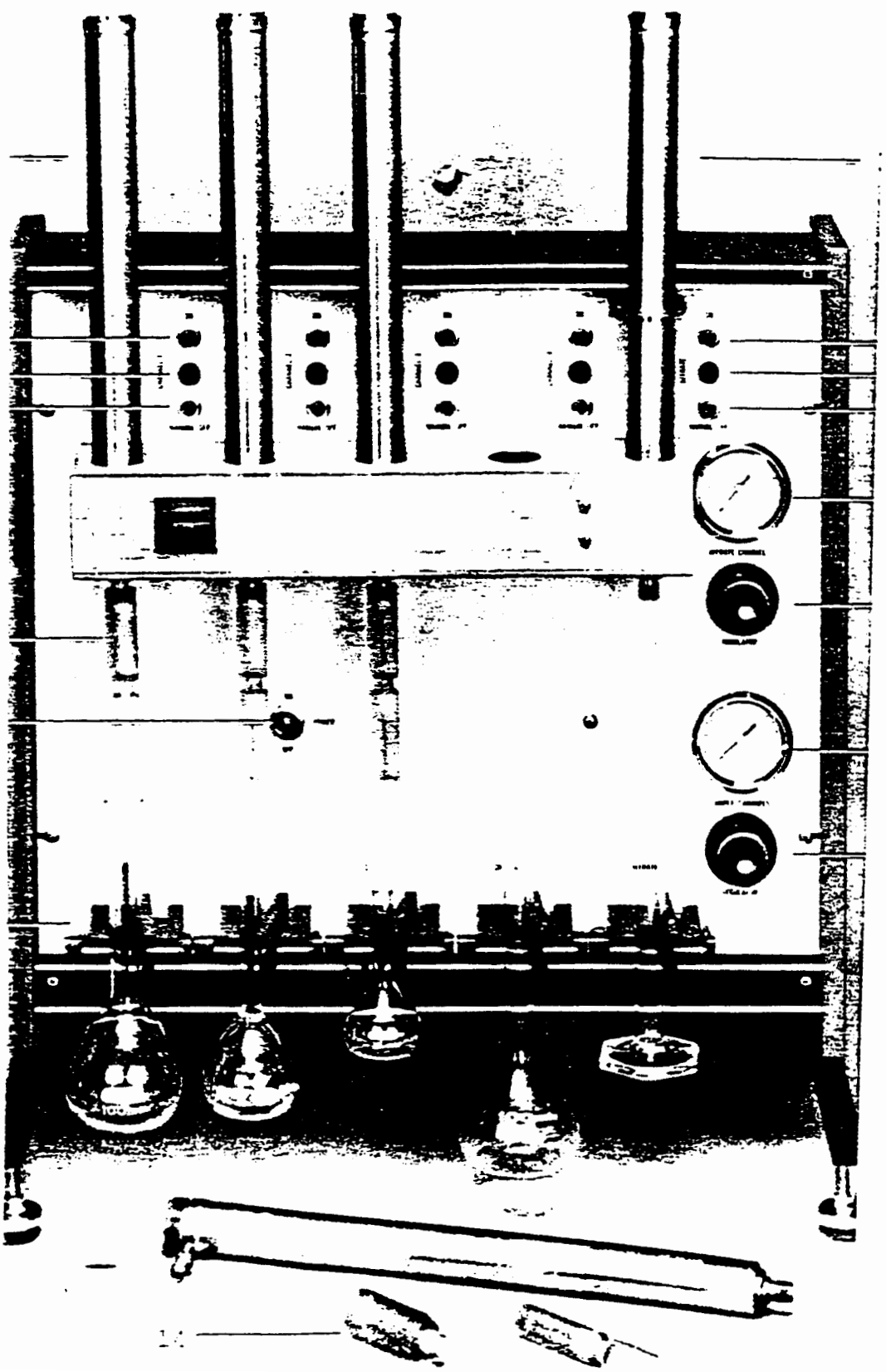


Figure 3

Use of CPI-packed™ TOX Columns

I. Installing adsorption module adapter fittings

Adapter fittings must be installed to use CPI-packed™ TOX columns. The procedure is very simple, takes only minutes and does not alter the equipment factory warranty.

Important: To ensure contamination free analysis, the adsorption module adapters and ejection tool must be thoroughly cleaned before use.

A. CPI adapter for DOHRMANN® adsorption module

(1) Insert Teflon® washers (P/N 4010-772) into the inner base of the adapters (P/N 4010-770).
(2) Screw cleaned column adapters (P/N 4010-770) directly onto adsorption module threaded ends.
(3) Tighten adapters to prevent leakage. Once installed, there is no need to remove the adapter.

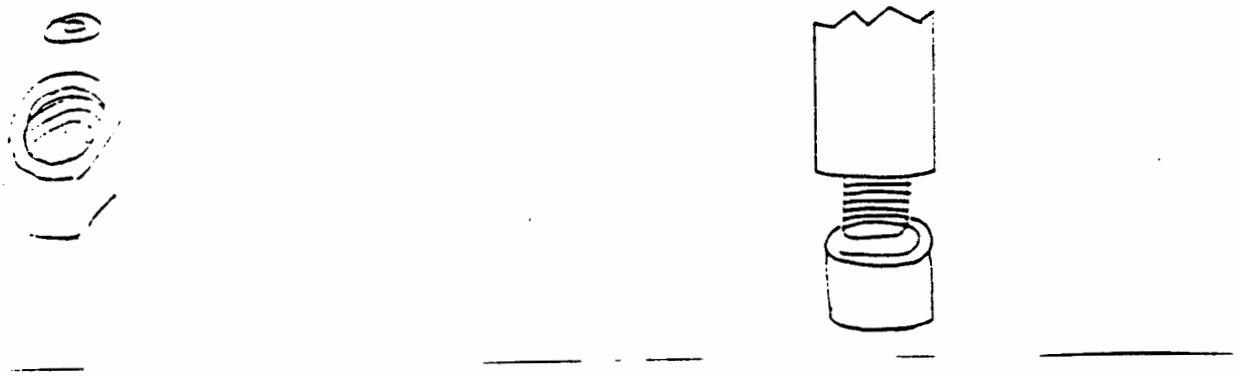


Figure 4

II. Preparing CPI-packed™ Columns

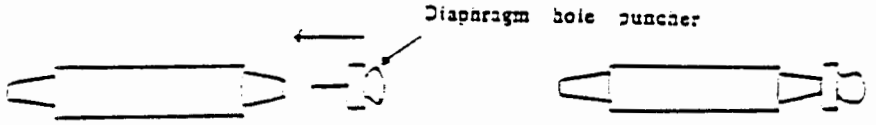
Each CPI-packed™ TOX column contains 40 mg Activated Carbon in an airtight cavity formed by two thin diaphragms. In order for the column to work properly, the bottom and top diaphragms must have a small hole pierced in them before use. To minimize contamination, do not puncture diaphragms until you are ready to use them.

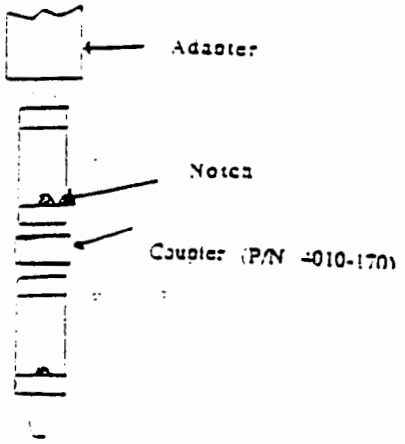
A. Piercing diaphragms and inserting columns into adsorption module:

(1) Hold the TOX column firmly in one hand and pierce diaphragm using the diaphragm hole puncher (P/N 4010-175) as shown.
(2) Repeat the above step for both ends of the column!
(3) Promptly insert column notched side down into adsorption module to prevent contamination, or spillage of GAC.
(4) Repeat steps 1 through 3 for a second column.
(5) Connect the first and second column together using a coupler (P/N 4010-170). Push firmly with a twisting motion (Use enough pressure to ensure a leakproof seal).

Note: It is extremely important to insert the column *notched side down*. This prevents GAC from escaping when the column is in use!

Note: When using a DOHRMANN® adsorption module, adjust the pressure to achieve the desired flow rate (10 to 20 psi).





5.8.6 When 100 mls of sample has been put through columns, take columns off of the sample reservoir and put them on the nitrate rinse reservoir. Turn on nitrogen pressure and force 2 mls of nitrate rinse solution through columns. For samples with high inorganic chloride use 10 mls of nitrate solution.

Detach columns and put each column into a clean sealable vial. Label each vial with sample number and whether column was the top or bottom column in the 2 column sequence. Rinse column holders and sample reservoirs well with type I water.

5.9 Pyrolysis Procedure:

5.9.1 Using the ejector tool, push the carbon out of the column (one column at a time) into the quartz boat.

5.9.2 Wait for a steady baseline on red electronic ug reading.

5.9.3 After baseline reading is steady, turn "MODE Selector Dial" from DET to INT and push START. The ready light will come on when analysis is complete and record ug reading on printer.

5.9.4 Vacuum out ash with Dustbuster.

5.9.5 Repeat for bottom column.

5.9.6 All samples must be done in at least duplicate (two columns of top and bottom each).

6.0 Daily Shutdown Procedure:

6.0.1 Disconnect the titration cell from the pyrolysis tube mount it on its rack in the cell housing.

6.0.2 Set the "FUNCTION Selection Dial" to STANDBY. <u>DO NOT</u> turn off the gas flows while the titration cell is connected to the pyrolysis tube, electrolyte may be sucked back into the pyrolysis tube unless positive gas flow is present.

6.0.3 Stop operating gas flows using the toggle valves. Do not disturb regulator settings.

6.1 Calculations:

$$\frac{(C_1 - C_3) + (C_2 - C_3)}{V} = \text{ug/L Total Organic Halide}$$

where:

C_1 = ug Cl^- on the top column in series.
C_2 = ug Cl^- on the bottom column in series.
C_3 = average ug for method blanks.
V = the sample volume in liters.

6.2 QUALITY CONTROL

6.2.1 Each day after the blank average is determined, run a standard consisting of 100 mL of the 100 ug/L Cl^- trichlorophenol standard. Theoretical value = 100.0 ug/L. Rerun this standard if it is not within 5% of the theoretical value.

6.2.2 Do a 'cell flush', a 'blank', and a 'calibration standard' after every eight pyrolysis burns (4 top and bottom samples).

6.2.3 Run a matrix spike and a matrix spike duplicate for every 20 samples of the same matrix.

6.2.4 Run one titration cell standardization each day before any sample analysis. Once baseline is steady, switch "MODE Selection Dial" to POX and "FUNCTION Selection Dial" to DET and make sure baseline is steady. Switch "Function Selection Dial" to INT, push start, wait 4 seconds and then inject 5 ul of 1000 ppm Cl^- solution into titration cell below level of electrolyte. This procedure should give a 5 ug reading. If the value obtained is not within ± 5% of the theoretical value, correct the problem and reanalyze.

DOHRMANN DX-20A
MISCELLANEOUS EXPLANATIONS/DEFINITIONS

1. TOX = TOTAL ORGANIC HALIDES

   Boat moves in and out.
   Vaporization/cool down↓ O_2 ← CO_2; oxidation↓ O_2 ← O_2
   Use adsorption module.

2 TX = TOTAL HALIDES (Extractables)

   Boat moves in and out.
   Vaporization/cool down↓ O_2↓ CO_2; oxidation↓ CO_2 ← O_2
   Syringe injection into little boat in ball cell

** Preferred method if there are no extractions or dilutions on original liquid sample; it will be the same as TOX.

3. EOX = EXTRACTABLE ORGANIC HALIDES

   No boat, straight injection.
   Vaporization/cool down↓ O_2↓ CO_2
   Press start first.
   Put needle in for 1 minute first to burn excess; push over period of time 40 ul after pulling back to approximately 45 ul until air bubble; then when done subtract to find actual injection amount.

4. POX = PURGIBLE ORGANIC HALIDES

   No boat; injection into sparger.
   Vaporization/cool down↓ CO_2 ← CO_2
   Have closed valve before running.
   Press start first, then inject.
   Aspirate sample back out with syringe to dispose.

ROSS	SOP Standard Operating Procedure	SOP No: ~~11-006-00~~ Inital Date: ~~2/1/91~~ Page 1 of 7 Date Revised:

Title:

Non-Purgable Organic Carbon (NPOC) in Waters

Prepared by:	Approved by:	Date:	QC Approval:
Belinda Minter	CHCaldwell	5/23/91	CHC

1.0 PURPOSE

To define the analytical procedures for the determination of NPOC in water samples.

2.0 SCOPE

Waters are analyzed for NPOC by UV-promoted wet chemical oxidation. Nonpurgeable organic carbon is converted to CO_2 which is then measured directly by an infra-red detector.

3.0 REFERENCES

3.1 EPA SW-846, 3rd Edition, Method 9060.

3.2 EPA Methods for Chemical Analysis of Water and Wastes, 1983 Method 415.1

3.3 Rosemount/Dohrman DC-80 Total Organic Carbon Systems Manual.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Apparatus

5.1.1 Dohrmann Model DC-80 Total Organic Carbon Analyzer with Autosampler.

5.1.2 Syringe, 1 mL.

5.2 Reagents

5.2.1 Oxygen, 99.9% pure.

5.2.2 Reagent Water - Type I water from the Labconco system.

5.2.3 Potassium Persulfate Solution, 2% - dissolve 20 g of reagent grade $K_2S_2O_8$ in one liter of Type I water. Add 1 ml concentrated phosphoric acid and mix well. Store in a cool dark location. Shelf life is approximately one month.

5.2.4 Organic Carbon Standard, 2000 ppm - add 425 mg dried potassium hydrogen phthalate (KHP) to type I water in a 100 mL volumetric flask. Add 0.1 mL concentrated phosphoric acid and bring to volume. Store in dark glass under refrigeration and replace monthly.

5.2.5 Organic Carbon Standard, 400 ppm - dilute 20.0 mL of the 2000 ppm standard to 100 mL with type I water. Store in dark glass under referigeration and prepare fresh weekly.

5.2.6 Organic Carbon Standard, 10.0 ppm - dilute 1.0 mL of the 2000 ppm standard to 200 mL with type I water. Prepare fresh daily.

5.3 Instrument Set-Up

5.3.1 Fill reactor with potassium persulfate solution.

5.3.2 Connect all tubing as in Figure 1.

5.3.3 Put reagent line in reagent bottle.

5.3.4 Set pump tubing and put pressure fingers on.

5.3.5 Turn on in order: autosampler POWER, detector POWER (the detector should be already on, so hit the POWER button twice), and reactor (POWER, PUMP, LAMP). The printer should print 'SELFTEST, NO ERRORS'.

5.3.6 The gas should be on at 30 psi; if so, turn the gas toggle switch on. View the reactor through the viewing port in the door to insure the lamp is lit and carrier gas is flowing.

5.3.7 With DETECTOR/PPM switch in <u>DETECTOR</u> position, allow baseline to stablilize and adjust to 0.0100.

5.3.8 Let the lamp warm up for at least 10 minutes.

5.3.9 Set DETECTOR/PPM switch to PPM. Set TOC/POC switch to TOC.

5.3.10 Remove any previous calibrations by depressing the yellow CALIBRATE button for several seconds. The printer will print 'NO CAL' and the range and the yellow CALIBRATE light should go out.

5.4 Calibration

5.4.1 Low-Level TOC - For samples with an expected TOC level between 0.1 - 20 mg/L, use the 1000 uL sample range. Calibration is performed using the 10.0 ppm organic carbon standard. Begin instrument calibration by injecting 1000 uL of 10 ppm standard. Repeat until you get a consistent reading and press CALIBRATE button. The yellow CALIBRATE button should illuminate.

5.4.2 Mid-Level TOC - For samples with an expected TOC level between 10-800 mg/L, use the 200 uL sample loop and range. Calibration is performed as described for the Low-Level Method, but using the 400 ppm TOC standard.

5.4.3 High-Level TOC - For water samples with an expected TOC level between 100-4000 mg/L, use the 40 uL loop and range. Calibration is performed as described for the Low-Level Method, but using the 2000 ppm TOC standard.

5.5 Analysis

5.5.1 A system blank (used only in Low-Level TOC analysis) is obtained by withdrawing 1.0 mL of reactor fluid with a syringe through the septum located at the front of the reactor. This is manually injected into the sample port immediately after the green START button has been depressed. Repeat until a consistent blank is obtained.

5.5.2 The sample must be preserved to a pH ≤ 2 with H_2SO_4 and purged to remove inorganic carbon due to carbonates and bicarbonates. During the removal of inorganic carbon, purgeable organic carbon is also removed. The autosampler is designed to purge the samples before injection into the system (Note 1). Once tubes are filled and positioned in the tray, automatic operation is used (Note 2).

Figure 1
Liquid Connectionn, Extrnal, DC-80 and ASM-1

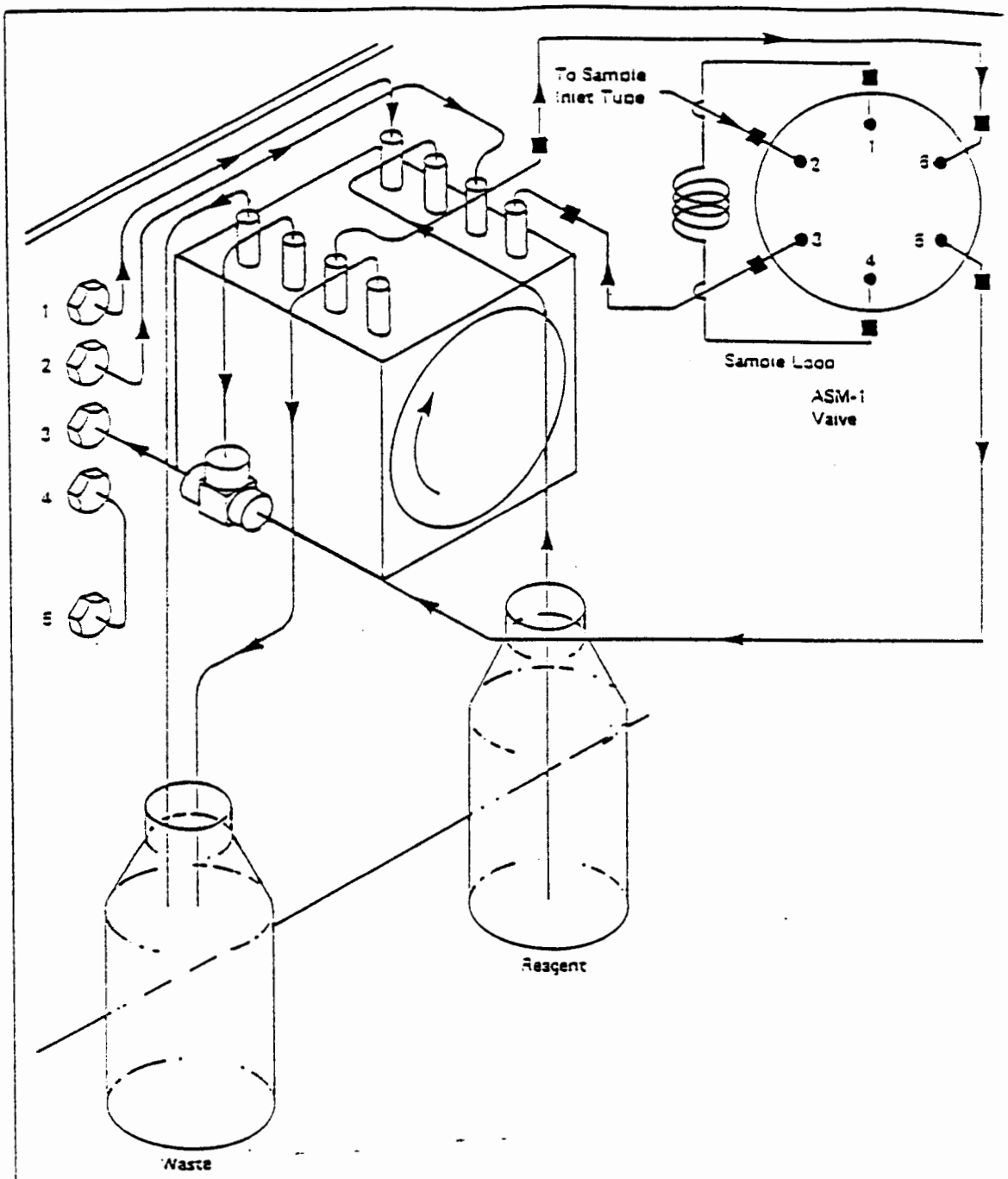


Figure 1
Liquid Connections, External, DC-80 and ASM-1

5.6 Autosampler

5.6.1 Load the sample table in clockwise direction starting in the outside row to the right of gray plastic plug. The first tube in the inside row should be placed directly in line with the gray plastic plug if you have >60 samples. Position numbers 1 and 2 (60 and 61 if inside row is needed) should have standards.

5.6.2 Rotate the sample table manually COUNTERCLOCKWISE to position the sample dip tube over tube one. **The sample table must not be rotated clockwise as this will damage the drive motor clutch.** If the dip tubes are not positioned for the outside row, they can be moved by pulling out on the base of the pneumatic operator mechanism at the rear of the sampler with the autosampler power OFF.

5.6.3 Place the magnet two spaces over from the last test tube (a deionized rinse) to automatically stop the autosampler after the last sample.

5.6.4 Each time you run the autosampler make sure you have the correct sample loop and syringe. (i.e. 40 uL = black/yellow together, no loop; 200 uL = 200 uL loop; and 1000 uL = 1000 uL loop).

5.6.5 Using the manual mode of the autosampler two tubes of of the standard are analyzed. With the first tube located under the dip tube, turn on autosampler power. Press yellow MANUAL button, then press green GO button. After the reading is displayed and the READY button is illuminated, press the CALIBRATE button. Do not hold the button longer than one second so as not to disengage the previous calibration. Verify the correct calibration by pressing the green GO button for another MANUAL run. If calibration is satisfactory, press the red AUTO button and the system will run out the group of samples and record the data on the printer.

5.7 Calibration

mg/L NPOC = (mg/L of sample - mg/L of system blank)

5.8 Instrument Shutdown:

5.8.1 Turn off autosampler POWER and lamp POWER, leave detector POWER on. The red ERROR button should illuminate.

5.8.2 Remove pressure fingers.

5.8.3 Remove reagent line from reagent bottle.

5.8.4 Turn off gas toggle switch.

6.0 QUALITY CONTROL

6.1 Analyze an SRS as an initial calibration verification standard (4th tube), after every 10 samples, and just before the deionized rinse at the end of a run.

6.2 Analyze a matrix spike/matrix spike duplicate pair every twenty samples.

6.3 All samples should be run in duplicate.

NOTES

1. Remember that this analysis actually gives a value for Non-Purgeable Organic Carbon (NPOC) though this is the test which is expected by most clients when they request TOC. Any volatile organic carbon is removed with the inorganic carbon during purging. To get true Total Organic Carbon (TOC), Purgeable Organic Carbon (POC) must also be determined and added to the NPOC value.

2. Sometimes interferences (such as solid material or high chloride) are present which do not allow the sample to be completely chemically oxidized in the time of the run (See Manual).

MISCELLANEOUS EXPLANATIONS/DEFINITIONS

1. TOC = Total Organic Carbon = (POC + NPOC) or (TC - IC) (reported as NPOC or as TOC by sparging)

2. NPOC = Non-Purgeable Organic Carbon = (OC - POC) (remaining carbon matter after sample is pretreated with acid and sparged)

3. POC = Purgeable Organic Carbon (organic carbon matter that is transferred to the gas phase when the sample is sparged and passes through the CO_2 scrubber)

4. OC = Organic Carbon = POC + NPOC

5. IC = Inorganic Carbon (converted to CO_2 when acid is added and released during sparging)

6. TC = Total Carbon = (IC + OC) or (IC + POC + NPOC) (straight sample; no pretreatment, no sparging)

   TC = Total Carbon = IC (This can be done by running the sample with sparging, no pretreatment, and the UV lamp off.)

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title:	Determination of Metals Using the Thermo Jarrell Ash Enviro 36 ICP by EPA SW-846 Method 6010A		
Prepared by:	Approved by:	Date: 10/21/96	QA Approval:

1.0 PURPOSE

The purpose of this SOP is to describe the determination of metals that have been appropriately prepared, using the Thermo Jarrell Ash Enviro 36 ICP.

2.0 SCOPE AND APPLICATION

This SOP applies to ICP emission analyses performed using the Thermo Jarrell Ash Enviro 36. The method described includes calibration and quality control criteria defined by EPA SW-846 Method 6010A.

The method may be used for all matrices, including ground water, aqueous samples, mobility extracts, industrial and organic wastes, soils, sludges, sediments, biologic substances and other solid or semisolid wastes.

These matrices require digestion prior to analysis. The digestion procedures (e.g., SW-846, Methods 3005 - 3050) are not provided by this document but may be found in other SOPs. When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

The elements for which this method may be employed and their respective reporting limits for clean aqueous samples using pneumatic nebulization are provided in Section 12.0, Table 1. The reporting limits, called Estimated Quantitation Limits (EQLs), are established by RASI. These have been experimentally verified with the system in use.

The TJA Enviro 36 performs simultaneous detection of elements. The method measures element-emitted light by optical spectrometry. Samples and standards are nebulized and the aerosol transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio frequency (RF) generated, inductively coupled plasma. The torch is radially mounted (in a vertical position). The spectra are dispersed by a grating spectrometer, and intensities of the lines are monitored by photomultiplier tubes. Intensities are calibrated using known concentrations and used to quantify sample results.

Note: This SOP combines the former SOPs MT-001-01 (Calibration and Operation of the Thermo Jarrell Ash Enviro 36) and MT-002-00 (Quality Control Procedures for the Thermo Jarrell Ash Enviro 36 Using Method 6010A).

3.0 REFERENCES

ICAP 61E Spectrometer Operators Manual. Thermo Jarrell Ash Corporation, Franklin, MA, Feb. 1991

EPA Test Methods for Evaluating Solid Waste, SW-846 Method 6010A, Rev. 1, July, 1992

4.0 ASSOCIATED SOPs

MT-002, Analysis of Metals Using the Thermo Jarrell Ash Enviro 36 ICP by EPA Method 200.7

IP-003, Digestion Procedure for Metals in Aqueous Samples Using SW-846, Methods 3010A, 3020A

IP-004, Digestion Procedure for Metals in Solids and Non-Aqueous Wastes Using SW-846, Method 3050A

5.0 EQUIPMENT

5.1 Apparatus

5.1.1 Thermo Jarrell Ash Enviro 36 ICAP, with host computer (minimum 386), printer, coolant recirculating pump, autosampler, peristaltic pump and ancillary gas connections

5.1.2 Class A volumetric flasks - 100, 200, 500, 1000 and 2000 mL

5.1.3 Class A volumetric pipettes - 1, 2, 5, 10 and 20 mL

5.1.3 Auto pipettors and pipet tips, 1 - 10 mL and 1 - 1000 uL

5.1.4 Ventilation hood with minimum flow of 100 cfm

5.1.5 Peristaltic pump tubing (0.045 mm diameter - red/red)

5.1.6 Autosampler standard cups and sample test tubes

5.1.7 Polyethylene storage bottles

5.2 Reagents

5.2.1 ASTM Type 1 water otherwise known as high purity reagent water

5.2.2 Argon, gaseous bleed from liquid source (minimum 70 psi)

5.2.3 Nitric acid, concentrated

5.2.4 Hydrochloric acid, concentrated

5.2.5 Nitric acid, (1:1) - Add 500 mL concentrated acid to 400 mL water and dilute to 1 liter

5.2.6 Hydrochloric acid, (1:1) - Add 500 mL concentrated acid to 400 mL water and dilute to 1 liter

5.3 Standards

5.3.1 Calibration Blank Standard - 2% HNO_3 / 5% HCl

To a 2 liter flask add approximately 1 liter of reagent water. Then, add 40 mL concentrated HNO_3 and 100 mL concentrated HCl . Bring to volume with reagent water and mix well. Store in a polyethylene bottle.

5.3.2 Calibration Standard (STD95) - mixed element standard in 2% HNO_3 / 5% HCl

To a 500 mL flask add approximately 300 mL reagent water. Then, add 10 mL concentrated HNO_3 and 25 mL concentrated HCl . Then add 5 mL each of Plasmachem XRAS1, XRAS2 and XRAS3. Bring to volume with reagent water and mix well. Store in a polyethylene bottle. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use.

The Plasmachem XRAS standards are custom mixes. When prepared as described, the standard will contain the following elements and respective concentrations:

CALIBRATION STD95

ELEMENT	CONCENTRATION (mg/L)
Aluminum	50
Antimony	5.0
Arsenic	10
Barium	1.0
Beryllium	0.5
Boron	2.5
Cadmium	1.0
Calcium	50
Chromium	2.5
Cobalt	2.5
Copper	2.5
Iron	50
Lead	5.0
Lithium	2.5
Magnesium	50
Manganese	1.0
Molybdenum	2.5
Nickel	2.5
Potassium	50
Selenium	10
Silicon	10
Strontium	1.0
Silver	0.5
Sodium	50
Thallium	5.0
Tin	10
Titanium	2.5
Vanadium	1.0
Zinc	1.0
Zirconium	10

5.3.3 Initial Calibration Verification Standard (ICV95) - mixed element standard in 2% HNO_3 / 5% HCl

To a 500 mL flask add approximately 300 mL reagent water. Then, add 10 mL concentrated HNO_3 and 25 mL concentrated HCl . Then add 5 mL each of Exaxol, PE Pure, Plasmachem, Solutions Plus or equivalent QC-19A, QC-19B and QC-19C. Bring to volume with reagent water and mix well. Store in a polyethylene bottle. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use.

The Exaxol, PE Pure, Plasmachem, Solutions Plus or equivalent standards are custom mixes. The source for the ICV must be different than that used for the calibration. The same vendor may be used as long as the source is different. Note that different lot numbers for solutions may still be from the same source. When prepared as described, the standard will contain the following elements and respective concentrations:

INITIAL CALIBRATION VERIFICATION STANDARD - ICV95

ELEMENT	CONCENTRATION (mg/L)
Aluminum	10
Antimony	5.0
Arsenic	5.0
Barium	0.5
Beryllium	0.5
Boron	1.0
Cadmium	0.5
Calcium	10
Chromium	1.0
Cobalt	1.0
Copper	1.0
Iron	10
Lead	5.0
Lithium	1.0
Magnesium	10
Manganese	0.5
Molybdenum	1.0
Nickel	1.0
Potassium	10
Selenium	5.0
Silicon	5.0
Strontium	0.5
Silver	0.5
Sodium	10
Thallium	5.0
Tin	5.0
Titanium	1.0
Vanadium	1.0
Zinc	1.0
Zirconium	5.0

5.3.4 Low-Level Check Standard Stock Solution (CRDL-2) - mixed element standard in 2% HNO_3 / 5% HCl

To a 100 mL flask add approximately 10 mL reagent water. Then, add 2 mL concentrated HNO_3 and 5 mL concentrated HCl. Add the following amounts of single element standards and bring to volume. Store in a polyethylene bottle. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use. When made as described the following mixture of elements and respective concentrations are achieved:

LOW-LEVEL CHECK STANDARD STOCK SOLUTION - CRDL-2

ELEMENT	STOCK CONC. (mg/L)	VOLUME (uL)	CONCENTRATION(mg/L)
Aluminum	10,000	200	20
Antimony	1000	2000	20
Barium	1000	80	0.8
Arsenic	1000	4000	40
Beryllium	1000	40	0.4
Boron	1000	1000	10
Cadmium	1000	100	1.0
Calcium	10,000	400	40
Chromium	1000	200	2.0
Cobalt	1000	200	2.0
Copper	1000	400	4.0
Iron	10,000	200	20
Lead	1000	1000	10
Lithium	1000	400	4.0
Magnesium	10,000	200	20
Manganese	1000	100	1.0
Molybdenum	1000	200	2.0
Nickel	1000	400	4.0
Potassium	10,000	400	40
Selenium	1000	2000	20
Silicon	1000	10000	100
Strontium	1000	400	4.0
Silver	1000	200	2.0
Sodium	10,000	1000	100
Thallium	1000	4000	40
Tin	1000	2000	20
Titanium	1000	1000	10
Vanadium	1000	200	2.0
Zinc	1000	400	4.0
Zirconium	1000	600	6.0

5.3.5 Low-Level Check Standard (CRI6010A) - mixed element standard in 2% HNO_3 / 5% HCl

To a 100 mL flask add approximately 50 mL reagent water. Add 2 mL concentrated HNO_3 and 5 mL concentrated HCl. Then, add 1 mL of Low Level Check Standard Stock Solution (CRDL-2) and bring to volume. The resulting concentrations will be 100 times lower than that for CRDL-2, provided above. The solution should be prepared more frequently than that for the calibration and ICV standards because the low levels are not as stable for long periods. Weekly preparation of this standard is recommended.

5.3.6 Interference Check Standard solution A. (ICSA) - mixed element standard in 2% HNO_3 / 5% HCl

To a 500 mL flask add approximately 300 mL reagent water. Then, add 10 mL concentrated HNO_3 and 25 mL concentrated HCl . Add 50 mL of Exaxol, PE Pure, Plasmachem, Solutions Plus or equivalent ICSA solution. Listed below are the elements and concentrations for the stock solution of ICSA solution and the final concentration when diluted:

INTERFERENCE CHECK STANDARD A - ICSA

Elements	Stock Concentration (mg/L)	Final Concentration (mg/L)
Al, Ca, Mg	5000	500
Fe	2000	200

5.3.7 Interference Check Standard solution AB (ICSAB95) - mixed element standard in 2% HNO_3 / 5% HCl

This standard is prepared in two parts. The first part consists of preparing the Odd Analytes B Stock Standard. This standard is then added with others to form the final mixture.

Odd Analytes B Solution - To a 100 mL flask add 50 mL reagent water, 2 mL of concentrated HNO_3 and 5 mL concentrated HCl . Add the following amounts of 1000 ppm, single element standards and dilute to a final volume of 100 mL to achieve the following element mix and respective concentrations:

ODD ANALYTES B SOLUTION

ELEMENT	VOLUME (uL)	CONCENTRATION (mg/L)
Lithium	1000	10
Potassium	1000	10
Silicon	5000	50
Strontium	1000	10
Tin	1000	10
Titanium	1000	10
Zirconium	1000	10

Alternatively add 100 uL of 10,000 ppm stock (500 uL for silicon); other analytes may also be added in this manner if needed.

Final ICSAB solution - To a 500 mL volumetric flask add about 250 mL reagent water, 5 mL concentrated HNO_3 and 10 mL concentrated HCl. Then, add 50 mL ICSA solution (see Section 5.3.6), 5 mL ANALYTES B solution, 5 mL ALT ANALYTES B solution, and 50 mL of ODD ANALYTES B solution. The ANALYTES B and ALT ANALYTES B solutions are mixed standards purchased directly from a standard distributor, (e.g. Plasmachem, Exaxol, Solutions Plus, etc.). Bring to a final volume of 500 mL to achieve the following element mix and respective concentrations:

INTERFERENCE CHECK STANDARD WITH ANALYTES SOLUTION - ICSAB95

ELEMENT	CONCENTRATION (mg/L)
Aluminum	500
Antimony	1.0
Arsenic	1.0
Barium	0.5
Beryllium	0.5
Boron	1.0
Cadmium	1.0
Calcium	500
Chromium	0.5
Cobalt	0.5
Copper	0.5
Iron	200
Lead	1.0
Lithium	1.0
Magnesium	500
Manganese	0.5
Molybdenum	1.0
Nickel	1.0
Potassium	1.0
Selenium	1.0
Silicon	5.0
Strontium	1.0
Silver	1.0
Sodium	1.0
Thallium	1.0
Tin	1.0
Titanium	1.0
Vanadium	0.5
Zinc	1.0
Zirconium	1.0

5.3.8 Continuing Calibration Verification Standard (CCV95) - mixed element standard in 2% HNO_3 / 5% HCl

To a 500 mL flask add approximately 100 mL reagent water. Then, add 10 mL concentrated HNO_3 and 25 mL concentrated HCl . Add 10 mL each of QC-19A, QC-19B and QC-19C stock solutions. Bring to volume with reagent water and mix well. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use.

The CCV source may be the same as that of the calibration standards. When prepared as described, the standard will contain the following elements and respective concentrations:

CONTINUING CALIBRATION VERIFICATION STANDARD - CCV95

ELEMENT	CONCENTRATION (mg/L)
Aluminum	20
Antimony	10
Arsenic	10
Barium	1.0
Beryllium	1.0
Boron	2.0
Cadmium	1.0
Calcium	20
Chromium	2.0
Cobalt	2.0
Copper	2.0
Iron	20
Lead	10
Lithium	2.0
Magnesium	20
Manganese	1.0
Molybdenum	2.0
Nickel	2.0
Potassium	20
Selenium	10
Silicon	10
Strontium	1.0
Silver	1.0
Sodium	20
Thallium	10
Tin	10
Titanium	2.0
Vanadium	2.0
Zinc	2.0
Zirconium	10

5.3.9 High Calibration Standard Check (HSTD95) - Mixed element check standard

Prepare in the same manner as STD95 described in section 5.3.2, (an aliquot of STD 95 is used for HSTD95).

6.0 PROCEDURE

6.1 Instrument Set-up

6.1.1 The hardware should already be set up. The instrument requires electrical power, coolant water, argon gas and ventilation. It should be operated in a thermally stable environment at a temperature between 15 to 20°C.

6.1.1 After turning on the computer, the ThermoSPEC software should be booted automatically. If it didn't, type in at the C:\> prompt STNRUN then press ENTER.

6.2 Instrument Ignition.

6.2.1 Check the supply of argon weekly. The tank is located outside. It is filled with liquid argon which gasses to supply the ICP. A gauge displays the volume of argon remaining in the tank. When the gauge drops to 1/4 full level place an order for a refill. Under typical work load the tank will last several months between fillings.

6.2.2 The argon gas is regulated using a single stage regulator prior to connection to the ICP. The regulator pressure should be set to 60 psi. A second regulator is located in the torch chamber of the ICP. The gauge for it can be viewed by looking up from underneath the unit. This regulator has an adjustment valve that is also accessed from under the torch chamber. It should be adjusted to supply a pressure of 50 psi to the unit when the torch is lit.

6.2.3 Check the drain container to make sure that the black drain tube is looped and the end is immersed at least eight inches in the fluid. The container should be empty enough to allow for the drainage of the current run.

6.2.4 The on/off switch for the recirculating pump is located on the power cord. Turn the power on.

6.2.5 Wrap two peristaltic pump tubes (0.045 mm - red/red) around the pump wheel. Rotate the tension arm against the tubing, making sure that the tubing fits in the grove on the tension arm completely. Then rotate the tension arm holder until the holder locks into place. The upper tubing will have one end attached to the capillary tubing coming from the Autosampler and the other end attached to capillary tubing going to the torch nebulizer. The bottom tubing will have one end attached to the capillary tubing coming from the rinse beaker and the other end attached to the capillary tubing going to the rinse tube on the Autosampler. Replace the tubing daily or more frequently if they look worn out or highly discolored. While looking at the front of the pump the right side of the tubing holder will contain the ends that come from the Autosampler probe and from the rinse beaker. Turn the toggle switch to the left and the wheel will be turning counterclockwise. The pump speed must be set at 550 RPM. Check that all flow directions are correct.

6.2.6 Fill the rinse beaker with a 5% solution of nitric acid. Although it has been suggested to match the acid concentration of standards and samples it has been found that excessive carryover occurs for several elements using this protocol.

6.2.7 Raise the argon toggle valves, located to the right of the nebulizer on the front of the instrument, for the TORCH and SAMPLE. Keep the valves in this position for a few minutes before igniting the torch.

6.2.8 Turn the POWER Control knob, located between the FORWARD KILOWATTS RF POWER Meter and the REFLECTED WATTS RF POWER Meter, counterclockwise until it stops.

6.2.9 Turn the AUTOMATIC POWER CONTROL switch, located on the right hand panel in the upper left hand corner, to MANUAL. Check to make sure the FORWARD POWER switch, located below AUTOMATIC FORWARD POWER CONTROL knob, is ON.

6.2.10 The blue RF OFF button should be illuminated. If it isn't, check to make sure all the compartment doors are closed, the argon is on, and the recirculating pump is on.

6.2.11 Lower the argon toggle valve for the SAMPLE. Press the RF ON button. This button should now be illuminated.

6.2.12 Slowly turn the POWER Control Knob clockwise until the Forward RF Power meter reads about 0.5 kilowatts. Press the IGNITOR button located below the TORCH Argon toggle switch. The Argon should be swirling around inside the torch. Slowly, keep turning the POWER Control Knob clockwise until the plasma forms, then immediately turn the AUTOMATIC POWER CONTROL SWITCH up, rotate the POWER CONTROL knob clockwise until it stops, and slowly raise the argon toggle valve for sample. The FORWARD KILOWATTS RF POWER METER should now read about 1 kilowatt.

6.3 Instrument Operation

6.3.1 Before starting any analyses, and after igniting the plasma, let the instrument thermally stabilize for at least 30 minutes.

6.3.2 Profile the instrument by aspirating a manganese standard. Highlight the **Setup** pulldown menu and then highlight **Profile** and press Enter.

6.3.3 Press F1 for **Manual Profile**, or F3 for **AUTOMATIC PROFILE** then press Enter.

6.3.4 For automatic profiling enter a flush time of 25 seconds and aspirate the manganese standard. Press F1 (run) to begin the automatic profile operation. When profiling is completed check the peak position. If it is greater than ±0.5 perform manual profiling described in Section 6.3.5. If the peak position is less than 0.5, press F1 (calculate SS). When the vernier position comes on the screen press ENTER. The new vernier position will appear on the upper-right of the screen. Move the caliper to the new position and press ESC. Check the precision of the profile by repeating the above steps. When the peak position reads within ±0.1 accept the profile by pressing F9.

6.3.5 For a manual profile enter a flush time of 25 seconds and aspirate the manganese standard. The CONTROLLER meter located on the left-hand side of the instrument must be reading greater than 100. In the event it is not, double check that the standard is being aspirated and that the message "Performing Exposure" is flashing in the upper left-hand corner of the screen.

6.3.5 Open the hatch located at the top-middle of the instrument. Adjust the caliper counterclockwise until the CONTROLLER meter reads exactly 60, then adjust it clockwise to 70. Read the caliper and enter the number for the LOW value, then press Enter. Next, adjust the caliper clockwise until the CONTROLLER meter rises to 100, then back it down to 70. Read the caliper and enter the number for the HIGH value, then press Enter. The software will give the average of the HIGH and LOW values. Adjust the caliper counterclockwise to the average. Press F9 to accept the profile calibration and then ESC to get back to the Main menu.

6.4 Developing an Autosampler Table

6.4.1 There are several useful function keys while editing an Autosampler Table. There are three different menus for the function keys. The first menu is always shown when either the Alt or Crtl key is pressed. The second is the menu shown when the Alt key is held down. The third is the menu shown when the Crtl key is held down. The beginning analyst should use only the main and Alt menu functions. Those two menus allow the editing of the Autosampler Table.

6.4.2 Choose the **OPERATION** pulldown menu.

6.4.3 Under the **OPERATION** Menu Choose **AUTOSAMPLER SETUP**. If a large number of sample positions are needed (more than 40), build the table using ASEDIT.EXE under the **EXIT** menu.

6.4.4 Enter a name for the Autosampler Table. The default is the last one modified. Use the following guideline for naming the autosampler table: Numerical date followed by alphabetical sequence of runs performed that day (i.e. For a table analyzed on the second run of the day on April, 1 1995 the table should be labeled 040195B.) After typing in a name press ENTER.

6.4.5 Press F3 to **ADD SET** to the Autosampler file. The arrow keys will allow the analyst to move between the prompts. For the Set Description type ICP RUN and the date of analysis. For the Method Name type ICP95. The other fields except "Auto-Rinse Between Samples" and "Rinse Time" are not required but are only there to help distinguish between runs for different clients or projects and when used, aid in data base searches. The "Auto-Rinse Between Samples" must be set to YES. Highlight the answer and press the space bar to toggle between "YES" and "NO". Type in 60 for the amount of seconds rinsed between samples. A 60 second rinse has been found to be adequate for most analyses. The analyst can increase this time if carryover is apparent, but must not decrease the time as it is a method requirement.

6.4.6 The next prompt is "# Sample Positions needed". A rule of thumb here is to guess high. It is easier to delete unused sample positions than to add more. The default for the next prompt "Starting Rack Number" is 2, (Rack 1 is used for calibration and QC standards). The default for the prompt "Starting Position Number" is 1. Both of the above can be changed if needed. Highlight the answer area for "Default Sample Name". Press the space bar once and this will delete the default "Sample-1". Highlight the answer area for the prompt "Auto Increment?". Press the space bar to toggle to "No" or "YES" depending on how many sequential sample ID numbers are in the analysis set and the ease afforded the operator in building the table. The prompt "Default Limit Check Table Name" should be left as is. Unless all the samples have the same dilution factors the prompt "Default Correction Factor" should be left at 1.

6.4.7 Press F1 to Edit Samples. Then press F3 and add HSTD95 . While the first sample is highlighted press F5 to Insert Standards. Use the arrow keys to go to the CheckTable column for the ICB. The default is CCB6010A. Use the arrow keys to go to the CheckTable column for CCB. Change the table to reflect the current analysis (CCB6010A.) Press Crtl-F3, and type ICS, then press ENTER to insert the Interference Check Standards. Use the arrow keys to go to the CheckTable column for ICSA and ICSAB. The defaults are ICSA95 and ICSAB95. The analyst may now begin entering samples. Listed below are a few rules for entering samples to make data importation and reduction easier. The series of calibration and check standards should be as follows: Blank, STD95, HSTD95, ICV, ICB, CRI6010A, ICSAI, ICSABI, CCV1 AND CCB1.

6.4.8 Always start off with the Preparation Blank for a set of samples. Enter the method and date of preparation for the set (i.e. if the samples were prepared by Method 3050 on 02/11/91 then type in PB 3050 02/11/91), or use a unique ID provided by sample preparation.

6.4.9 Enter the Laboratory Control Sample for a set of samples, right after the Preparation Blank. Enter the preparation method and the date of preparation (i.e. if the samples were prepared by Method 3010 on 02/11/91 then type in LCS 3010 02/11/91), or use a unique ID provided by sample preparation.

6.4.10 Enter the entire Ross Analytical work order number for the sample, for example 95-00-000-00A. If the sample is a duplicate, enter 95-00-000-00AD. If the sample is a spike or spike duplicate, enter 95-00-000-00AS or 95-00-000-00ASD. On samples that need dilutions, try to fit the whole work order number with the dilution. If necessary, truncate from the left of the work order number until the dilution and sample fit in the space allowed.

6.4.11 After entering each set of 10 samples, (including preparation blanks, LCS, MS/MSD, PS/PSD and other QC samples) insert CCVs and CCBs. The ICS standards must be run at least twice every eight hours or at the beginning and end of an analytical run, which ever is more frequent. If the analysis is anticipated to last more than eight hours insert an ICS macro to conform with this rule. Always end your table with an ICS sequence and CCV/CCB set. Remember to change the CheckTable column to reflect the method QC requirements for the CCV, CCB and ICSAB.

6.4.12 After all the samples have been added to the table, delete any unused sample positions by highlighting the unused position and pressing F2. After all the unused sample positions have been deleted press F9 until the computer displays in the upper left-hand corner the message "Examining Autosampler Table." Press F2 to print a listing of the autosampler table developed. When printing is complete, press F9 to save the autosampler table and return to the main menu.
Update the method as described in the next section.

6.5 Updating the Method

6.5.1 Select the **Development** pulldown menu from the **Main** menu and then select **Methods**.

6.5.2 Type in ICP95, if it is not the default answer to the prompt, then hit Enter.

6.5.3 Highlight the answer to the prompt "Analysis Data File". Type in the date of analysis (i.e. if the date is 04/01/95 type in APR0195.)

6.5.4 Highlight the answer to the prompt "Autosampler Table". Type in the name of the autosampler table (see Section 6.4 for autosampler table set up.)

6.6 Limit Check Tables

6.6.1 Select the **DEVELOPMENT** menu from the main menu by either using the arrow keys or by pressing the letter D.

6.6.2 Select **SAMPLE LIMIT CHECKS** by either using the arrow keys or by pressing the letter L.

6.6.3 Type in the name of the limit table, if known, then press ENTER, or press F6 to get a list of available tables. When choosing from a list enter the number of the corresponding table then press ENTER twice to edit the table.

6.6.4 The table LIM6010A contains the Linear Range for HIGH LIMIT and the EQL for the LOW LIMIT. It is used for checking acceptable result ranges for unknown samples.

6.6.5 The table CCB6010A is set up to flag analyte concentrations that exceed the absolute value of the EQL.

6.6.6 The table ICSA95 is set up to flag concentrations of interferences (i.e. Al, Ca, Fe and Mg) that exceed a range of 80 - 120% of the true value, and to flag analyte concentrations that exceed the absolute value of the EQL .

6.6.7 The table ICSAB95 is set up to flag concentrations of interferences and analytes that exceed a range of 80 - 120% of the true value.

6.7 Quality Control Check Tables

6.7.1 The table CCV95 is set up to flag analyte concentrations which exceed a range of 90 - 110% of the true value.

6.8 Instrument Shutdown Following Completion of Analysis

6.8.1 Extinguish the plasma by pressing the **RF OFF** button located on the middle instrument panel on the right hand side.

6.8.2 Remove the pump tubing from around the pump wheel by pressing the tension arm release located on the right hand side of the peristaltic pump. The pressure must be released when the pump is not turning or it causes deformation of the tubing and will lead to analysis problems.

6.8.3 Turn the **POWER CONTROL** knob counterclockwise until it stops.

6.8.4 Flip the **AUTOMATIC POWER CONTROL** switch to MANUAL.

6.8.5 Flip down the **TORCH** and **SAMPLE** argon toggle switches.

6.8.6 Turn off the timer.

7.0 ANALYSIS

7.1 After starting and equilibrating the instrument, setting up the autosampler table, defining the method, checking and defining limit and QC check tables, and loading samples and standards into their correct positions in the autosampler tray, analysis can begin.

7.1.1 To begin analysis first select **Operation** from the main menu, then select **Analysis**.

7.1.2 The software will prompt what method to use. **ICP95** is currently the method of choice for analyses under SW-846 Method 6010A protocol. If it is not already the default answer for the prompt type in **ICP95** and press ENTER.

7.1.3 Press F9 to run an analysis using a developed autosampler table.

7.1.4 The software will display the autosampler table specified in Section 6.4.4; if it is correct, press ENTER.

7.1.5 The software then displays the first solution to be analyzed. For most cases this will be a blank. This can be changed by using the arrow keys.

7.1.6 Pour at least 5 mL of each sample into a sample tube. Using the printout obtained from development of the autosampler table as a guide (see Section 6.6), place the sample tubes into the correct positions.

7.1.7 Press F1 to begin the analysis. The analysis may be halted when the autosampler is running a sample (not rinsing) by pressing F1 then ENTER twice. Do not stop the system while rinsing, as it can cause the computer software to lock up. The software then displays the same screen as in Section 6.5.6. Repeat procedure from this section to restart analysis.

7.2 Calibration Standards

7.2.1 Section 7.3 of SW-846, Method 6010A states "Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures . . . The calibration curve should consist of a blank and three standards." This statement is contradictory if the "manufacturer's recommended procedures" is a two point calibration, (i.e. a blank and one standard), as is the case with the TJA Enviro 36 ICP. It has been experimentally verified that the procedures recommended by the manufacturer, when followed correctly, produce more accurate data in a more efficient manner than that obtained using a three point calibration curve, especially for data at or near the detection limit. Therefore the manufacturer's recommendations for calibration have been adopted by this SOP. The EPA has confirmed that this procedure is compliant with the method.

7.2.2 Validation of the entire calibration range is accomplished using check standards at low medium and high levels during the analytical run.

7.3 Samples

7.3.1 All samples and quality control checks are analyzed in the same manner. No special consideration which would bias a result is provided to any analysis (such as longer or shorter rinse times). Duplicate readings are achieved for each sample and results averaged.

7.3.2 Sample results must fall within the established linear range. This includes analytes which may not be required for reporting by the client, but whose presence may influence correction factors or target analyte behavior.

7.3.2 Some samples may require dilution due to their viscosity and ability to be handled by the delivery system of the instrument. When performing dilutions use caution to achieve the least dilution possible without affecting the validity of the results or operation of the system.

7.3.3 If a sample is causing contamination problems due to high levels of analyte, the system may be interrupted and allowed to rinse prior to analysis of another sample. The efficiency of the rinse must be verified by running a blank as a sample before continuing analysis. The blank must meet the same acceptance criteria as the CCB.

8.0 CALCULATIONS

8.1 Soil

$$\text{Sample Result (mg/Kg)} = \text{instrument result (mg/L)} \times \text{instrument dilution} \times \frac{\text{final prep vol (mL)}}{\text{sample wt (g)}}$$

Dry weight basis (mg/Kg) = Sample Result (mg/Kg) / Sample % solids

8.2 Water

$$\text{Sample Result (mg/L)} = \text{instrument result (mg/L)} \times \text{instrument dilution} \times \frac{\text{final prep vol (mL)}}{\text{initial prep vol (mL)}}$$

9.0 QUALITY CONTROL

9.1 Before beginning sample analysis several check standards and other quality control samples must be analyzed.

9.1.1 First, analyze HSTD95 (see Section 5.3.9) as a sample. Results must agree within ±5% of the known values. If this control limit is not met, determine and correct the cause of the problem and reanalyze the standard. Recalibration may be necessary.

9.1.2 Analyze the Initial Calibration Verification Standard(s), ICV, after the highest calibration standard has been reanalyzed. This standard is used to validate the source and correct procedure used for preparation of the calibration standards, and the accuracy of the calibration curve. Results must fall within ± 10% of the true value. If this control limit is not met, determine and correct the cause of the problem and reanalyze the standard. Recalibration may be necessary.

9.1.3 Analyze the Initial Calibration Blank Standard, ICB, after the ICV. This standard is used to verify that carryover is not occurring, and that results near the detection limit are not biased by the slope and intercept of the calibration curve. The absolute value for the results of the ICB shall not be greater than the reported EQL. If this control limit is not met, determine and correct the cause of the problem and reanalyze both the ICV and ICB standards. Recalibration may be necessary. ICB results greater than the EQL may be accepted if they are no higher than 5% of the lowest concentration in the samples.

9.1.4 Analyze the Low-Level Check Standard, CRI, after the ICB. This standard is used to verify the accuracy of results near the detection limit. Results of the CRI must agree within ±20% of the true value. Although this is not a method requirement, this criterion has been adopted to validate the use of the calibration curve described above. If this control limit is not met, determine and correct the cause of the problem, reanalyze the CRI, and all check standards preceding it in their appropriate order. Recalibration may be necessary.

9.1.5 Analyze the Interference Check Standard, ICSA, after the CRI. This standard is used to verify the accuracy of the interferences, the accuracy of correction factors applied, and their effect on results in the absence of analyte. Results for the interfering elements (Al, Ca, Fe, and Mg) must agree within ±20% of the true value. Results for the analytes affected by the interferences, must not be greater than the absolute value of the reported EQL. If these control limits are not met, determine the cause and correct the problem. Reanalyze all check standards previously mentioned in their appropriate order. Recalibration may be necessary. The ICSA must be run at the beginning and end of the analytic run, or twice during each 8-hour shift, whichever is more frequent.

9.1.6 Analyze the Interference Check Standard with Analytes, ICSAB, after the ICSA. This standard is used to verify the accuracy of the interferences, the accuracy of interelement and background correction factors, and their effect on results in the presence of analyte. Results for the interfering elements and the analytes must agree within ± 20% of their true values. If these control limits are not met, determine the cause and correct the problem. Reanalyze all check standards previously mentioned in their appropriate order. Recalibration may be necessary. The ICSAB must be run at the beginning and end of the analytic run, or twice during each 8-hour shift, whichever is more frequent.

9.1.7 Although not a method requirement, it is prudent to "close out" the sequence of check standards that are run just after the calibration with a Continuing Calibration Verification Standard (CCV) and a Continuing Calibration Blank (CCB). Control limit criteria for these standards is the same as that for the ICV and ICB. The CCV and CCB must then be analyzed after every ten samples (Preparation Blanks, LCS, MS/MSD, PS/PSD and other QC samples are counted as samples), and at the end of the analytical run.

9.1.8 A serial dilution of a sample within a batch must be performed if matrix interference is suspected. Dilution results must agree within ± 10% of the original values if the analyte concentrations are greater than a factor of 10 above the EQL after the dilution. Further dilution will be necessary if the criteria is not met.

9.1.9 A minimum of one method blank per sample batch must be prepared and analyzed to determine if contamination from the preparation procedures has occurred. The method blank is carried through the entire process as a sample. Results for the method blank must be less than the reported EQL. If this criteria is not met determine the cause and correct the problem. Repreparation of the blank, and the entire batch associated with it, may be required. Sample results may also be reported when the blank exceeds the EQL if the concentrations in the samples are at a level twenty times that of the EQL.

9.1.10 Analysis of a Laboratory Control Sample (LCS), described as a "quality control sample" in Section 5.8 of Method 6010A, must be performed for each batch. If possible, the LCS matrix should mimic that of the samples in the batch. This helps to validate the applicability of the method utilized for the matrix. Results of the LCS must agree within ± 20% of the theoretical value or within the confidence interval established by the manufacturer. If this criterion is not met reprepare and reanalyze the samples associated with the batch. In some cases repreparation is not possible when all of the sample is used during initial preparation. In these cases flag the data associated with the QC failure using a narrative form and/or a non-conformance report form.

9.1.11 Spiked replicate samples are to be analyzed at a minimum frequency of 5% (one set of replicates per twenty samples) or per analytical batch, whichever is more frequent.. Analyzing spiked replicate samples also fulfills the requirement for analysis of a replicate sample (SW-846 Method 6010A, Section 8.4). The spikes must be made prior to preparative procedures and then processed as samples.

These matrix spikes and matrix spike duplicates are identified by adding MS and MSD, or S and SD to the ends of sample IDs. Spike levels are specific to the matrix and are covered in SOPs for the preparation methods used. Recovery and reproducibility must be determined using results of these samples. The MS and MSD recoveries are to be within ±20% of the theoretical value, as determined using the following formula (note- all dilution factors must be corrected for prior to use in this formula):

$$\%REC = \frac{\text{(spike result - sample result)}}{\text{spike added}} \times 100$$

If the recovery is not within control limits a procedural error or matrix interference may be suspected. Presence of the interference and its effect on the analytic system employed must be determined. This is accomplished by diluting and reanalyzing the replicate spikes and samples until control limits are met, or by performing a post-digestion spike.

The reproducibility, expressed as relative percent difference (RPD), is to be within 20% for sample values greater than ten times the IDL. The following formula is to be used to calculate RPD:

$$\text{RPD} = \text{Absolute Value of } \frac{(\text{first sample result - second sample result})}{\text{average of first and second results}} \times 100$$

If the reproducibility is not within control limits a procedural error or matrix effect may be suspected. Analysis of a post digest spike and spike duplicate, and respective RPD values is required to determine the cause of the problem.

9.1.12 Post digest spikes are performed to determine if quality control failures from the MS/MSD analyses are due to matrix effects or to procedural problems, and if the effects are matrix related, what degree of bias is encountered. Post digest spikes and spike duplicate samples are identified with a PS/PSD or A/AD at the end of the sample ID. Post spike recovery and reproducibility are calculated in the same manner as that for matrix spikes. The post spike must recover within 75% to 125% of the theoretical value for analytes whose concentrations are less than 4x the spike level added. If this limit is not met, a matrix interference is suspected and results are flagged using a narrative with the report.

9.1.13 Instrument Detection Limits (IDL) must be determined quarterly and whenever a new element is added. IDLs are determined by running seven replicates of a solution that is at a concentration 3 to 5 times the IDL, then multiplying the standard deviation of the seven replicates by 3. Average results from three, non-consecutive days are used for the final IDL value.

9.1.14 Method Detection Limits (MDL) must be determined annually and whenever a new element is added. The MDL must be determined for each matrix (i.e. aqueous and solid). The MDLs are experimentally determined by averaging results from analysis of seven replicate samples which have been spiked at a level approximating the EQL. Each matrix is spiked and prepared for analysis according to a defined method for that matrix.

9.1.15 The Estimated Quantitation Level (EQL) is a value derived from the MDL which is rounded and adjusted to reflect other laboratory constraints (e.g. background contamination). The EQL is typically one to ten times the MDL, but at no time may it be lower.

9.1.16 Instrument Linear Ranges (LR) must be determined quarterly and whenever a new element is added. The LR of an element is determined by analyzing the element at successively higher concentrations until self absorption or other effects cause the accuracy of results to fall outside a range of ±5 % of the true value. The result for the LR is then rounded to a whole number.

9.1.17 Prior to receipt, samples should be preserved to a pH of <2 with HNO_3. Samples are stored at 4 °C +/- 2 °C in plastic containers. The holding time for metals, except mercury, is 6 months.

10.0 INTERFERENCES

10.1 Spectral Interferences

Spectral interferences are caused by: (1) overlap of a spectral waveline from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or recombinant phenomena; and, (4) stray light from the line emission of high concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element (i.e. applying interelement correction factors, IECs). Unresolved overlap requires selection of alternate

wavelengths. Background and stray light can be corrected using background correction (BGC) adjacent to the analytic line.

The wavelengths and background correction points utilized in this method have been carefully chosen after extensive experimental evaluation. The IDL, MDL, LR and IEC values are all affected by the choice of the BGC points. Therefore, if the wavelines and BGC points for each element are altered, new IDLs, MDLs, LRs and IECs must be determined.

10.1.1 Element-specific interference is expressed as analyte concentration equivalents (e.g., false analyte concentrations) arising from the interfering elements. The interference effects of each element on every other element must be experimentally determined under conditions which mimic normal operations. The values achieved are used to calculate IECs.

For example: If the effect of 1000 ppm of aluminum produces a false concentration for arsenic of 4.814 ppm the IEC factor is 0.004814 (4.814/1000).

Interference correction is applied during analysis by multiplying the IEC by the concentration of the interfering element in a sample and subtracting or adding the result to the analyte concentration. It is important to use the correct sign (+ or -) for the IEC as there may be additive or subtractive forces at work. If the effect of the interference is additive to analyte concentration, the sign used for the IEC in the ThermoSpec software is also positive. If the effect of the interference is subtractive to analyte concentration, the sign used is negative.

10.2 Physical Interferences

Physical interferences are effects associated with the sample nebulization and transport processes. Differences in viscosity, volatility and surface tension between calibration standards and samples can cause inaccuracies. This is most apparent for organics and samples with high TDS levels or acid concentrations.

10.2.1 Physical interferences can be reduced by diluting the sample, using a peristaltic pump and by normalizing the data with an internal standard.

10.2.2 Salt buildup from samples with high TDS levels can clog the nebulizer tip and torch injector, affecting aerosol flow, and resulting in instrument drift. This problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. The nebulizer frequently requires cleaning to prevent salt buildup. This is recommended as a daily preventative maintenance task.

10.2.3 Temperature fluctuations in the environment cause differences in the density and surface tension of samples and standards resulting in inaccuracies and instrument drift. This problem is best controlled by maintaining a stable temperature in the room, but can be corrected for using an internal standard.

10.3 Chemical Interferences

Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects.

10.3.1 Molecular compound formation is controlled in part by utilizing preparation procedures that break down complex structures and stabilize dissolved elements. The ICP technique itself aids to minimize this phenomena.

10.3.2 Ionization effects can be overcome by adding high concentrations of alkali salts to force ion equilibrium to favor analytes of interest. Lithium carbonate is typically used for this.

10.3.3 Solute vaporization effects are controlled by matrix matching or by method of standard additions.

11.0 TROUBLESHOOTING

11.1 Printer Does Not Work

11.1.1 Not plugged in

11.1.2 Not turned on

11.1.3 Not connected to computer

11.1.4 Paper jammed or empty

11.2 Burn Will Not Commence

11.2.1 10 Amp fuse on power distribution panel blown

11.2.2 Double toggle on source not on

11.2.3 Double toggle on power distribution panel not on

11.2.4 Reset button not pressed

11.2.5 Unit not warmed up

11.2.6 Door to sample chamber has not been opened and closed since last burn

11.2.7 Source not functioning properly

11.2.8 1 Amp fuse on low-voltage power supply blown

11.2.9 Safety interlock switches not shut off

11.3 Poor Analytical Results

11.3.1 Instrument not profiled correctly

11.3.2 Sample and standards not matrix matched

11.3.3 Argon gas flows and pressures wrong

11.3.4 Leaks in argon system

11.3.5 Poor argon gas quality

11.3.6 Blockage, leaks or disconnections in delivery system tubing

11.3.6 Room temperature not stabilized (2 degrees per hour maximum change)

11.3.7 2 Amp high-voltage power supply fuse blown

11.3.8 Hg alignment lamp in optical path

11.3.9 Standards contaminated

11.3.9 Standards prepared incorrectly

11.3.10 Standards too old

11.3.11 Torch dirty

11.3.12 Pump tubing worn, deformed or contaminated

11.3.13 Peristaltic pump not turning smoothly

11.3.14 BGC points not appropriate

11.3.15 IEC factors not applied, or applied incorrectly

12.0 TABLE(S)

TABLE 1 - ESTIMATED QUANTITATION LIMITS

ELEMENT	EQL (ug/L)
Aluminum	100
Antimony	100
Arsenic	200
Barium	4.0
Beryllium	2.0
Boron	50
Cadmium	5.0
Calcium	200
Chromium	10
Cobalt	10
Copper	20
Iron	100
Lead	50
Lithium	20
Magnesium	100
Manganese	5.0
Molybdenum	10
Nickel	20
Potassium	100
Selenium	100
Silicon	500
Strontium	20
Silver	10
Sodium	500
Thallium	400
Tin	100
Titanium	50
Vanadium	10
Zinc	20
Zirconium	30

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Mercury Analysis by CVAA , EPA SW-846 Methods 7470A and 7471A			
Prepared by:	Approved by:	Date: 10/21/96	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to describe the analysis of mercury in samples that have been appropriately prepared. Although methods 7470A and 7471A were written for manual determinations, it must be clarified that the following SOP is for automated analysis.

2.0 SCOPE AND APPLICATION

Analysis is accomplished using Cold Vapor Atomic Absorption with a Leeman Labs PS200 Automated Mercury Analyzer. The procedure originates from SW-846 Methods 7470A and 7471A. Method 7470A may be used for analysis of mercury concentrations in mobility procedure extracts (e.g., TCLP, EP TOX and STLC solutions), aqueous wastes, and ground waters. Method 7471A may be used for soils, sediments, bottom deposits, sludges and various other solid, semi-solid or non-aqueous liquid matrices. Both methods are based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state, aerated from solution and passed through a cell positioned in the light path of the spectrometer. Absorbance (peak height) is measured as a function of mercury concentration.

Unlike standard AA spectrometers, the PS200 system is designed for optimal performance with mercury analysis. The system utilizes a high intensity, low pressure mercury vapor lamp, a dual beam photometer and solid state photo detectors and eliminates the need for a light-limiting monochromater. It has a wider dynamic range, greater sensitivity and quicker response time than typical AA systems. A system schematic is provided (see Section 11.0, Figure 1.)

The estimated quantitation limit (EQL) is 0.2 ug/L for analyte in solution. A typical digestion ratio of 0.5 g/200 mL for solid samples yields an EQL of 0.08 mg/Kg. Other dilution ratios must be applied if used.

3.0 REFERENCES/ASSOCIATED SOPs

USEPA SW-846, Third Edition, Methods 7470A and 7471A

USEPA Methods for Chemical Analysis of Waters and Wastes, Methods 245.1, 245.2 and 245.5

SOP MT-011, Mercury Analysis By CVAA, EPA Method 245.2

4.0 EQUIPMENT/REAGENTS/STANDARDS

4.1 Apparatus

4.1.1 PS200 Automated Mercury Analyzer with autosampler tray, computer, video monitor, keyboard and printer

4.1.2 Magnesium perchlorate drying tube

4.1.3 Color coded pump tubing (black, blue, red, orange and gray) and pump cassette

4.1.4 Teflon tubing (1/16")

4.1.5 Glass test tubes (16 x 100 mm)

4.1.6 One liter reagent bottles with access holes in lids

4.1.7 Auto pipettes, 1 - 10 mL and 1 - 1000 uL

4.1.8 Ventilation hood with minimum flow of 100 cfm

4.1.9 Quartz wool

4.2 Reagents, Standards and Solutions

4.2.1 ASTM Type I water otherwise known as reagent water

4.2.2 Nitrogen, (currently using gaseous bleed from liquid N_2 tank)

4.2.3 Concentrated nitric acid

4.2.4 50% nitric acid solution - 500 mL HNO_3 added slowly to 500 mL H_2O

4.2.5 10% hydrochloric acid solution - 100 mL HCl diluted to 1 L H_2O

4.2.6 0.5 N sulfuric acid solution - 14.0 mL H_2SO_4 diluted to 1 L H_2O

4.2.7 10% stannous sulfate solution - Dissolve 100 g ACS reagent grade stannous sulfate in 1 L 0.5N H_2SO_4 (stannous chloride may also be used, but the sulfate is less expensive)

4.2.8 Magnesium perchlorate solid, ACS reagent grade (Coarse size)

4.2.9 Mercury stock standard #1, Solutions Plus 1,000 ug/L from mercury metal

4.2.10 Mercury stock standard #2, Solutions Plus 1,000 ug/L from mercury oxide

4.2.11 Calibration Standards, LCS, MS/MSD and ICV preparations are described in SOP IP-001 and IP-002 (mercury preparation procedures)

5.0 PROCEDURE

5.1 Instrument Set-Up

5.1.1 The hardware should be set up already. If not, refer to the manufacturers manual.

5.1.2 Plumbing - It is critical to make plumbing connections correctly or the system will not function properly.

The PS200 comes with one drain tube and four color-coded pump tubes, two for drainage (black or gray), one for sample (blue or gray), and one for reductant (red or orange).

One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic stop is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and lock the clamp, lever up.

Insert the tab end of the drain tubing into the twist lock fitting (black) on the instrument chemical panel and connect the other end to the waste line supplied. Place the other end of the waste line into a waste container.

Connect the pump tubes to the PS200 as follows:

1. Reductant (red) - Insert the twist lock end of the Teflon tubing assembly into the reductant bottle fitting, and the other end of the Teflon tubing assembly into the tab end of the reductant pump tube. Insert the lower tee fitting tubing into the non-tab end of the reductant pump tube.

2. Sample (blue) - Insert the autosampler tip tubing into the tab end of the sample pump tube. Insert the upper tee fitting tubing into the non-tab end of the sample pump tube.

3. Drain (black) - Lock the tab end of the drain tubing twist lock into the chemical panel bulkhead fitting (black). Connect the non-tab end of the drain tubing to the long waste tubing. Place the end of the waste tubing into the 5 gallon waste jug under the bench top.

5.1.3 Packing and Installing the Drying Tube

Plug the glass drying tube on one end with quartz wool. Loosely pack magnesium perchlorate into the tube and plug the other end with quartz wool. It is important to use a coarse particle size and not to pack too tightly, to allow free flow of mercury vapor through the tube.

Loosen each nut and install the tube into the fittings in the chemical panel of the PS200. Tighten the fittings with your fingers enough to make a gas tight seal.

5.1.4 Preparing the Sample Tray

Snap the sample tray into the left side of the system. Make sure that the two screws on the sample tray fit snugly with the PS200 unit. Fill the rinse tray with 10% HCl and slide it into the sample tray. Fill the reductant bottle with 10% stannous sulfate (or stannous chloride) solution and place it on the tray next to the pump head. Connect the reductant tubing.

5.1.5 Powering Up the System

Press the green button on the lower right front of the unit to turn on the PS200, and press the blue button to turn on the mercury lamp. Press the POWER ON button on the printer, monitor and computer. When the system prompt appears on the monitor, type PS and press <enter>. The system will move the autosampler tip into the rinse tank and the PS200 menu will appear on the monitor.

Press the F2 macro key on the keyboard, type COLD START and press <enter>. This begins the cold start procedure, which warms up the instrument in preparation for initial systems test. The cold start procedure takes about two hours. Do not use the system before the procedure is

complete. The initial systems test is described in detail in the instrument manual. It describes techniques to determine if reagent bottles are leaching mercury, how to prepare standards, loading sample cups, setting the optics and running a systems test. These items should already be covered and the system in a standby mode if it has not been previously turned off.

5.1.6 Routine Operation

The system should be in an "overnight" mode. If it has been completely shut down proceed with the COLD START macro described in Section 5.1.5 . The WARM START macro is used to prepare the PS200 for operation after being started up from an overnight shutdown.

To run the WARM START macro press the <macro>F2 key on the keyboard. Type WARMSTART and press <enter>. The system will wait for several minutes and then turn on the pump and the gas flow to protocol speed. When the system is stable, a beep sound and a "system ready" message will appear on the screen.

After the system is ready, return to the main menu. Press <U> to go to the UTILITY column. Type <G> to enter the DIAGNOSTICS catalog. Check the aperture test by moving the highlight to OPT TEST and press <enter>. The acceptance value should be within ± 100. Move the highlight to OPT TEST and press <enter>. The first two digits of OPT TEST should be equal.

To run samples, you must enter all information regarding protocol, sample ID's, calibration values, and autosampler parameters into the software. This information is entered into a series of screens which are accessed from the main menu. The main menu is accessed by pressing <menu>F1.

First a protocol name must be selected. From the main menu, select PROTOCOL (type <P>) and then select GET (type <G>). The protocol screen will appear with a list of protocols on the disk. Type the name of the protocol and press <enter>. Press <menu>F1 to return to the main menu. The protocol name for the methods governed by this SOP is SW-846.

Once a protocol is selected a folder must be opened or created to hold data generated by the protocol selected. The folder is a place to store data on disk and is created to make reporting batches easier. The folder contains data for standards, samples, scans, updates and batch markers for a single protocol. Whenever a new protocol is created you must also create a new folder.

To create a new folder, from the main menu select DATA (type <D>), and then select OPEN (type <O>). An "Enter folder name" message will appear at the bottom of the screen. Type a folder name (new or existing) and press <enter>. IF YOU DO NOT OPEN A FOLDER, NO DATA WILL BE STORED ON DISK. Data cannot be transferred from one folder to another. Each folder can only store data for the protocol it was created under; however, many folders can be created for a single protocol. Folder names are assigned using the date, mmddyy. Different runs run during that day are assigned different sequence or batch numbers.

Verify all entered values. This is done by selecting PROTOCOL (type <P>), then selecting VALUES (type <V>). For normal operation set the following values:

UPTAKE TIME - 10
WEIGHT - N
DILUTION - N

Enter values for on/offs, times, and gains. This is done by selecting PROTOCOL <P>, then selecting ON/OFFS, TIMES, GAINS (type <O>). To change the integration time press <T> and

enter the time desired (1 to 30 seconds). Normally integration is set at 10 seconds. Increasing the integration time can help to increase precision if required.

Enter concentrations for calibration standards by selecting CALIBRATION (type <C>), STANDARDS (type <S>), and then UNITS (type <U>). Enter the units to be used (e.g., ug/L) and press <enter>. Using the hot key , select each standard on the screen (S1-S6) and enter the appropriate concentration. For normal operation the following concentrations are used:

S1 = .000 ug/L
S2 = .200 ug/L
S3 = .500 ug/L
S4 = 1.00 ug/L
S5 = 5.00 ug/L
S6 = 10.0 ug/L

Reset the calibration intensity data. From the main menu select CALIBRATION (type <C>), RESET (type <R>), and NEW (type <N>). The reset screen appears and a "reset all new calibration intensity data" prompt appears at the bottom of the screen. Erase calibration data that may have already been done with the protocol by typing <Y> and press <enter>. An "all data reset" message will appear. If new calibration intensity data is not desired Press <N> instead of <Y>.

Set the autosampler rinse time. From the main menu select AUTOSAMPLER <A>, SET UP <S>, and RINSE TIME <R>. The set up screen appears and an "enter rinse time" message is displayed. Type the desired rinse time in, (50 seconds) and press <enter>.

5.1.7 Rack Set Up

From the main menu select AUTOSAMPLER <A> and then RACK ENTRY <R>. The rack screen appears and an "enter rack name" message is displayed. Type a rack name and press <enter>. Rack names are assigned in the same manner as folders, i.e., by month, day and year (mmddyy), followed by a sequential alphabetic letter a,b,c . . . . The rack entry screen also allows designation of sample IDs and assigned cup positions.

The system can run two complete racks unattended. To use both, each must be defined. To define another rack first EXIT (type <E>) and repeat the procedure above.

The set up for standards, samples, and internal QC for SW-846 method criteria is provided in Section 11.0, Figure 2.

5.1.8 Autosampler Set Up

After loading the autosampler tray with standards, samples and QC in the correct positions the first and last cups to be sampled must be defined. From the main menu select AUTOSAMPLER and then SET-UP <S>. Type the rack number to be run (1 or 2), then the rack name and press <enter>. A "begin cup" prompt is displayed. Enter the number of the first cup to be sampled and press <enter>. An "end cup" prompt will appear. Enter the number of the last cup to be sampled and press <enter>. Repeat to make entries for the second rack.

5.2 Calibration Procedure

Fill the Calibration standard cups with standards (see Section 5.1.6). Press the yellow, <menu> and return to the main menu. Type <C> to go to the CALIBRATION column. In order to erase any old calibration

data, go to CALIBRATION RESETS and type <yes> for all four reset options. Once the resets are complete press the <macro> key and type <CAL 245> then press <enter>. This will start the calibration routine. The calibration data will be stored automatically. To review the calibration results after they have been stored select CALIBRATION LINE under the CALIBRATION column. If acceptable, type <A> to keep the curve generated and press the <F3> key to print the calibration graph.

5.3 Validation of the Calibration Curve

The calibration curve must be linear. This is established by verifying that the correlation coefficient is 0.995 or greater.

After running the standard curve and checking linearity, verify the validity of the curve using an initial calibration verification standard (ICV) and an initial calibration blank (ICB). The ICV must be from a source different from that used for the curve. It may be from the same vendor, but must be from a different lot. The ICV must read within 10% of the true value. The ICB must be less than the established EQL. If this criteria is not met, correct the problem, recalibrate and reanalyze the check standards. Since the calibration standards and ICV are prepared as samples it may mean that re-digestion will be necessary.

6.0 ANALYSIS

6.1 From the main menu select AUTOSAMPLE and then type <S> to go to the set up screen. Type <K> to set up the frequency of QC check standards. The frequency is established in Section 8.1 . Type <N> to label the names of the QC check standards. Load the standards and samples as described in Section 5.1.6 Press the blue <SAMPLE> button to start analysis.

6.2 Sample results must fall within 10% of the highest standard of the calibration curve. If the concentrations are higher than this the samples must be diluted and reanalyzed.

6.3 Analysis is generally performed in duplicate for standards and samples. This helps to validate high results and system performance. However, it is not a requirement of the method. Some circumstances may dictate that single readings be performed.

7.0 CALCULATIONS

7.1 Mercury results are expressed by the instrument in ug/L. Conversion to units appropriate for the end-user must be made by the analyst:

7.1.1 Calculate the mercury concentration for liquids as follows:

$$\text{Hg (mg/L)} = \frac{\text{ug/L}}{1000} \text{ divided by digestion or dilution ratio}$$

7.1.2 Calculate mercury concentration for solids using the following equation:

$$\text{Hg (mg/Kg)} = \frac{\text{(instrument result) (any dilution factor) (final prep. volume)}}{\text{initial sample weight}}$$

Dry weight basis (mg/Kg) = Sample Result (mg/Kg) / Sample % solids

An example:

digest ratio = 0.5 g sample / 200 mL final volume

instrument dilution = 1 mL /10 mL

instrument result = 3.0 ug/L

$$\text{Hg (mg/kg)} = \frac{(3.0\ \text{ug/L})\ (10)\ (0.200\ \text{L})}{0.5\ \text{g}} = 12\ \text{ug/g or mg/Kg}$$

8.0 QUALITY CONTROL

8.1.1 After calibration verify the curve with a standard from a source other than that used for the calibration. The initial calibration check standard (ICV) should be run at a concentration of between 1 to 5 ug/L. Results for the ICV must agree within 90 - 110% of the true value.

8.1.2 After the ICV, verify that carryover is not occurring with an initial calibration blank (ICB). The ICB should be a blank prepared with the same reagents as that of the calibration standards. Results of the ICB must be less than the established EQL.

8.1.3 Analyze a continuing calibration verification standard (CCV) and continuing calibration blank (CCB) after every ten (10) samples (PB, LCS, MS/MSD and PS/PSD solutions, and dilutions are considered samples and must be counted). The CCV should be run at a concentration between 1 to 5 ug/L. Results for the CCV must agree within 80 - 120% of the true value. Results of the CCB must be less than the established EQL.

8.1.4 A preparation method blank (PB) and laboratory control standard (LCS) must accompany each batch. The matrix used for the PB and LCS should mimic that of the samples. *Recovery of aqueous LCS results must fall within 20% of the true value. Recovery of solid LCS results must fall within the manufacturers confidence interval. The PB must be less than the EQL. If these criteria are not met, re-prepare the batch. (In some cases re-preparation is not possible if all of the sample was used. In this case, analyze the batch and qualify the data with a nonconformance report.)*

8.1.5 A matrix spike/matrix spike duplicate pair is analyzed every batch of samples. *If results are outside (80-120)%, perform a post spike. If the post spike is outside of (80-120)%, flag the data as having matrix interferences.*

8.1.6 Before use, all glassware should be soaked in 1:1 HNO_3 for a minimum of 30 minutes, then rinsed twice with reagent water.

8.1.7 Prior to receipt, samples should be preserved to a pH of <2 with HNO_3. Samples are stored at 4 °C +/- 2 °C in plastic containers. The holding time for mercury is 28 days.

8.1.8 Analysis is performed in duplicate for all standards and samples. The relative percent difference between duplicate readings must be less than 20%. Re-analysis of samples with RPDs of greater than 20% is necessary. If on the second attempt, the RPD is still greater than 20%, perform a dilution to alleviate possible matrix interferences. If after the dilution, the RPD is still greater than 20%, report the value and flag the results as a matrix problem.

9.0 INTERFERENCES

9.1 Moisture interferes with the quantitation of mercury at the 253.7 nm waveline. Avoid this interference by changing the dessicant in the drying tube frequently. (Once a day is recommended.)

9.1.2 Sulfide interferes at concentrations higher than 20 mg/L. Concentrations of sulfide, as sodium sulfide, do not interfere at lower concentrations when potassium permanganate is added.

9.1.3 Copper has been reported as an interference at concentrations greater than 10 mg/L.

9.1.4 Free Chlorine interferes at the 253.7 nm waveline. Chlorine is a byproduct of the reduction reaction when hydroxylamine hydrochloride is added. The gas evolved should be purged from the sample digestion bottles prior to analysis. Other sources containing high amounts of chlorine and chloride may also contribute to this interference, including sea water and brines.

9.1.5 Volatile organic compounds that absorb at this waveline can also be an interference.

10.0 TROUBLESHOOTING

10.1 Results Problems

10.1.1 Sample results are zero or all low

1. Check the optical cell for obstructions and clean if necessary.
2. Check that the reductant pump tubing lines are not blocked or worn and replace if necessary.
3. Check that the calibration curve has been stored into memory, if not rerun standards and 'accept' the curve.
4. Check that gas is flowing by observing bubbles in the gas separator.
5. Check that the mercury lamp is on, replace if necessary.

10.1.2 Standard response is low

Check for all of the problems described in Section 10.1.1.

10.1.3 Instrument detection limits are too high

1. Check that the pump tubing is conditioned. For best results pump water through the tubing for one hour prior to performing DL studies.
2. Allow the instrument to warm up completely (2 hours for the cell and 45 min. for the lamp.)
3. Check that the lamp has not deteriorated and replace if necessary.
4. Check that all of the reagents and standards have been freshly prepared.
5. Check that gas flow is set properly (0.25L/min.)
6. Check that the pump flow rate is set properly (5 mL/min.)

10.1.4 ICV results consistently low or high

1. Check that the primary and secondary sources are accurate, not expired and prepared fresh.
2. Check that digestion procedures are adequate and performed correctly.

10.1.5 Sample results low

1. Check that organic forms of mercury have been fully destroyed in the digestion procedure.
2. Check that dilution factors and calculations are applied correctly.

10.1.6 Sample results high

Check for moisture in the system, organics in the samples and possible contamination sources.

10.2 Pump Problems

10.2.1 Pump not turning

1. Check to ensure the tubing is clamped correctly inside the guidepath of the pump cassette.
2. Check that the tubing hasn't broken and spilled acids inside the pump. Clean if necessary.
3. Check that the pump power has been turned on.
4. The pump itself or the driver board may require repair. Consult a service technician.

10.2.2 Pump speed does not change

1. A bad encoder on the pump motor could be the cause. Replace the pump.
2. A bad pump motor cable could be the cause. Replace the cable.

10.3 System Problems

10.3.1 Power switch is not lit

1. Check the fuses and replace if necessary.
2. Check that the power switch is pushed in.
3. Check that the system is plugged in.

10.3.2 PS200 software state is "none"

1. Check that the RS232 serial cable is connected.
2. Check that the CPU is working properly. Exit from the PS Series software and power off the unit. Power up the system again, enter PS at the computer prompt and press <enter>.

10.3.3 Autosampler is off

Check that ALT-A has not been accidentally hit. Hit ALT-A keys until autosampler screen indicates autosampler is 'ON'.

10.4 Gas Problems

10.4.1 Gas is not bubbling or argon control malfunction is on screen

1. Check that incoming pressure is at least 60 psi.
2. Check the drying tube and make sure it is not blocked.

10.5 No fluid flow

10.5.1 Review same problems with pump (Section 10.2).

10.5.2 Teflon tubing is blocked

1. Check that the rinse tank contains diluted acid and replace blocked lines if any precipitant has formed.
2. Check the liquid/gas separator and clean if necessary.

11.0 FIGURES

FIGURE 1 - System Schematic

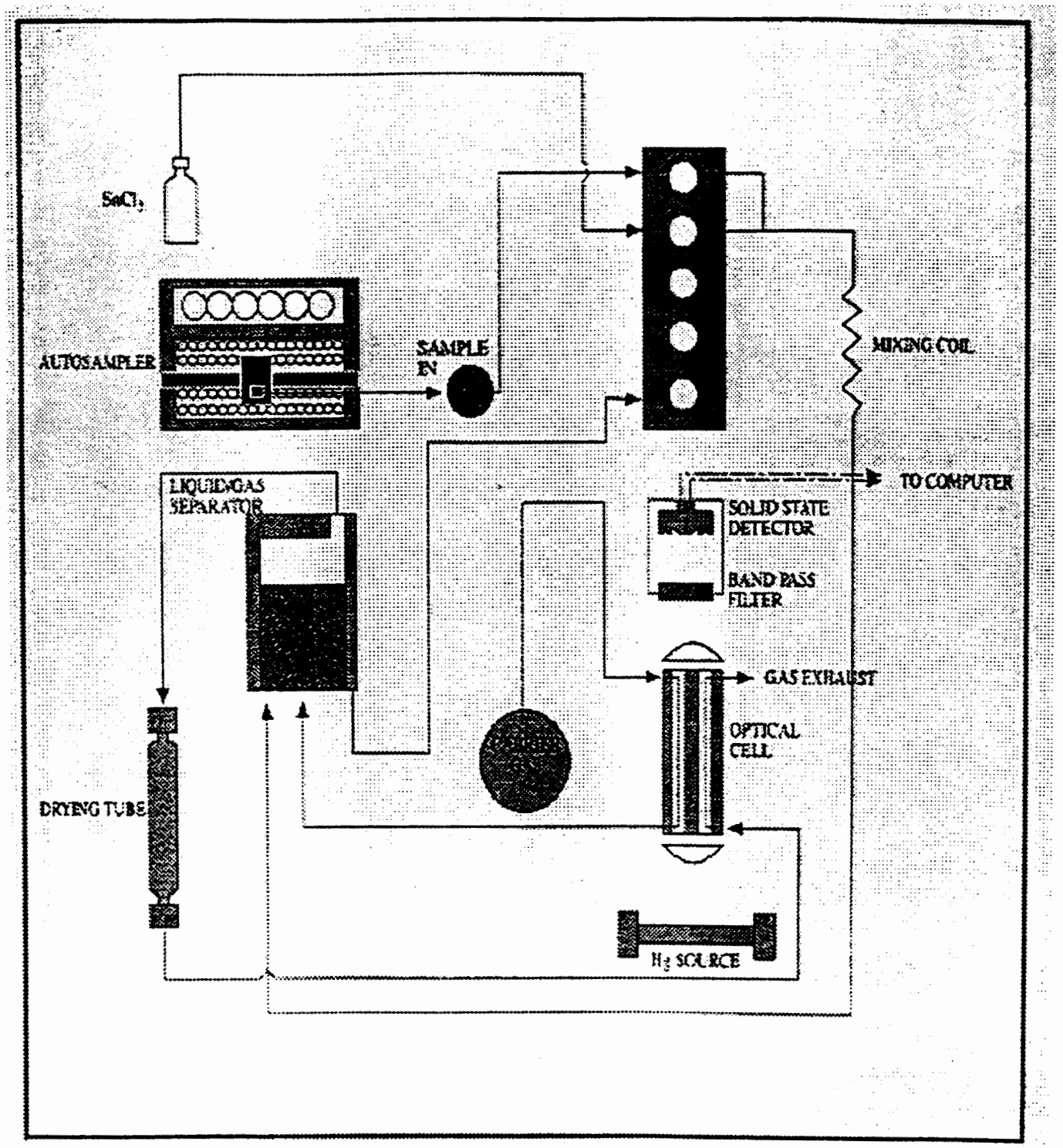
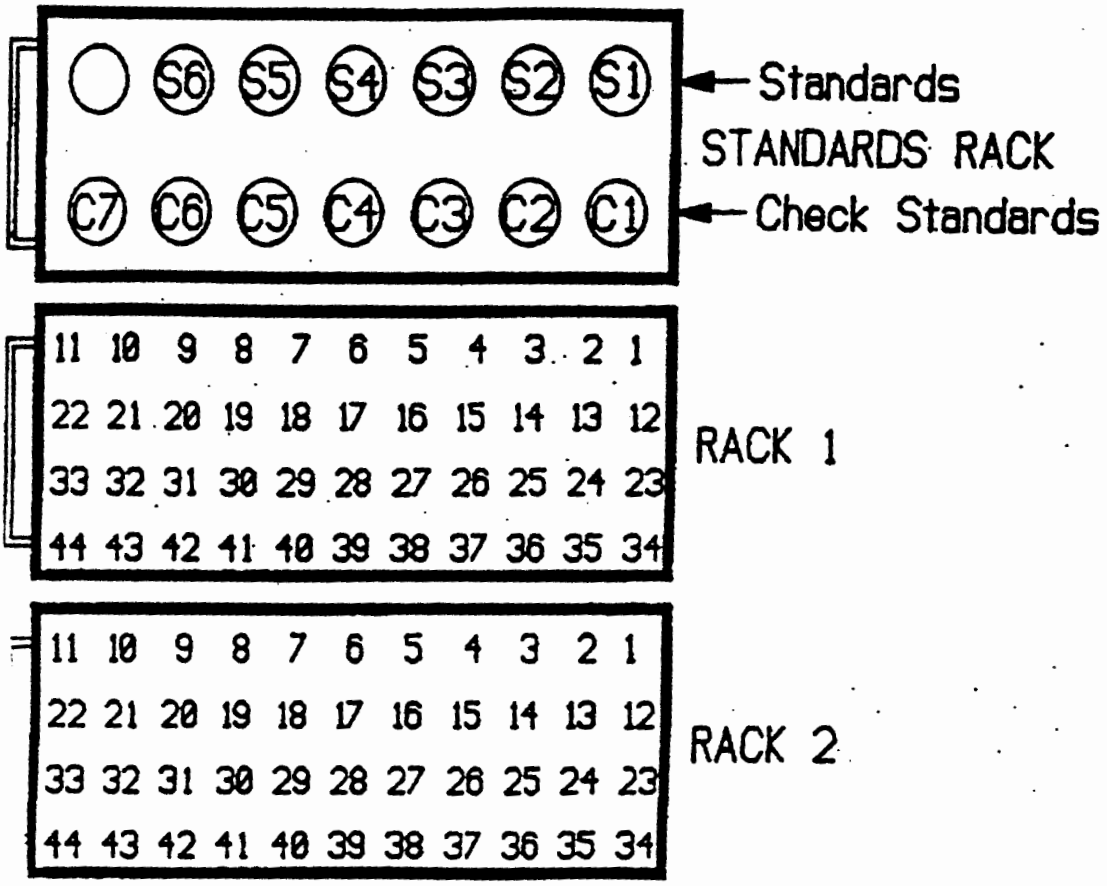


FIGURE 2 - Standard and Sample Rack



Autosampler layout

ROSS	SOP Standard Operating Procedure	SOP No: MT-005-00 Inital Date: 2/1/91 Page 1 of 4 Date Revised:

Title:

Metals Standards Documentation

Prepared by:	Approved by:	Date:	QC Approval:
Belinda Minter	CHCaldwell	5/23/91	CHC

1.0 PURPOSE

To describe the documentation of the preparation of metals standards.

2.0 APPLICATION

The SOP is intended to apply for all stock, intermediate stock, and working standards for use with ICP Emission, Graphite Furnace AA, Flame AA, and Cold Vapor AA.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 All stock standard solutions purchased from an outside source must have a Certificate of Analysis and Material Safety Data Sheet. The stock should also be labeled by Sample Control with an appropriate reagent control number.

5.2 Intermediate solutions are be recorded in the Standards Log book located in the desk area of the Inorganic Instrumentation Laboratory. Working standards for Flame AA or ICP must also be recorded, as must dilutions of independent check standards.

5.3 Figure 1 shows a blank sample entry. The name of the standard the analyst is making is entered beside STANDARD. STANDARD NUMBER references the log number for the book. The log number always starts with "II" (for Inorganic Instrumentation) and the entry order number in the book (i.e. II-001, II-002, etc.) The REAGENT NUMBER(S) is for the reagent control number(s) of the stock standard(s) used. Beside CONCENTRATION is the resulting concentration of the standard being made. Because ICP standards frequently have several elements only enter the dilution factor and the name of the stock standard used (i.e. 1:200 of RASI #1.) Enter analyst initials and date made beside PREPARED BY/DATE.

5.3.1 A Standards EXPIRATION DATE is determined by its concentration. For multielement standards the lowest concentration of any element is used to determine the expiration date. Listed below are the concentrations and periods that the standards are good for use.

Concentrations > 100 mg/l: six months
Concentrations > 10 mg/l and < 100 mg/l: three months
Concentrations > 1 mg/l and < 10 mg/l: one month
Concentrations < 1 mg/l made fresh for each days use

Under the METHOD OF PREPARATION, provide a brief description of the standard that was prepared and the matrix used.

5.4 Label the storage bottle the standard will be kept in with a ANALYTICAL STANDARD Label. A blank example label is shown in figure 2. Include the STANDARD name, CONCENTRATION, STANDARD NUMBER, DATE PREPARED, PREPARED BY initials, and EXPIRATION DATE. For Flame AA and ICP standards, write the matrix in the upper right hand corner of the label.

Figure 1

STANDARD_____ STANDARD NUMBER_____

REAGENT NUMBER(S)_____ CONCENTRATION_____

PREPARED BY/DATE_____ EXPIRATION DATE_____

METHOD OF PREPARATION_____
_____
_____
_____

SPECIAL INSTRUCTION:_____
_____
_____

Figure 2

ANALYTICAL STANDARD

STANDARD _____ CONCENTRATION _____

STANDARD NO. _____ DATE PREPARED _____

PREPARED BY _____ EXPIRATION DATE _____

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date

Terminated by	Date

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Aqueous Waste Disposal in the Metals Laboratory			
Prepared by:	Approved by:	Date: 7/18/95	QA Approval:

1.0 PURPOSE

To provide a safe, environmentally sound approach for handling, treatment and disposal of laboratory wastewater generated in the Metals Instrument Laboratory at Ross Analytical Services, Inc., (RASI).

2.0 APPLICATION

This standard operation procedure (SOP) is to be used to dispose of expired, aqueous standards, aqueous reagents, sample digestates and other aqueous wastes generated in the Metals Instrument Lab. This SOP has been prepared to provide direction for RASI employees for the disposal of these laboratory wastes in a safe and environmentally sound manner. This SOP is not intended for use in disposal of solid or extremely hazardous waste (Section 7.0, Definitions).

3.0 REFERENCES

All Metals Analysis SOPs

4.0 ASSOCIATED SOPs

SOP MT-012, Solid Waste Disposal in the Metals Laboratory

5.0 PROCEDURE

5.1 Reagents and Equipment

5.1.1 Apron

5.1.2 Faceshield

5.1.3 Safety glasses

5.1.4 Rubber gloves

5.1.5 Lab coat

5.1.6 Operating fume hood

5.1.7 Large funnel

5.1.8 Plastic spoon or scoopula

5.1.9 Mixing stick

5.1.10 Paper towels

5.1.11 Sodium hydroxide, granular

5.1.12 Hydrochloric acid (1:1)

5.1.13 pH Paper

5.1.14 Beaker tongs

5.1.15 Accumulation container(s) with top, 5 gallon

5.1.16 Plastic bucket, 5 gallon

5.1.17 Spill control equipment

5.2 Accumulation

5.2.1 Segregate hazardous aqueous waste from non-hazardous aqueous waste (Section 7.0, Definitions). Collect the wastestreams locally, at each area where they are produced, in containers that have sealable lids (glass or plastic, wide mouth jars are acceptable). Label the containers appropriately.

5.2.2 Put on safety glasses, gloves and a labcoat before handling aqueous wastes. Have an adequate supply of paper towels at hand and be aware of emergency spill kit locations.

5.2.3 Transfer the contents of the jars to 5 gallon, polyethylene containers when they are about 75% full. Transferring the contents before the jars are full helps to reduce spillage.

5.2.4 Transfer hazardous waste to containers with red bands and non-hazardous waste to containers with blue bands.

5.2.5 Fill the 5 gallon, polyethylene jugs to about 90% full. Again, this will help reduce spillage and aid in handling during neutralization procedures.

5.2.5 Accumulate no more than two hazardous (red banded), and two non-hazardous waste (blue banded) containers at a time. When the 5 gallon jugs are 90% full take them to the Wet Chemistry Lab and begin the neutralization procedure.

5.3 Neutralization

5.3.1 While still wearing gloves, safety glasses, and lab jacket, put on rubber apron and faceshield.

5.3.2 Place the 5 gallon accumulation jug in the fume hood.

5.3.3 Using pH paper, check the pH of the solution. (A pair of beaker tongs works well to hold the pH strip when dipping in the waste solution.)

5.3.4 Carefully add granular sodium hydroxide (NaOH) to the solution. Using the stirring rod, mix in the NaOH by gently swirling the solution. If the container becomes hot to the touch, discontinue adding the caustic until it has cooled. Periodically check the pH of the solution to see it has become neutral. (When the NaOH is added, the solution will change color. This occurs just before the pH becomes neutral. At this stage add very small amounts of caustic as it will cause large jumps in the pH.)

5.3.5 If the pH rises above 7.0 add more acidic waste or 1:1 nitric acid to the bucket to lower it again, and repeat step 5.3.4.

5.3.6 Allow the solution to cool somewhat and then recheck the pH.

5.3.7 Tightly seal the container and contact the SHE Department for transporting it to the waste containment building. If unable to transport to the waste containment building immediately, temporarily store the container in a safe, low-traffic area.

5.3.8 Thoroughly clean the work area when finished. Discard all contaminated solids, e.g., gloves and paper towels, in an appropriate manner (see SOP MT-012, Solid Waste Disposal in the Metals Laboratory).

NOTE: All laboratory employees are responsible for the disposal of laboratory wastes in accordance with the procedure established herein.

6.0 SAFETY

6.1 Always wear personal protection equipment as listed under Section 5.1, e.g., gloves, faceshield, apron, safety glasses, and lab jacket when performing pH adjustments.

6.2 Perform the neutralization in a well-ventilated fume hood.

6.3 This operation should only be performed when others are present in the same room.

6.4 Keep spill control equipment near this operation.

6.5 Waste containers must be kept closed except when adding or removing waste.

7.0 DEFINITIONS

7.1 Hazardous Aqueous Waste - This waste stream is identified with a red band on the satellite accumulation container. This waste is also defined as Metals Waste #2. It is an acidic, water-soluble waste which may contain high concentrations of metals (concentrations equal to or greater than RCRA MCLs) and/or small amounts of solvents generated by certain extraction procedures; e.g. chloroform ,acetone, MIBK or xylene.

7.2 Non-hazardous Aqueous Waste - This waste stream is identified with a blue band on the satellite container. Also defined as Metals Waste #1, is an acidic, water-soluble waste with low or no concentrations of heavy metals. All solutions, including standards, samples, rinsates, and digests, which have trace metal concentrations below RCRA MCLs may be accumulated. No solvents may be accumulated.

7.3 RCRA Hazardous Waste Characteristics for Metals -

Metal	Concentration
Arsenic	5.0 mg/L
Barium	100 mg/L
Cadmium	1.0 mg/L
Chromium	5.0 mg/L
Lead	5.0 mg/L
Mercury	0.2 mg/L
Selenium	1.0 mg/L
Silver	5.0 mg/L

7.4 Extremely Hazardous Waste - Poisons, acute toxins and explosives. At this time there are no compounds in the metals instrument laboratory with this description.

7.5 Solid Waste - Any material that has been contaminated from activities in the laboratory or during waste neutralization procedures that can not be cleaned or reused. This includes gloves, paper towels, pipet tips, weighing paper or boats, GAC columns and various other disposable items.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title:	Analysis of Metals Using the Thermo Jarrell Ash 61E Trace ICP by EPA SW-846 Method 6010A			
Prepared by:	Approved by:	Date:	QA Approval:	

1.0 PURPOSE

The purpose of this SOP is to describe the analysis of metals that have been appropriately prepared using the Thermo Jarrell Ash 61E Trace ICP.

2.0 SCOPE AND APPLICATION

This SOP applies to all ICP emission analyses performed using the Thermo Jarrell Ash 61E Trace ICP. The method described includes calibration and quality control criteria defined by EPA SW-846, Method 6010A.

The method may be used for the following matrices: groundwaters, aqueous samples, industrial and organic wastes, soils, sludges, sediments, biologic substances, and other solid or semisolid wastes.

These matrices require digestion prior to analysis. The digestion procedures (e.g., SW-846, Methods 3005 - 3050) are not provided by this document but may be found in other SOPs. When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

The elements for which this method may be employed and their respective Estimated Quantitation Limits (EQLs) established by RASI for clean aqueous samples using pneumatic nebulization are provided in Section 12.0, Table 1. These EQLs have been experimentally verified with the system in use.

The TJA 61E Trace ICP performs simultaneous detection of elements. The method measures element emitted light by optical spectrometry. Samples and standards are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by an RF generated, inductively coupled plasma. The plasma torch is mounted horizontally. The spectra are dispersed by a grating spectrometer and intensities of the lines are monitored by photomultiplier tubes. Intensities are calibrated using known standard concentrations and used to quantify sample results.

3.0 REFERENCES

ICAP 61E Trace Analyzer Operator's Manual. Thermo Jarrell Ash Corporation, Franklin, MA, July, 1993

"Inductively Coupled Plasma-Atomic Emission Spectroscopy", Test Methods for Evaluating Solid Waste (SW-846), Method 6010A, Revision 1, July, 1992

4.0 ASSOCIATED SOPs

None

5.0 EQUIPMENT

5.1 Apparatus

5.1.1 Thermo Jarrell Ash 61E Trace ICP, with host computer (minimum 386), printer, coolant water recirculator, polychromator vacuum pump, autosampler, and ancillary gas connections

5.1.2 Class A volumetric flasks - 100, 200, 500, 1000 and 2000 mL

5.1.3 Class A volumetric pipets - 1, 2, 5, 10 and 20 mL

5.1.4 Auto pipettes, 2 - 10 mL, 1 - 5 mL, 100 - 1000 uL, and 10 - 100 uL and pipet tips

5.1.5 Plastic sample cups, 30 mL

5.1.6 Ventilation hood with minimum flow of 100 cfm

5.1.7 Peristaltic pump tubing (0.89 mm inside diameter - orange/orange)

5.1.8 Autosampler standard cups and sample test tubes (16 X 100 mm)

5.1.9 Polyethylene storage bottles

5.2 Reagents

5.2.1 Reagent water

5.2.2 Argon, gaseous bleed from liquid source (minimum 70 psi)

5.2.3 Nitric acid (HNO_3), concentrated

5.2.4 Hydrochloric acid (HCl), concentrated

5.2.5 Nitric acid (HNO_3), 50% - Add 500 mL concentrated acid to 400 mL reagent water then dilute to 1 L.

5.2.6 Hydrochloric acid (HCl), 50% - Add 500 mL concentrated acid to 400 mL reagent water then dilute to 1 L.

5.3 Standards

5.3.1 Calibration Blank Standard - 2% HNO_3 / 5% HCl

To a 2 L flask add approximately 1 L of reagent water. Add 40 mL concentrated HNO_3 and 100 mL concentrated HCl. Bring to volume with reagent water and mix well. Add 20 mL of 100 ppm Y (See Section 5.3.3 for preparation procedure) and 40 mL of stock Li standard (10,000 ppm) and mix well. Store in a polyethylene bottle.

5.3.2 Calibration Standards

5.3.2.1 Mixed Calibration Standards (STD1, STD2) - 2% HNO_3 / 5% HCl

STD1 (mixed standard containing Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Tl, Sn, Ti, V, Zn): To a 200 mL flask add approximately 100 mL reagent water. Add 2 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 2.0 mL of *Solutions Plus* or equivalent QC-27. QC-27 is a custom blend. Add 200 uL of stock single element Sn standard (1000 ppm). Bring to volume with reagent water and mix well. Add 1.0 mL 100 ppm Y (See Section 5.3.3 for preparation procedure) and 2.0 mL stock Li standard (10,000 ppm) and mix well. Store in a polyethylene bottle. Light reduces the stability of several elements in this solution. It is preferable to store the solution in the dark when not in use. When prepared as described above, the following concentrations will result:

CALIBRATION STD1

ELEMENT	CONCENTRATION (mg/L)
Antimony	1.0
Arsenic	1.0
Barium	1.0
Beryllium	1.0
Cadmium	1.0
Chromium	1.0
Cobalt	1.0
Copper	1.0
Lead	1.0
Manganese	1.0
Molybdenum	1.0
Nickel	1.0
Selenium	1.0
Silver	1.0
Thallium	1.0
Tin	1.0
Titanium	1.0
Vanadium	1.0
Zinc	1.0

STD2 (mixed standard containing Al, Ca, Fe, Mg): To a 200 mL flask, add approximately 100 mL reagent water. Add 2 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 10 mL stock single element Al standard (10,000 ppm), 10 mL stock single element Ca standard (10,000 ppm), 4.0 mL stock single element Fe standard (10,000 ppm), and 10 mL stock single element Mg standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 1.0 mL 100 ppm Y (See Section 5.3.3 for preparation procedure) and 2.0 mL stock Li standard (10,000 ppm) and mix well. Store in a polyethylene bottle. The following concentrations will result:

CALIBRATION STD2

ELEMENT	CONCENTRATION (mg/L)
Aluminum	500
Calcium	500
Iron	200
Magnesium	500

5.3.2.2 Single Element Calibration Standards (STD3, STD_C4) - 2% HNO_3 / 5% HCl

STD3 (Na): To a 200 mL flask, add approximately 100 mL reagent water. Add 2 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 2.0 mL stock single element Na standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 1.0 mL 100 ppm Y (See Section 5.3.3 for preparation procedure) and 2.0 mL stock Li standard (10,000 ppm) and mix well. Store in a polyethylene bottle. When prepared as described above, the following concentration will result:

CALIBRATION STD3

ELEMENT	CONCENTRATION (mg/L)
Sodium	100

STDC_4 (K): To a 200 mL flask, add approximately 100 mL reagent water. Add 2 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 1.0 mL stock single element K standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 1.0 mL 100 ppm Y (See Section 5.3.3 for preparation procedure) and 2.0 mL stock Li standard (10,000 ppm) and mix well. Store in a polyethylene bottle. When prepared as described above, the following concentration will result:

CALIBRATION STDC_4

ELEMENT	CONCENTRATION (mg/L)
Potassium	50.0

5.3.3 Internal Yttrium Standard - 2% HNO_3 / 5% HCl

To a 2 L flask add approximately 1 L reagent water. Add 40 mL concentrated HNO_3 and 100 mL concentrated HCl. Add 20.0 mL stock single element Y standard (10,000 ppm). Bring to volume with reagent water. Store in a polyethylene bottle.

5.3.4 Calibration Curve Standards

Note: A five point curve is prepared for potassium because it is prone to a high degree of interference.

STDC_1 (5.0 ppm K): To a 200 mL flask add approximately 100 mL reagent water. Add 4 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 100 uL stock single element K standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 2.0 mL 100 ppm Y standard (see Section 5.3.3) and 4.0 mL stock Li standard and mix well. Store in a polyethylene bottle.

STDC_2 (10.0 ppm K): To a 200 mL flask add approximately 100 mL reagent water. Add 4 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 200 uL stock single element K standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 2.0 mL 100 ppm Y standard (see Section 5.3.3) and 4.0 mL stock Li standard and mix well. Store in a polyethylene bottle.

STDC_3 (25.0 ppm K): To a 200 mL flask add approximately 100 mL reagent water. Add 4 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 500 uL stock single element K standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 2.0 mL 100 ppm Y standard (see Section 5.3.3) and 4.0 mL stock Li standard and mix well. Store in a polyethylene bottle.

STD_C4 (50.0 ppm K): For procedure, please see Section 5.3.2.2.

STDC_5 (100.0 ppm K): To a 200 mL flask add approximately 100 mL reagent water. Add 4 mL concentrated HNO_3 and 10 mL concentrated HCl. Add 2000 uL stock single element K standard (10,000 ppm). Bring to volume with reagent water and mix well. Add 2.0 mL 100 ppm Y standard (see Section 5.3.3) and 4.0 mL stock Li standard and mix well. Store in a polyethylene bottle.

CALIBRATION CURVE STANDARDS

Analyte	STDC_1	STDC_2	STDC_3	STDC_4	STDC_5
Potassium	5.0 ppm	10.0 ppm	25.0 ppm	50.0 ppm	100.0 ppm

5.3.5 Initial Calibration Verification Standard (ICV) - mixed standard in 2% HNO_3 / 5% HCl

To a 1000 mL flask add approximately 500 mL reagent water. Add 20 mL concentrated HNO_3 and 50 mL concentrated HCl . Add 5.0 mL *Solutions Plus* or equivalent QC-31. Bring to volume with reagent water and mix well. Add 10.0 mL 100 ppm Y standard (see Section 5.3.3) and 20.0 mL stock Li standard and mix well. Store in a polyethylene bottle. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use.

The *Solutions Plus* or equivalent standard is a custom mix. The source for the ICV must be different than that used for the calibration. The same vendor may be used as long as the source is different. Note that different lot numbers for solutions may still be from the same source. When prepared as described, the standard will contain the following elements and respective concentrations:

INITIAL CALIBRATION VERIFICATION STANDARD - ICV

ELEMENT	CONCENTRATION (mg/L)
Aluminum	12.5
Antimony	0.50
Arsenic	0.50
Barium	0.50
Beryllium	0.50
Cadmium	0.50
Calcium	12.5
Chromium	0.50
Cobalt	0.50
Copper	0.50
Iron	10.0
Lead	0.50
Magnesium	10.0
Manganese	0.50
Molybdenum	0.50
Nickel	0.50
Potassium	10.0
Selenium	0.50
Silver	0.50
Sodium	12.5
Thallium	0.50
Tin	0.50
Titanium	0.50
Vanadium	0.50
Zinc	0.50

5.3.6 Low Level Check Standard (CRI6010A) - mixed standard in 2% HNO_3 / 5% HCl

To a 100 mL flask add approximately 50 mL reagent water. Add 2 mL concentrated HNO_3 and 5 mL concentrated HCl. Add 100 uL *Solutions Plus* or equivalent CRDL-T stock solution. CRDL-T is a custom blend. Bring to volume with reagent water and mix well. Add 1.0 mL 100 ppm Y standard (see Section 5.3.3) and 2.0 mL stock Li standard and mix well. Store in a polyethylene bottle. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use. When made as described the following mixture of elements and respective concentrations (which should be 2 times the EQL) are achieved:

LOW-LEVEL CHECK STANDARD STOCK SOLUTION - CRI6010A

ELEMENT	CONCENTRATION (mg/L)
Aluminum	0.200
Antimony	0.010
Arsenic	0.010
Barium	0.004
Beryllium	0.002
Cadmium	0.002
Calcium	0.400
Chromium	0.020
Cobalt	0.010
Copper	0.010
Iron	0.100
Lead	0.010
Magnesium	0.100
Manganese	0.010
Molybdenum	0.010
Nickel	0.020
Potassium	0.400
Selenium	0.010
Silver	0.010
Sodium	1.000
Thallium	0.010
Tin	0.020
Titanium	0.010
Vanadium	0.020
Zinc	0.040

5.3.7 Interference Check Standard solution A (ICSA) - mixed element standard in 2% HNO_3 / 5% HCl

To a 1000 mL flask add approximately 500 mL reagent water. Add 20 mL concentrated HNO_3 and 50 mL concentrated HCl. Add 100 mL of *CPI* or equivalent INTFA solution. Bring to volume with reagent water and mix well. Add 10.0 mL 100 ppm Y standard (see Section 5.3.3) and 20.0 mL stock Li standard and mix well. Store in a polyethylene bottle. Listed below are the elements and concentrations for the stock solution of INTFA solution and the final concentration when diluted:

INTERFERENCE CHECK STANDARD A - ICSA

Elements	Stock Concentration (mg/L)	Final Concentration (mg/L)
Al, Ca, Mg	5000	500
Fe	2000	200

5.3.8 Interference Check Standard solution AB (ICSAB) - mixed standard in 2% HNO_3 / 5% HCl

To a 1000 mL flask add about 400 mL reagent water, 20 mL concentrated HNO_3 and 50 mL concentrated HCl . Add 100 mL INTFA solution (see Section 5.3.6), 10.0 mL *CPI* or equivalent ANALYTES B solution, 10.0 mL *CPI* or equivalent ALTERNATIVE ANALYTES B solution. The INTFA, ANALYTES B, and ALTERNATIVE ANALYTES B solutions are names of standard mixes provided by several manufacturers (e.g. *Plasmachem, CPI, Solutions Plus*). Add 500 uL stock single element K standard (10,000 ppm), 400 uL stock single element Na standard (10,000 ppm), 1.0 mL stock single element Sn standard (1000 ppm), and 1.0 mL stock single element Ti standard (1000 ppm). Bring to a final volume with reagent water and mix well. Add 10.0 mL 100 ppm Y standard (see Section 5.3.3) and 20.0 mL stock Li standard and mix well. Store in a polyethylene bottle. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use. The following element mix and respective concentrations results:

INTERFERENCE CHECK STANDARD WITH ANALYTES SOLUTION - ICSAB

ELEMENT	CONCENTRATION (mg/L)
Aluminum	500
Antimony	1.0
Arsenic	1.0
Barium	0.5
Beryllium	0.5
Cadmium	1.0
Calcium	500
Chromium	0.5
Cobalt	0.5
Copper	0.5
Iron	200
Lead	1.0
Magnesium	500
Manganese	0.5
Molybdenum	1.0
Nickel	1.0
Potassium	5.0
Selenium	1.0
Silver	1.0
Sodium	5.0
Thallium	1.0
Tin	1.0
Titanium	1.0
Vanadium	0.5
Zinc	1.0

5.3.9 Continuing Calibration Verification Standard (CCV) - mixed standard in 2% HNO_3 / 5% HCl

To a 1000 mL flask add approximately 500 mL reagent water. Add 20 mL concentrated HNO_3 and 50 mL concentrated HCl . Add 10 mL *Solutions Plus* or equivalent QC-31. Bring to volume with reagent water and mix well. Add 10.0 mL 100 ppm Y standard (see Section 5.3.3) and 20.0 mL stock Li standard and mix well. Light reduces the stability of several elements in solution. It is preferable to store the solution in the dark when not in use.

The CCV source may be the same as that of the calibration standards. When prepared as described, the standard will contain the following elements and respective concentrations:

CONTINUING CALIBRATION VERIFICATION STANDARD - CCV

ELEMENT	CONCENTRATION (mg/L)
Aluminum	25
Antimony	1.0
Arsenic	1.0
Barium	1.0
Beryllium	1.0
Cadmium	1.0
Calcium	25
Chromium	1.0
Cobalt	1.0
Copper	1.0
Iron	20
Lead	1.0
Magnesium	20
Manganese	1.0
Molybdenum	1.0
Nickel	1.0
Potassium	20
Selenium	1.0
Silver	1.0
Sodium	25
Thallium	1.0
Tin	1.0
Titanium	1.0
Vanadium	1.0
Zinc	1.0

6.0 PROCEDURE

6.1 Instrument Set-up

6.1.1 The hardware should already be set up. The instrument requires electrical power, coolant water, argon gas, a vacuum pump, and ventilation. It should be operated in a thermally stable environment at a temperature between 15 to 20°C .

6.1.2 After turning on the computer, the ThermoSPEC software should be booted automatically. If it did not, type in at the C:\> prompt CD\STATION. While in C:\>STATION directory, type STNRUN then press ENTER.

6.2 Instrument Ignition

6.2.1 Check the supply of Argon weekly. The tank is located outside. It is filled with liquid argon with gas to supply the ICP. A gauge displays the volume of argon remaining in the tank. When the gauge drops to 1/4 full level place an order for a refill. Under typical work load the tank will last several months between fillings.

6.2.2 The argon gas is regulated using a single stage regulator prior to connection to the ICP. The regulator pressure should be set to 60 psi. A second regulator is located within the torch chamber of the ICP. The pressure is regulated using the software. From the main pull down menu, choose **Development**, highlight **Methods,** press ENTER, then choose option F6 = PlasmaInfo. Under Gas Flow, the torch gas should be set at high flow, auxiliary gas flow should be set to low (0.5 L/minute), and nebulizer pressure should be 25 psi.

6.2.3 Check the sample and rinse station drain buoys to make sure that the end of each drain tube is immersed in at least eight inches of fluid. The buoys should be empty enough to allow for the drainage of the current run.

6.2.4 Place two peristaltic pump tubes (0.89 mm - orange/orange) into the white pump tubing cassettes and snap into place. Rotate the tension arm against the tubing. Rotate until the tension is taut and sample flow is steady. Optimum position for the tension arm is at approximately 5 o'clock. The upper tubing should have one end attached to the capillary tubing coming from the Autosampler and the other end attached to capillary tubing going to the torch nebulizer. The bottom tubing should have one end attached to the capillary tubing coming from the rinse beaker and the other end attached to the capillary tubing going to the rinse tube on the autosampler. Replace the tubing daily or more frequently if they look worn out or highly discolored. While looking at the front of the pump, the right side of the tubing holder should contain the ends that come from the autosampler probe and from the rinse beaker. The pump speed is regulated from the software. From the main pull down menu, choose **Development**, highlight **Methods,** press ENTER, then choose option F6 = PlasmaInfo. Under peristaltic pump parameters, analysis pump rate should be set at 110 rpm, flush pump rate should be set at 110 rpm, relaxation time should be 0 second, and pump tubing type should be Tygon-orange. Check that all flow directions are correct. The pump will not be operating at this point. The software will start the pump at the appropriate time.

6.2.6 Fill the rinse bottle with a 5% solution of nitric acid. Although it has been suggested to match the acid concentration of standards and samples, it has been found that excessive carryover occurs for several elements using this protocol. Place the autosampler probe into the rinse bottle.

6.2.7 Make sure that all vents are turned on.

6.2.8 The torch is controlled by the software. From the main pulldown menu, choose **Setup,** highlight **Plasma Control Panel**, press ENTER, then choose option F1 = Startup. To start the ignition procedure, you will now need to press F9 = Continue. The system will purge for 90 seconds and then the torch will automatically ignite. Even though the peristaltic pump will not be operating, you will need to keep the probe tip immersed in water.

6.3 Instrument Operation

6.3.1 Before starting any analyses, and after igniting the plasma, let the instrument thermally stabilize for at least 60 minutes.

6.3.2 Profile the instrument by aspirating a 5.0 ppm arsenic standard. NOTE: This standard needs to contain the same concentrations of Yttrium and Lithium that the calibration standards contain. Choose the **Setup** pulldown menu and then highlight **Profile** and press ENTER. Press F3 = automatic profile. Enter a flush time of 25 seconds and an integration time of 0.5 seconds. Aspirate the arsenic standard by placing the probe tip into the bottle. Press F1 = run to begin the automatic profile operation. When profiling is completed, check the peak position. If the resulting profile does not show a peak position of between ±0.5, unlock the dial and move the vernier 100 units in either direction; lock the dial and repeat the profile. If the peak position from the profile is between ±0.5, press F1 = Calculate SS. When the vernier position comes on the screen, press ENTER. The new vernier position will appear on the upper-right of the screen. Unlock the dial and move it to the new position; lock the dial and press ESC. Check the precision of the profile by repeating the above steps. When the peak position reads within ±0.1, accept the profile by pressing F9.

6.3.3 To perform a manual profile refer to the *ICAP 61E Trace Analyzer Operator's Manual,* Section 7 (Setup Menu) pages 6 - 8.

6.4 Developing an Autosampler Table

6.4.1 There are several useful function keys which may be used to edit the autosampler table. There are 3 different menus for the function keys. The first menu is always shown when either the Alt or Crtl key is pressed. The second menu is shown when the Alt key is held down. The third menu is shown when the Crtl key is held down. The beginning analyst should use only the main and Alt menu functions. Those two menus allow the editing of the autosampler table.

6.4.2 Choose the **Exit** pulldown menu.

6.4.3 Under the **Exit** menu, highlight **Asedit.exe** and press ENTER. Enter a name for the Autosampler Table. The default is the last one modified. Use the following guideline for naming the autosampler table: Numerical date followed by alphabetical sequence of runs performed that day (e.g., For a table analyzed on the second run of the day on April, 1 1996, the table should be labeled 040196_B.) After typing in a name, press ENTER.

6.4.5 Press F3 = Add set. The arrow keys will allow the analyst to move between the prompts. For the "Set Description", type ICP RUN and the date of analysis. For the "Method Name", type CLPNEW1. Under "Default Operator ID", enter your initials. The other fields except "Auto-Rinse Between Samples" and "Rinse Time" are not required but are available to help distinguish between runs for different clients or projects and used to aid in database searches. The "Auto-Rinse Between Samples" should be set to YES. Highlight the answer and press the space bar to

toggle between "YES" and "NO". Type in 60 for the amount of seconds the instrument rinses between samples. A 60 second rinse has been found to be adequate for most analyses. The analyst can increase this time if carryover is apparent but should not decrease the time as it is a method requirement.

6.4.6 The next prompt is "# Sample Positions needed". A rule of thumb here is to guess high. It is easier to delete unused sample positions than to add more. The default for the next prompt "Starting Rack Number" is 2, (Rack 1 is used for calibration and QC standards). The default for the prompt "Starting Position Number" is 1. Both of the above can be changed, if needed. Highlight the answer area for "Default Sample Name". Press the space bar once and this should delete the default "Sample-1". Highlight the answer area for the prompt "Auto Increment?". Press the space bar to toggle to "NO" or "YES" depending on how many sequential sample ID numbers are in the analysis set and the ease afforded the operator in building the table. The prompt "Default Limit Check Table Name" should be left as is. Unless all the samples have the same dilution factors, the prompt "Default Correction Factor" should be left at 1.

6.4.7 Press F1 to "Edit Samples". Press F5 to insert standards. The standards defined for the method chosen will automatically be inserted into the table. For more information about defining standards, see *ICAP 61E Trace Analyzer Operator's Manual,* Section 5 (Development Menu) pages 10 - 12. Use the arrow key to highlight the position below the last standard and press F3 to insert standard. Type in all of the standards preceded by "H" to indicate the method required readback. Use the arrow keys to go to the CheckTable column for the "HSTDs". Press SHIFT and the key with ?/ symbols simultaneously. This will bring up the options menu. Choose the appropriate QC check table and press enter. Enter all of the standard readbacks in this manner. After the standard readbacks have been entered, press F3 to insert QC and type ICV. Go to the CheckTable column and insert the appropriate check table in the same manner as the standard readbacks were entered. Next, use the arrow keys to highlight the sample under the ICV. Press F7 to insert a blank. Type ICB. Again, go to the CheckTable column, press SHIFT, and the key with ?/ symbols simultaneously. This will bring up the options menu. Choose the appropriate Blank check table and press ENTER. The default is CCB6010A. Follow this procedure to insert QC samples such as HSTDs, ICV< ICB< CCV< CCB<etc. The procedure is similar for other QC samples such as the CRI, ICSA, and the ICSAB. The only difference is that F3 (insert standard) and F7 (insert blank) are not employed. They are treated as samples within rack 2. The series of calibration and check standards should be as follows: Blank, STD2, STD1, STDC_4, STD3, HSTD2, HSTD1, HSTDC4, HSTD3, ICV, ICB, CRI6010A, ICSAI, ICSABI, CCV, and CCB.

6.4.8 Always start off with the preparation blank for a set of samples. Enter the complete preparation blank name as given by the sample preparation group with the addition of a W to indicate an aqueous preparation or an S to indicate a solid preparation (e.g., PBW1992 or PBS2003).

6.4.9 Enter the Laboratory Control Sample for a set of samples directly after the Preparation Blank. Follow the naming convention discussed above for the preparation blank.

6.4.10 Enter the entire Ross Analytical work order number for the sample. Example 96-00-000-00A. If the sample is a duplicate, enter 96-00-000-00AD. If the sample is a spike or spike duplicate, enter 96-00-000-00AS or 96-00-000-00ASD. If the sample is a post-spike or post-spike duplicate, enter 96-00-000-00AA or 96-00-000-00AAD. On samples that need dilutions, try to fit the whole work order number with the dilution. If necessary, truncate from the left of the work order number until the dilution and sample fit in the space allowed.

6.4.11 After entering each set of 10 samples, (including PB, LCS, S/SD, A/AD and other QC samples) insert CCVs and CCBs. The ICS standards must be run at least twice every eight hours or at the beginning and end of an analytic run, which ever is more frequent. If the analysis is anticipated to last more than eight hours, insert an ICSA/AB to conform with this rule. Always end your table with an ICS sequence and CCV/CCB set. Remember to change the CheckTable column to reflect the method QC requirements for the CCV, CCB and ICSA/AB.

6.4.12 After all the samples have been added to the table, delete any unused sample positions by highlighting the unused position and pressing F2. After all the unused sample positions have been deleted, press F9 until the computer displays in the upper left-hand corner the message "Examining Autosampler Table." Press F2 to print a listing of the autosampler table developed. When printing is complete, press F9 to save the autosampler table and return to the main menu. Update the method as described in the next section.

6.5 Updating the Method

6.5.1 Select the **Development** pulldown menu, highlight **Methods**, and press ENTER.

6.5.2 Type in CLPNEW1, if it is not the default answer in the prompt, press ENTER.

6.5.3 Highlight the answer to the prompt "Analysis Data File". Type in the date of analysis and which run of the day it is (e.g., if the date is 01/01/96 and it is the first run of the day, type in JA0196A.).

6.5.4 Highlight the answer to the prompt "Autosampler Table". Type in the name of the autosampler table (see Section 6.4 for autosampler table set up).

6.6 Limit Check Tables

6.6.1 Select the **Development** pulldown menu, highlight **Sample Limit Checks**, and press ENTER.

6.6.2 Type in the name of the limit table, if known, then press ENTER or press F6 to get a list of available tables. When choosing from a list, enter the number of the corresponding table then press ENTER twice to edit the table.

6.6.3 The table LIM6010A contains the linear range for HIGH LIMIT and the EQL for the LOW LIMIT. It is used for checking acceptable result ranges for unknown samples.

6.6.4 The table CCB6010A is set up to flag analyte concentrations that exceed the absolute value of the EQL.

6.6.5 The table ICSA is set up to flag concentrations of interferences (e.g., Al, Ca, Fe and Mg) that exceed a range of 80 - 120% of the true value, and to flag analyte concentrations that exceed the absolute value of the 2 times the EQL .

6.6.6 The table ICSAB is set up to flag concentrations of interferences and analytes that exceed a range of 80 - 120% of the true value.

6.6.7 The table CRI6010A is set up to flag concentrations of all analytes that exceed a range of ±4 times the EQL.

6.7 Quality Control Check Tables

6.7.1 The table ICV is set up to flag analyte concentrations which exceed a range of 90 - 110% of the true value.

6.7.2 The table CCV is set up to flag analyte concentrations which exceed a range of 90 - 110% of the true value.

6.8 Instrument Shutdown

6.8.1 Extinguish the plasma by choosing **Setup** from the main pulldown menu, highlighting **Plasma Control Panel**, then pressing ENTER. Choose option F7 = Shutdown. The plasma will automatically shut off.

6.8.2 Release the pump tubing cassette from the pump and release the tension arm to relieve the tension on the pump tubing. The pressure must be released when the pump is not turning or it causes deformation of the tubing and will lead to analysis problems.

7.0 ANALYSIS

7.1 Sample preparation

7.1.1 Line up the number of 30 mL sample cups needed to prepare each sample in the analysis set including all PBs, LCSs, and MS/MSD pairs on the bench top.

7.1.2 Using a 100 uL pipette, place 100 uL of 100 ppm Y standard (see Section 5.3.3) and 200 uL of stock Li standard (10,000 ppm) into each cup.

7.1.3 Using a 10 mL pipette, place 10 mL of each sample into each cup. Mix by gently swirling the cup. Pour the sample into the 16 X 100 mm plastic ICP autosampler tubes, filling the tube to the top (approximately 9 mL).

7.1.4 Follow the printout of the autosampler table to place the samples correctly into the rack.

7.2 After starting and equilibrating the instrument, setting up the autosampler table, defining the method, checking and defining limit and QC check tables, and loading samples and standards into their correct positions in the autosampler tray, analysis can begin.

7.2.1 To begin analysis first select **Operation** from the main pulldown menu, then highlight **Analysis** and press ENTER.

7.2.2 The software will prompt what method to use. CLPNEW1 is currently the method of choice for analyses under SW-846, Method 6010A protocol. If it is not already the default answer for the prompt, type in CLPNEW1 and press ENTER.

7.2.3 Press F9 to run an analysis using a developed autosampler table.

7.2.4 The software will display the autosampler table specified in Section 6.4.4; if it is correct, press ENTER.

7.2.5 The software then displays the first solution to be analyzed. For most cases this will be a BLANK. This can be changed by using the arrow keys.

7.2.6 Press F1 to begin the analysis. The analysis may be halted when the autosampler is running a sample (not rinsing) by pressing F1 then ENTER twice. The software then displays the same screen as in Section 6.5.6. Repeat procedure from this section to restart analysis.

7.3 Calibration Standards

7.3.1 Section 7.3 of SW-846, Method 6010A states "Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. The calibration curve should consist of a blank and three standards." This statement is contradictory if the "manufacturer's recommended procedures" is a two point calibration, (e.g.,, a blank and one standard), as is the case with the TJA 61E Trace ICP. It has been experimentally verified that the procedures recommended by the manufacturer, when followed correctly, produce more accurate data in a more efficient manner than that obtained using a three point calibration curve, especially for data at or near the detection limit. Therefore the manufacturer's recommendations for calibration have been adopted by this SOP.

7.3.2 Validation of the entire calibration range is accomplished using check standards at low medium and high levels during the analytic run.

7.4 Samples

7.4.1 All samples and quality control checks are analyzed in the same manner. No special consideration is provided to any analysis which would bias a result (such as longer or shorter rinse times). Triplicate readings are achieved for each sample and results averaged.

7.4.2 Sample results must fall within the established linear range. This includes analytes which may not be required for reporting to the client but for which their presence may influence correction factors or analyte behavior.

7.4.3 Some samples may require dilution due to their viscosity and ability to be handled by the delivery system of the instrument. When performing dilutions, use caution to achieve the least dilution possible without affecting the validity of the results or operation of the system.

7.4.4 If a sample is causing contamination problems due to high levels of analyte, the system may be interrupted and allowed to rinse prior to analysis of another sample. The efficiency of the rinse must be verified by running a blank as a sample before continuing analysis.

8.0 CALCULATIONS

8.1 Soil

$$\text{Sample Result (mg/kg)} = \text{instrument result (mg/L)} \times \text{instrument dilution} \times \frac{\text{final prep vol (mL)}}{\text{sample wt (g)}}$$

8.2 Water

$$\text{Sample Result (mg/L)} = \text{instrument result (mg/L)} \times \text{instrument dilution} \times \frac{\text{final prep vol (mL)}}{\text{initial prep vol (mL)}}$$

9.0 QUALITY CONTROL

9.1 Before beginning sample analysis check standards and other quality control samples must be analyzed.

9.1.1 First, reanalyze the calibration standards as samples. Results should agree within ±5% of the known values. If this control limit is not met, determine and correct the cause of the problem. Recalibrate the instrument.

9.1.2 Analyze the Initial Calibration Verification Standard(s), ICV, after the highest calibration standard has been reanalyzed. This standard is used to validate the source, ensure that the correct procedure was used for preparation of the calibration standards, and the accuracy of the calibration curve. Results must fall within ±10% of the true value. If this control limit is not met, determine and correct the cause of the problem. Recalibrate the instrument.

9.1.3 Analyze the Initial Calibration Blank Standard, ICB, after the ICV. This standard is used to verify that carryover is not occurring and that results near the detection limit are not biased by the slope and intercept of the calibration curve. The absolute value for the results of the ICB shall not be greater than the reported EQL. If this control limit is not met, determine and correct the cause of the problem. Recalibrate the instrument.

9.1.4 Analyze the Low Level Check Standard, CRI, after the ICB. This standard is used to verify the accuracy of results near the detection limit. Results of the CRI should agree within ±4 times the EQL (e.g., if the EQL is 10 ppb, the acceptance range is 0 - 40 ppb). Although this is not a method requirement, this criteria has been adopted to validate the use of the calibration curve described above. If this control limit is not met, determine and correct the cause of the problem, reanalyze the CRI. Recalibration may be necessary.

9.1.5 Analyze the Interference Check Standard, ICSA, after the CRI. This standard is used to verify the accuracy of the interferences, the accuracy of correction factors applied, and their effect on results in the absence of analyte. Results for the interfering elements (Al, Ca, Fe, and Mg) should agree within ±20% of their true value. Results for the analytes affected by the interferences should not be greater than ±2 times the reported EQL. If these control limits are not met, determine the cause and correct the problem. Recalibration may be necessary. The ICSA must be run at the beginning and end of the analytic run, or twice during each 8-hour shift, whichever is more frequent.

9.1.6 Analyze the Interference Check Standard with Analytes, ICSAB, after the ICSA. This standard is used to verify the accuracy of the interferences, the accuracy of interelement and background correction factors, and their effect on results in the presence of analyte. Results for the interfering elements and the analytes should agree within ±20% of their true values. If these control limits are not met, determine the cause and correct the problem. Reanalyze the ICSAB. Recalibration may be necessary. The ICSAB must be run at the beginning and end of the analytic run, or twice during each 8-hour shift, whichever is more frequent.

9.1.7 Although not a method requirement, it is prudent to "close out" the sequence of check standards that are run just after the calibration with a Continuing Calibration Verification Standard (CCV) and a Continuing Calibration Blank (CCB). Control limit criteria for these standards is the same as that for the ICV and ICB. The CCV and CCB should then be analyzed after every ten samples (Preparation Blanks, LCS, S/SD, A/AD and other QC samples are counted as samples), and at the end of the analytic run.

9.1.8 A serial dilution of a sample within a batch should be performed if matrix interference is suspected. Dilution results should agree within ±10% of the original values if the analyte concentrations are greater than a factor of 10 above the EQL after the dilution. Further dilution will be necessary if the criteria is not met.

9.1.9 A minimum of one method preparation blank per sample batch must be prepared and analyzed to determine if contamination from the preparation procedures has occurred. The method preparation blank is carried through the entire process as a sample. Results for the method preparation blank should be less than the reported EQL. If these criteria are not met, determine the cause and correct the problem. Repreparation of the blank, and the entire batch associated with it, may be required. In some cases, the blank may be subtracted from the sample results if it is verified that the cause was consistent for all results. Results may also be reported when the blank exceeds the EQL if sample results exceed the blank concentrations at a level twenty times that of the EQL.

9.1.10 Analysis of a Laboratory Control Sample (LCS), described as a "quality control sample" in Section 5.8 of Method 6010A, should be performed for each batch. If possible, the LCS matrix should mimic that of the samples in the batch. This helps to validate the applicability of the method utilized for the matrix. Results of the LCS should agree within ±20% of the theoretical value or within the confidence interval established by the manufacturer. If this criteria is not met reprepare and reanalyze the samples associated with the batch. In some cases repreparation is not possible when all of the sample is used during initial preparation. In these cases flag the data associated with the QC failure using a narrative form and/or a nonconformance report form.

9.1.11 Spiked replicate samples are to be analyzed at a frequency of 5% (one set of replicates per twenty samples) or per analytic batch, whichever is more frequent. Analyzing spiked replicate samples also fulfills the requirement for analysis of a replicate sample (SW-846 Method 6010A, Section 8.4). The spikes should be made prior to preparative procedures and then processed as samples.

These matrix spikes and matrix spike duplicates are identified by adding S and SD to the ends of sample ID's. Spike levels are specific to the matrix and are covered in SOPs for the preparation methods used. Recovery and reproducibility must be determined using results of these samples. The S and SD recovery must be within ±20% of the theoretical value, as determined using the following formula (note - all dilution factors must be corrected prior to use in this formula):

$$\%REC = \frac{(\text{spike result - sample result})}{\text{spike added}} \times 100$$

If the recovery is not within control limits, a procedural error or matrix interference may be suspected. Presence of the interference and its effect on the analytic system employed must be determined. This is accomplished by diluting and reanalyzing the replicate spikes and samples until control limits are met, or by performing a post-digestion spike.

The reproducibility, expressed as relative percent difference (RPD), is to be within 20% for sample values greater than ten times the IDL. The following formula is to be used to calculate RPD:

$$RPD = \frac{(\text{first sample result - second sample result})}{\text{average of first and second results}} \times 100$$

If the reproducibility is not within control limits, a procedural error or matrix effect may be suspected. Analysis of a post digestion spike and post spike duplicate, and respective RPD values, are required to determine the cause of the problem.

9.1.12 Post digestion spikes are performed to determine if quality control failures from the S/SD analyses are due to matrix effects or to procedural problems and if the effects are matrix related. If matrix related, post digestion spikes provide information regarding what degree of bias is encountered. Post digestion spikes and spike duplicate samples are identified with an A/AD at the end of the sample ID. Post spike recovery and reproducibility are calculated in the same manner as that for matrix spikes. The post spike should be recovered within 75% to 125% of the theoretical value for analytes whose concentrations are less than 4x the spike level added. If this limit is not met, matrix interference is suspected and results shall be flagged using a narrative with the report.

9.1.13 Instrument Detection Limits (IDL) must be determined quarterly and whenever a new element is added. IDLs are determined by running seven replicates of a solution that is at a concentration 3 to 5 times the expected IDLs of the elements, then multiplying the standard deviation of the seven replicates by 3. Average results from three, nonconsecutive days are used for determining the final IDL value.

9.1.14 Method Detection Limits (MDL) must be determined annually and whenever a new element is added. The MDL must be determined for each matrix (e.g., aqueous and solid). The MDLs are experimentally determined by averaging results from analysis of seven replicate samples which have been spiked at a level approximating the EQL. Each matrix is spiked and prepared for analysis according to a defined method for that matrix.

9.1.15 The Estimated Quantitation Level (EQL) is a value derived from the MDL which is rounded and adjusted to reflect other laboratory constraints (e.g. background contamination), and market requirements. The EQL is typically one to ten times the MDL, but at no time may it be lower.

9.1.16 Instrument Linear Ranges (LR) must be determined quarterly and whenever a new element is added. The LR of an element is determined by analyzing the element at successively higher concentrations until self absorption or other effects cause the accuracy of results to fall outside a range of ± 5 % of the true value. The result for the LR is then rounded to a whole number.

10.0 INTERFERENCES

10.1 Spectral Interferences

Spectral interferences are caused by: (1) overlap of a spectral waveline from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or recombinant phenomena; and (4) stray light from the line emission of high concentration elements. Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element (e.g., applying IEC factors). Unresolved overlap requires selection of alternate wavelengths. Background and stray light can be corrected using background correction (BGC) adjacent to the analytic line.

The wavelengths and background correction points utilized in this method have been carefully chosen after extensive experimental evaluation. The IDL, MDL, LR and IEC values are all affected by the choice of the BGC points. Therefore, if the wavelines and BGC points for each element are altered, new IDLs, MDLs, LRs and IECs must be determined.

10.1.1 Element-specific interference is expressed as analyte concentration equivalents (e.g., false analyte concentrations) arising from the interfering elements. The interference effects of each element on every other element must be experimentally determined under conditions which mimic normal operations. The values achieved are used to calculate Interelement Correction Factors (IECs).

For example: If the effect of 1000 ppm of aluminum produces a false concentration for arsenic at a concentration of 4.814 ppm, the IEC factor is 0.004814 (4.814/1000).

10.1.2 Interference correction is applied during analysis by multiplying the IEC by the concentration of the interfering element in a sample and subtracting or adding the result to the analyte concentration. It is important to use the correct sign (+ or -) for the IEC as there may be additive or subtractive forces at work. If the effect of the interference is additive to the analyte concentration, the sign used for the IEC in the ThermoSpec software is also positive. If the effect of the interference is subtractive to analyte concentration, the sign used is negative.

10.2 Physical Interferences

Physical interferences are effects associated with the sample nebulization and transport processes. Differences in viscosity, volatility and surface tension between calibration standards and samples can cause inaccuracies. This is most apparent for organics and samples with high TDS levels or acid concentrations.

10.2.1 Physical interferences can be reduced by diluting the sample, using a peristaltic pump and by normalizing the data with an internal standard. In the case of the TJA61E Trace ICP, Y is used as an internal standard.

10.2.2 Salt buildup from samples with high TDS levels can clog the nebulizer tip and torch injector, affecting aerosol flow, and resulting in instrument drift. This problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. The nebulizer frequently requires cleaning to prevent salt buildup. This is recommended as a daily preventative maintenance task.

10.2.3 Temperature fluctuations in the environment cause differences in the density and surface tension of samples and standards resulting in inaccuracies and instrument drift. This problem is best controlled by maintaining a stable temperature in the room, but can be corrected for by using an internal standard.

10.3 Chemical Interferences

Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects.

10.3.1 Molecular compound formation is controlled in part by utilizing preparation procedures that break down complex structures and stabilize dissolved elements. The ICP technique itself aids to minimize this phenomena.

10.3.2 Ionization effects can be overcome by adding high concentrations of alkali salts to force ion equilibrium to favor analytes of interest. Lithium carbonate is used for this with the TJA 61E Trace ICP.

10.3.3 Solute vaporization effects are controlled by matrix matching or by method of standard additions.

11.0 TROUBLESHOOTING

11.1 Printer does not work

11.1.1 Not plugged in

11.1.2 Not turned on

11.1.3 Not connected to computer

11.1.4 Paper jammed or empty

11.2 Analysis will not commence

11.2.1 10 Amp fuse on power distribution panel blown

11.2.2 Double toggle on source not on

11.2.3 Double toggle on power distribution panel not on

11.2.4 Reset button not pressed

11.2.5 Unit not warmed up

11.2.6 Door to sample chamber has not been opened and closed since last burn

11.2.7 Source not functioning properly

11.2.8 1 Amp fuse on low voltage power supply blown

11.2.9 Safety interlock switches not shut off

11.3 Poor Analytical Results

11.3.1 Instrument not profiled correctly

11.3.2 Sample and standards not matrix matched

11.3.3 Argon gas flows and pressures wrong

11.3.4 Leaks in argon system

11.3.5 Poor argon gas quality

11.3.6 Blockage, leaks or broken connections in delivery system tubing

11.3.7 Room temperature not stabilized (2 degrees per hour maximum change)

11.3.8 2 Amp high voltage power supply fuse blown

11.3.9 IEC factors not applied, or applied incorrectly

11.3.10 Standards contaminated

11.3.11 Standards prepared incorrectly

11.3.12 Standards too old

11.3.13 Torch dirty

11.3.14 Pump tubing worn, deformed or contaminated

11.3.15 Peristaltic pump not turning smoothly

11.3.16 BGC points not appropriate

12.0 TABLE(S)

TABLE 1 - ESTIMATED QUANTITATION LIMITS

ELEMENT	EQL (ug/L)
Aluminum	100
Antimony	5.0
Arsenic	5.0
Barium	2.0
Beryllium	1.0
Cadmium	1.0
Calcium	200
Chromium	10
Cobalt	5.0
Copper	5.0
Iron	50
Lead	5.0
Magnesium	50
Manganese	5.0
Molybdenum	5.0
Nickel	10
Potassium	200
Selenium	5.0
Silver	5.0
Sodium	500
Thallium	5.0
Tin	10
Titanium	5.0
Vanadium	10
Zinc	20

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

SOP No.: GC-001-00
Initial Date: 12/4/96

Date Revised: NA

Title: New Column Testing			
Prepared by:	Approved by: 12/4/96	Date:	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to ensure that each new column (media or film coating) is tested and the chromatography checked for proper elution order prior to the column being used for sample analyses.

2.0 APPLICATION

This procedure is to be applied for all new columns which are purchased for use in the GC laboratory.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Following conditioning of the new column but prior to use for sample analyses, a standard(s) containing the compound(s) of interest is analyzed. The chromatogram is then compared to the chromatogram provided by the vendor.

If additional or other compounds are analyzed which are not contained on the chromatogram provided by the manufacturer, the individual components must be injected and analyzed separately to indicate where they elute on the column.

5.2 Once the data have been compiled, the analyst must submit it to the Group Leader, Operations Manager, or Quality Assurance Manager for sign-off approval.

5.3 Sample analysis may begin once sign-off approval has been obtained.

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Analysis of Organophosphorus Pesticides by GC/TSD			
Prepared by: [illegible]	Approved by: [illegible]	Date: 8-24-94	QA Approval: [illegible]

1.0 PURPOSE

The purpose of this procedure is to describe the analysis of organophosphorus pesticides by Gas Chromatography using a thermionic specific detector (TSD).

2.0 APPLICATION

This procedure utilizes information from EPA SW-846, methods 8140 for packed columns and 8141 for capillary columns. It describes the elements necessary to comply with these methods for the quantitative analysis of organophosphorus pesticides. Discussion of interferences is found in Section 9.0. Table 1 list the targets and their corresponding EQL/RLs. This procedure applies to the analysis of soil, water, and waste matrices.

3.0 REFERENCES

EPA SW-846, method 8140 - Organophosphorus pesticide analysis by packed column.
EPA SW-846, method 8141 - Organophosphorus pesticide analysis by capillary column.
EPA SW-846, method 8000 - Gas Chromatography.
EPA SW-846, method 3500A - Organic Extraction and Sample Preparation.
EPA SW-846, method 3510A - Separatory Funnel Liquid-Liquid Extraction
EPA SW-846, method 3550 - Sonication Extraction
EPA SW-846, method 3600A - Cleanup.
EPA SW-846, method 3660 - Sulfur Cleanup.
Varian Manual - Publication Number 03-914089-00, for Thermionic Specific Detectors, 1987.

4.0 ASSOCIATED SOPS

OP-019 - Extraction of Organophosphorus Pesticides from Soil Samples.
OP-018 - Extraction of Organophosphorus Pesticides from Water Samples.

5.0 EQUIPMENT

5.1 Gas Chromatograph - Varian 3400 suitable for direct- injections with a Thermionic Specific Nitrogen/Phosphorus detector.

5.2 Autosampler - Leap Technologies autosampler capable of being programmed to inject a sequence of samples using a 10uL syringe.

5.3 Gas Chromatographic Column - RTx-1701

5.4 Data System - currently "Chromperfect," capable of collecting data points to measure peak areas and calculate results. Also includes Perkin-Elmer data boxes which collect data during the run and then transfer this information to the system upon completion of a run.

5.5 Syringes - various volumes

5.6 Autosampler Vials - 2mL volume

5.7 Syringe rinse bottles for autosamplers

5.8 100mL and 200mL volumetric flasks

6.0 STANDARDS AND SOLUTIONS

6.1 Stock Standards

Organophosphorus standards are purchased from a commercial vendor at a concentration of 2,000 ug/mL. A Certificate of Analysis accompanies the ampules certifying that the material meets or exceeds QA/QC criteria established by the U.S. EPA for commercially purchased analytical standards. This certificate which comes with each ampule must be filed with the Manager of Quality Assurance. After coming to room temperature, this commercial standard is diluted down by adding 0.25mLs from the ampule to a well-rinsed 50.0 mL volumetric that is partially filled with hexane. A malathion stock must be added to this also since this surrogate is not part of the purchased mix. The malathion stock should be at 630 ug/mL, and 1.0 mL of this is added to the 50.0 mL volumetric. The volume is brought up to 50 mLs with hexane for a final concentration of 10.0 ug/mL and 12.0 ug/mL for malathion. From this stock solution the six working standards are made. All standards must be refrigerated and protected from light in amber bottles. The 10.0 ug/mL stock solution must be replaced after six months or sooner if comparison with check standards indicates a problem (method 8141-section 5.6.3). All standards must be logged into the standards logbook and given an identification according to the GC system. When these standards are verified, the verifying chromatograms and other information must be placed into the standards logbook behind the logbook page containing the serial dilutions and calculations.

6.2 Calibration Standards

Six levels of standards are made using the 10ug/mL stock solution containing the nine organophosphorus targets plus the surrogate. The levels are 50ng/mL, 100ng/mL, 150ng/mL, 200ng/mL, 1000ng/mL, and 2000ng/mL. These are made by diluting down the stock solution in hexane to the desired concentration. The lowest level is near, but above, the method detection limit. The highest level is utilized to correspond to the expected upper range of concentrations in the samples and/or the working range of the detectors. Calibration standards must be replaced after two months or sooner if comparison with a check standard indicates a problem. (per method 8141-Section 5.7).

6.3 Surrogates

Malathion is the surrogate used to monitor the performance for the sample extraction, sample cleanup (when performed), instrumentation, and the effectiveness of the method in dealing with each sample matrix. The working surrogate is prepared in the GC lab from a stock solution. A malathion standard stock solution is made by taking approximately 17mg of the malathion and adding it to a rinsed 100mL volumetric partially filled with hexane. The volume is then brought up to 100mLs giving a concentration of 170ug/mL. This stock is then diluted down to make the working surrogate. This is usually done by taking 2.5mLs of the 170ug/mL stock and adding it to a well-rinsed 200mL volumetric partially filled with acetone. The volume is then brought up to 200mLs for a concentration of approximately 2ug/mL.

One mL of this surrogate stock is spiked into the blanks, check samples, samples, matrix spikes, and matrix spike duplicates prior to extraction.

6.4 Laboratory Control Standards (LCSs), Matrix Spikes (MSs), And Matrix Spike Duplicates (MSDs)

The commercially-purchased stock standards containing the nine organophosphorus pesticides are diluted down to make a spike at the concentration of 1.0 ug/mL in acetone. The organic preparatory lab spikes 2.0 mLs of this spike into a blank to make the LCS, and 2mLs each into two duplicates of a sample to make the MS and the MSD. A LCS is extracted with every batch of samples or every twenty samples, whichever is more frequent. MS/MSDs are spiked for every twenty samples of each matrix and/or when specifically requested by the client.

6.5 Internal Standard

An internal standard (I.S.) is an analyte that has similar analytical behavior to the compounds of interest. It is utilized to ensure all calculations are done on a uniform basis. It should be noted that, generally, the internal standard being used should not be affected by method or matrix interferences. However, no internal standard will be applicable to all samples. Therefore the analyst must be aware of any fluctuations in internal standard response in the samples in order to ensure matrix interferences are not occurring and affecting the calculations. In this case dichlorvos is used as the I.S. The I.S. solution is prepared at a concentration of approximately 28ug/mL. An aliquot of 10uL of I.S. is spiked into exactly 1.0mL of all standards, blanks, LCSs, samples, MSs, and MSDs in the GC lab just prior to analysis.

7.0 PROCEDURE

7.1 GC Operating Conditions

Samples are analyzed using a Varian 3400 GC under the following conditions:

7.1.1. Detector: Thermionic Specific (TSD).
Column: 30 M x 0.53mm ID, 1um df RTx-1701 capillary column.
Confirmation Column: RTx-5
Injector Temperature: 250°C
Detector Temperature: 300°C
TSD Range: 12

7.1.2 Approximate Gas Flows:
Carrier Gas (Helium) : 6-8 mL/minute.
Make-up Gas (Helium): 22-24 mL/minute.
Air: 175 mL/minute.
Hydrogen: 4.5mL/minute.

7.1.3 Temperature Program: Initial temperature 45°C hold 0.50 minutes. Ramp1: 10°C/minute to 160° and hold for 2.0 minutes. Ramp2: 4°C/minute to 200°C and hold 0.0 minutes. Ramp3: 10°C/minute up to 275°C and hold for 2.50 minutes for a total run time of 34.00 minutes. The current temperature program should be kept current and on file in the analytical conditions logbook. With any modification of the temperature program, this SOP must be updated accordingly. A copy of the most recent analytical conditions logbook page follows this SOP as figure I.

7.1.4 The elution order of the organophosphorus pesticides on an RTx-1701 column is as follows:

1. O,O,O-TEPP
2. Dichlorvos (Internal Standard)
3. Thionazin
4. Phorate
5. Sulfotepp
6. Disulfoton
7. Dimethoate
8. Methyl Parathion
9. Malathion (Surrogate)
10. Ethyl Parathion
11. Famphur

For a sample chromatogram of the targets above, see Figure II. following this SOP.

7.2 Calibration Procedure

7.2.1 Initial Calibration

The six levels of the calibration standards are run first to create calibration curves. A 1uL injection is delivered to the instrument by the autosampler. Peak area is tabulated against concentration on column to give a response which is based on the internal standard called the Relative Response Factor (RRF).

RRF = As Cis / Ais Cs where: As = Response (area) for analyte to be measured.
Ais = Response for the internal standard.
Cis = Concentration of the I.S., ng on column
Cs = Concentration of the analyte to be measured, ng OC

This can be done "by hand" or more likely, by the computer software. If the percent relative deviation (%RSD) of the response factor is less than 20% over the working range, linearity through the origin can be assumed, and the average relative response factor can be used in place of a calibration curve. This average RRF would be utilized when calculating results "by hand." Generally speaking, the analyst will be using the calibration curve and computer generated results and not hand calculating. When the computer-generated curve is being used, the 20% rule is not applicable-there is no criteria. Note: The RRF values are only valid for calculating if the sample is being calculated using the internal standard. If a dilution is done then an appropriate volume of I.S. is added to the dilution to maintain I.S. levels.

7.2.2 Working Calibration Curve Verification

The working calibration curve must be verified on each working day by the injection of one or more calibration standards. These "continuing checks" must be the mid-level standard. A continuing check must also be run after every ten runs on the instrument and at the end of the analytical sequence to ensure that the initial calibration curve is still being matched. The response is checked by comparing the response factor of the continuing check (Area/concentration) to the mid-level standard response factor from the initial calibration. This is accomplished by doing a percent difference (%D) calculation where:

$$\%D = \frac{\text{initial cal RRF (mid std)} - \text{cont. check RRF}}{\text{initial cal RRF (mid std)}}$$

If the value varies by greater than + or - 15% for an analyte, then this analyte is considered to be out of control. A new calibration curve may need to be run for that analyte. However, other troubleshooting procedures may need to be employed before going to this measure, such as running solvent blanks through the instrument to make sure that no samples have contaminated the system. In this case, a second continuing check should be run after rinsing. If the analytes are still out of control, maintenance may need to be done, such as replacing the inserts and/or clipping the column. Then a new calibration curve would need to be run and the samples would be rerun. It is only necessary to rerun the calibration curve if the targets out of control are found in the samples. However, before the next set of samples is run, the proper maintenance should be done so that the quality control samples, such as LCSs, that may contain any out of control targets can be properly assessed.

7.2.3 Retention Time Windows

To calculate retention time windows, three injections are made of one level of the calibration curve over the course of a 72 hour period. Injections over less than a 72 hour period result in windows that are too tight (see Method 8000A, section 7.5) The standard deviation of the three retention times is calculated. Plus or minus three times the standard deviation of the retention times for each target will be used to define the windows. If a daily continuing check standard has compounds outside the windows, then the retention times are updated. If any continuing checks that are run later in that sequence fall outside the retention time windows, then these are considered out of control, and any samples following them must be rerun.

7.3 Analysis

After it is determined that the continuing checks match the calibration curve, then blanks, LCSs, samples, MSs, and MSDs are run in that order. If the responses (areas) of the samples exceed the linear range of the calibration curve, meaning their area counts exceed the highest standard's area counts, then dilute and reanalyze the sample. Results will be calculated without the internal standard being taken into consideration since the I.S. is diluted out (see section 7.2.1). Continuing calibration checks must be analyzed after every ten runs on the instrument and also at the end of the analytical sequence. If the continuing checks which run later in the sequence show retention time shifts or are out of control limits for other reasons, then the samples running after them must be reanalyzed.

7.4 Data Interpretation and Calculations

7.4.1 The analyst should follow this checklist for analyzing and interpreting data:

1. Inspect the chromatograms for any obvious problems such as peak and peak patterns other than targets that could interfere with the analysis.

2. Look for retention time shifts where the I.S. and surrogates are not called by the computer. If this occurs, run a standard to be sure the system is not at fault.

3. Calculate the surrogate recovery and check to see that it falls within the QC limits. If it does not, then reanalyze the sample. If it is still out of limits, request a re-extraction.

4. Look at the area counts for any hits to make sure they fall within the linear range of the calibration curve for that target. If over the curve, then dilute and reanalyze.

7.4.2 Target components are considered to be "hits" if their retention times fall within the retention time windows for a given analyte. Column confirmation is not routinely being employed at RASI

for this analysis. Methods 8140 and 8141 suggest that this be done. The concentrations of the analytes are determined by the following calculation:

ng/mL = (As)(Cis)/(Ais)(RRF)

where:
As=Response (area) of the analyte to be measured
Ais=Response of the internal standard
Cis=Concentration in ng on column of IS
RRF=relative response factor of analyte

The RRF is determined for each analyte where RRF=(As)(Cis)/(Ais)(Cs)

where:
As=Response (area) of analyte to be measured
Ais=Response of I.S.
Cis=Concentration in ng on column of I.S.
Cs=Concentration in ng on column of analyte

These formulas have already been set up in the software. If the results are to be reported in ug/Kg or ug/L then the calculation would go one step further:

$$\frac{\text{ng/mL} \times \text{final volume (mLs)} \times \text{any dilution factor}}{\text{weight (Kg) or volume (L)}}$$

This final calculation would be done by hand, and then the result would be written onto the client data sheet. Note: If the responses of the samples exceed the linear range of the calibration curve meaning, the area counts exceed the area counts of the highest standard, dilute and reanalyze the sample.

8.0 QUALITY CONTROL

Prior to performing this analysis, a one-time analyst proficiency study must take place for each of the individuals performing the analysis. It is the responsibility of the Supervisor to read the SOP describing how to perform an analyst proficiency study and to notify the Manager of Quality Assurance to set this up.

Sample extracts from the organic preparatory lab have a hold time of forty days and must be run within that period. Water samples have a hold time of seven days from the date of collection, and soils have a hold time of fourteen days. Therefore, it is essential that the analyst try to analyze the extracts as soon as they are extracted so that any deficiencies in the I.S. response or surrogate recoveries can be discovered and any re-extractions that may be necessary can be done before the samples. LCS limits are generated on an annual basis. If the LCS falls outside the limits the sample batch must be reextracted and reanalyzed. Matrix specific surrogate recovery limits are generated by the QA department on a quarterly basis. The analyst must check the surrogate recovery to ensure that it does not fall outside these limits. If a sample is noncompliant, usually a re-extraction is requested unless there has been an MS/MSD pair done on the sample which could confirm a "matrix effect." Also, there may be a number of samples in the set that show the same "trend" in recoveries or interferences. In such cases, it is best to discuss these trends with the supervisor or Manager of Quality Assurance in order to decide what corrective action should be taken. In some cases, the project manager may need to be included in the discussion so that the client can be contacted to make a decision.

9.0 INTERFERENCES

In most cases, the extracted sample is analyzed , and then if problems occur due to interferences, a cleanup is performed. It is recommended that cleanup also be performed on a series of standards to validate elution patterns for the compounds of interest and to verify the absence of interferences from reagents. As stated in method 8141,

section 1.7 and 7.5, Florisil cleanup and Gel Permeation Cleanup are not acceptable for organophosphorus samples because it has been demonstrated that they yield recoveries of less than 85% for many analytes. However, sulfur cleanup according to method 3660 is acceptable. According to the EPA methods 8140 and 8141 there are many factors that might interfere with the organophosphorus pesticide extraction. In EPA SW-846, method 3500A section 3.5 it is noted that soap residue on glassware may cause degradation of most organophosphorus analytes. Also, in Method 8141, section 7.1.2, it is noted that solvent exchange to 100% hexane during the extraction process has been shown in single laboratory data to cause loss of the more water-soluble organophosphorus compounds. It is recommended that a mixture of hexane and acetone be used instead. Due to the potential problems and the past problems that this laboratory has encountered with this analysis, it is essential that the analyst note any trends, and communicate these to the GC supervisor and the extraction laboratory.

10.0 TROUBLESHOOTING

It is highly recommended that the analyst read the Varian manual describing the Thermionic Specific Detector which detects nitrogen and phosphorus. There are very specific guidelines for conditioning the bead before initial and daily use. If followed, these guidelines will help to extend the lifetime of the bead. There is also information covered in the manual that would be helpful to the analyst when troubleshooting. Below are some common problems associated with this analysis and some suggestions for their correction. Analysts should add to this list on a regular basis to develop a more complete troubleshooting guide. These additions can be hand written and placed behind the GC lab copy of the SOPs.

PROBLEM:CORRECTIVE ACTION

1. Famphur degrades quickly in a dirty system. Corrective action to be taken is to swab out the insert or replace the insert if cleaning does not help.

2. Surrogates are out of limits: Make sure this is not due to the I.S., then rerun the sample. If the results are the same, request a re-extraction. A matrix effect has been confirmed if re-extract results are the same.

3. Decline in the response of the standards: Change the septa to eliminate any leaks. Rerun the std. If the response is still low then change the inserts-active sites may be affecting std. response. Clip some column from the insert side (bead does not need to be turned off during maintenance). Record all maintenance in weekly maintenance logbook. If these things do not help, try increasing the voltage through the bead to help increase sensitivity.

4. Tailing of peaks: Check the column installation and read the Restek column guide for help. Be aware that the silonizing reagents used to clean the inserts may be causing peak tailing. The brand currently used does not contain the N2 that would cause this, but another brand might. Decomposition of these reagents on the hot bead produces Si02 which deposits on the bead surface causing loss of response. The bead can be cleaned to remove these deposits.

TABLE 1
Organo-Phosphorus Targets and EQL/RLs

Compound	Waters EQL/RL (ug/L)	Soils EQL/RL (ug/Kg)
O,O,O-TEPP	0.25	8.33
Thionazin	0.25	8.33
Phorate	0.25	8.33
Sulfotepp	0.25	8.33
Disulfoton	0.25	8.33
Dimethoate	0.25	8.33
Methyl Parathion	0.25	8.33
Ethyl Parathion	0.25	8.33
Famphur	0.25	8.33

FIGURE I. This figure shows the most recent analytical conditions for the analysis of Organophosphorus Pesticides as it would be found in the Analyical Conditions Logbook in the GC lab.

ROSS
Ross Analytical Services, Inc. • 16433 Foltz Pkwy. • Strongsville, OH 44136 • (216) 572-3200 • FAX (216) 572-76.

ANALYTICAL CONDITIONS

CLIENT: Various
WORK ORDER:
METHOD: 8140 -8141
EPA SW-846

DATE: 7/21/94
ANALYST: TDM
STANDARD REF: S-G-718A → E

COMPOUND	R.T.	COMPOUND	R.T.
O,O,O-TEPP	8.702		
DICHLORVOS	10.476		
THIONAZIN	16.052		
PHORATE	17.464		
SULFOTEP	17.763		
DISULFOTON	20.076		
DIMETHOATE	21.609		
METHYL PARATHION	23.762		
MALATHION	24.638		
ETHYL PARATHION	25.374		
FAMPHUR	30.337		

DETECTOR TSD RANGE 12 DETECTOR TEMP 300 INJ. TEMP 250

SOLVENT Hexane COLUMN RTX-1701 .53

INITIAL TEMP 45 HOLD 0.50 RAMP1 10°/min TEMP1 160 HOLD 2.00
TEMP1 ~~200~~ 160 RAMP2 4°/min TEMP2 200 HOLD 0.00
TEMP2 200 RAMP3 10°/min TEMP3 275 HOLD 2.50
TEMP3 RAMP4 TEMP4 HOLD
TOTAL RUN TIME 34.00 min.

OBSERVATIONS

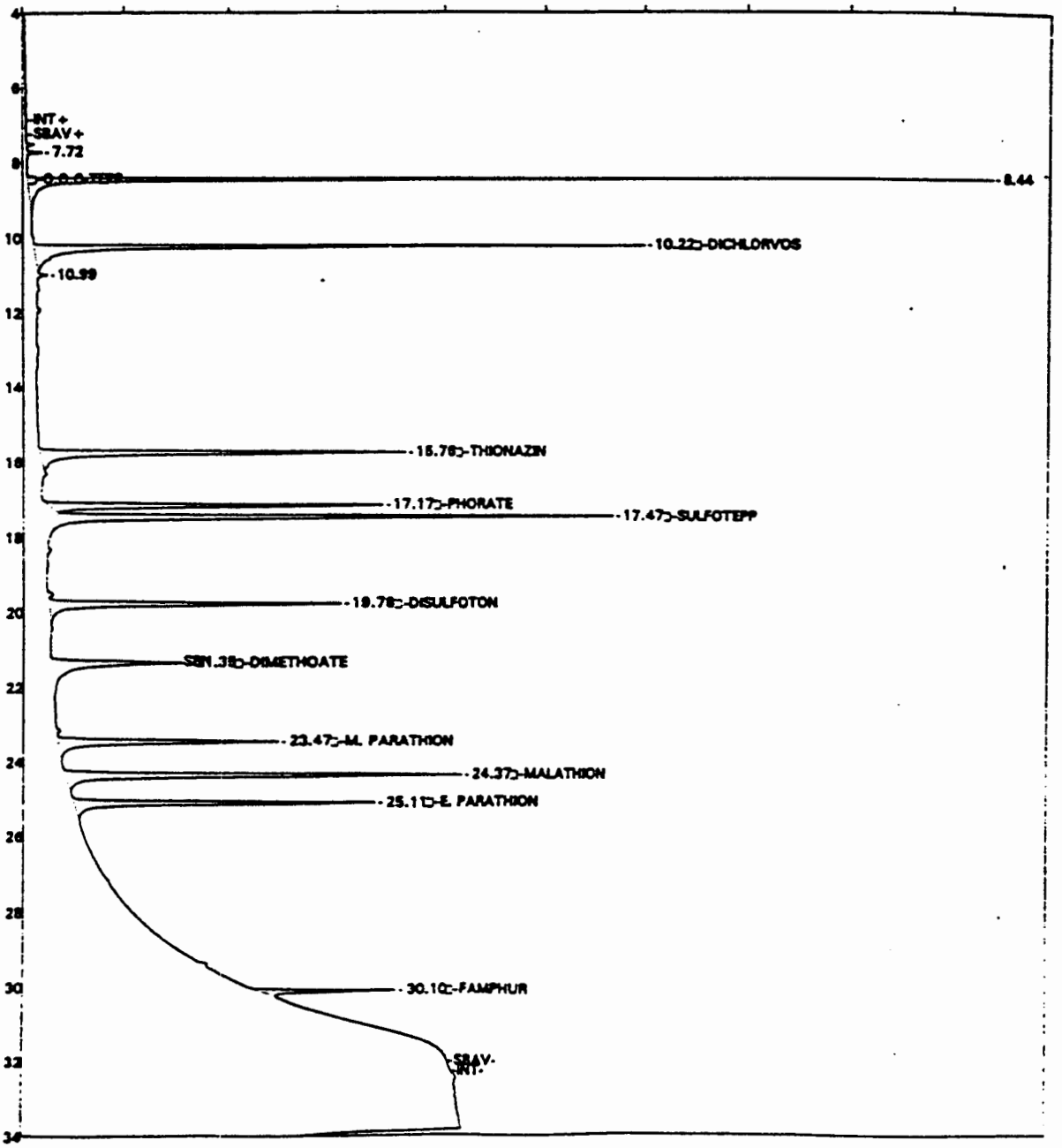
FIGURE II. This figure is of the plot of a chromatogram from the Organophosphorus Pesticide analysis done on an Rtx-1701 column on detector "T", a TSD detector.

SAMPLE CHROMATOGRAM

Ross Analytical Services, Inc.

File: C:\CP\ST\T0809.04R Date Injected: 08-09-1994 17:53:51
Sample Name: OPEST G-718D *CC* 200 NG/ML
Comments: GC/TSD Organophosphorous Pesticide Analysis
2ul inj., RTX-1701, 0.53 mm ID
Instrument: V3400T
Interface Box: 7 Date Printed: 08-09-1994 Time= 17:53:58

Chromatogram: 4.0 to 34.0 min. Low Y=5.132 High Y=39.078 mv Span=33.946



Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Organic Standard Preparation		
Prepared by: L. Anderson	Approved by: C. Caldwell Date: 4/1/93	QC Approval:

1.0 PURPOSE

To insure a uniform method of making standards in the organic laboratory.

2.0 APPLICATION

This procedure applies to all standards made for use in the gas chromatography lab, GC/MS lab and extractions lab.

3.0 REFERENCES

Tekmar Literature Lab No. 1

Laboratory Quality Assurance Manual

4.0 ASSOCIATED SOP's

MA-006 Analysis of SUMMA Polished Canisters by GC/MS for Non-CLP

5.0 PROCEDURE

5.1 Apparatus

5.1.1 Volumetric flasks.

5.1.2 Volumetric pipets, 0.5 and 1.0 mL.

5.1.3 Microliter (liquid) syringes, 100 uL and various sizes.

5.1.4 Disposable pipets (Pasteur).

5.1.5 Gas tight syringes.

5.1.6 "Mini-Nert" valves for screw top vials.

5.1.7 Flow controller and tee for lecture bottle in 5.6.1.

5.2 General

5.2.1 Check the appropriate method for concentration of stock and working standards, and the solvent to be used for dilution of the neat compound.

5.2.2 Estimate the levels for working standards and work backward from there. Keep in mind that the smallest quantity to be measured is 100 uL volume or 0.0100 g to retain 3 significant figures (if practically attainable, or needed).

5.2.3 Try to avoid using wiretrol pipets and syringes when possible. One half milliliter pipet is reliable for the lowest volume in stock preparation.

5.2.4 Find the neat standard with the highest purity (99+ purity is best).

5.2.5 If the purity is 95% or better, the density may be used uncorrected. If it is less than 95%, correct for the purity.

5.3 Solid Neat Standard

5.3.1 Calibrate the analytical balance (+/- 0.0001 g) and record the mass of the calibration weight in the appropriate book.

5.3.2 Record the volumetric flask mass and the final mass (with neat standard) in your lab book, or on the bottom of the standard preparation form (Figure 1), as appropriate. This will help to check for math errors.

5.3.3 Weigh the solid directly into a volumetric flask, being careful not to leave any material on the upper neck of the flask. Use a spatula for transfer or, if the mass is very small, place disposable pipet into the solid, put in bottom of flask and "blow-out" carefully with small bulb.

5.3.4 Never take standard material back out of the flask. If too much has been added, consider starting over unless more standard is not available; in that case, transfer to a larger volumetric flask with a minimum of three rinses.

5.3.5 Add solvent to 50-80% of flask volume. Swirl until solid is dissolved. Dilute to volume. Twist ground glass stopper to seal, invert the flask, and shake several times to mix thoroughly.

5.3.6 Do serial dilutions from this to get down to a level for working standards.

5.3.6.1 1 to 10 dilution is 1 mL in 10 mL vol.

5.3.6.2 1 to 20 dilution is 1/2 mL in 10 mL vol.

5.3.6.3 1 to 100 dilution is 100 uL in 10 mL vol.

5.3.7 If the standards are volatile, store with zero head space. Otherwise, transfer to vials and mark levels. Label with concentration date, initials, standard number, and expiration date (Figure 2). Refrigerate all calibration standards.

GC/LC STANDARDS PREP FORM

CLIENT: _____ PROJECT #: _____ CASE/W.O. #: _____ TO _____

DATE STOCK MADE: _____ SCALE #: _____ EXPIRATION DATE: _____

STOCK NAME: _____ ANALYST: _____ CHECKER: _____

COMPOUND	SUPPLIER	REAGENT #	PURITY	DENSITY	WT/VOL USED	SCALE #	FINAL VOL	STOCK CODE
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								

NOTES

_____

_____

_____

_____

Figure 1

Figure 2

ENVIRONMENTAL STANDARD

STANDARD _____ CONCENTRATION _____

STANDARD NO. _____ DATE PREPARED _____

PREPARED BY _____ EXPIRATION DATE _____

ENVIRONMENTAL STANDARD

Standard Name: _____ Prep. Date: _____
Use: _____ Exp. Date: _____
Other: _____ Prep. By: _____

Chemical	Conc.	Chemical	Conc.

5.3.8 Have standard preparation sheet checked by another lab member, then record the standard number in standards preparation log, and include original standard prep form.

5.3.9 Store standards at -20 ^{o}C +/- 2 ^{o}C.

5.4 Liquid Neat Compounds

5.4.1 If the compounds have a low volatility such as oils and liquids with a boiling point greater than 100 ^{o}C, the gravimetric method used for neat solids is preferred. (Pipets are calibrated with water and do not deliver the proper volume reliably with high viscosity liquids.)

5.4.2 For very volatile liquids (boiling point at or near room temperature) see Section 5.6.

5.4.3 If the volumetric approach is determined best, do the following, adding the least volatile solvent first and the most volatile solvent last.

5.4.3.1 Fill the flask with dilution solvent to just below the neck if syringes are to be used for neat compounds, and half full if pipets are being used.

5.4.3.2 Using volumetric pipet or syringes, deliver the determined amount: 1) on the surface with a pipet below the line on the volumetric, 2) below the surface of the solvent with a syringe.

5.4.3.3 If using a volumetric pipet, rinse the sides of the flask down with solvent after each addition. If you are using syringes this is not necessary (remember to flush syringes out between additions with wash solvent).

5.4.4 The volume of each target should be recorded on a standard prep form or in your lab book (as appropriate) along with the volume of the flask and other information.

5.4.5 Mix gently and fill to the line with solvent very carefully to avoid going over the line. Mix as before Section 5.3.5.

5.4.6 Do serial dilutions to working standards as before Section 5.3.6.

5.4.7 Store in septum-cap vial with zero head space. Label as before Section 5.3.7.

5.4.8 To calculate ug/mL or ng/uL, use the density closest to the temperature at which the standard was prepared.

5.4.9 Example for solvents (mid size charcoal tube) run on the FID.

$$\frac{0.5\ \text{mL} \times 0.800\ \text{g/mL}}{10.0\ \text{mL}} = 0.0400\ \text{g/mL or } 40\ \text{mg/mL}$$

1:100 is 400 ug/mL or ng/uL
1:10 of 1:100 is 40.0 ng/uL (1:1000)
1:10 of 1:1000 is 40.0 ng/uL (1:10000)

5.4.10 Label as before Section 5.3.7 and also use tags (Figure 2) if space allows on the bottle.

5.4.11 Store standards at -20 ^{o}C +/- 2 ^{o}C.

5.5 Making Standards for Liquid in Gas Phase

5.5.1 See the SOP for Organic Standard Preparation in a Static Dilution Bottle (MA-006). The only addition is to flush the 1 or 2 liter flask with inert gas (nitrogen). Run this as a blank before injecting the volatile solvent into the flask. Use a liquid microliter syringe for volume measurement. Use flush injection technique with a bubble of air before the solvent. An example is given below:

$$\frac{2\ \text{uL} \times \text{density}}{\text{liter}} = 1.32\ \text{mg/L (or ng/uL)}$$

$1000\ \text{L} = \text{M}^3$

Hexane:
mw = 86.17
D = 0.660 g/mL

$$\frac{1.32\ \text{mg}}{\text{L}} \times \frac{1000\ \text{L}}{\text{M}^3} = 1320\ \text{mg/M}^3$$

$$\text{conc.} = \frac{1320\ \text{mg}}{\text{M}^3} \times \frac{24.5}{\text{mw}} = 375\ \text{ppm}$$

5.6 Gaseous Targets in Liquid

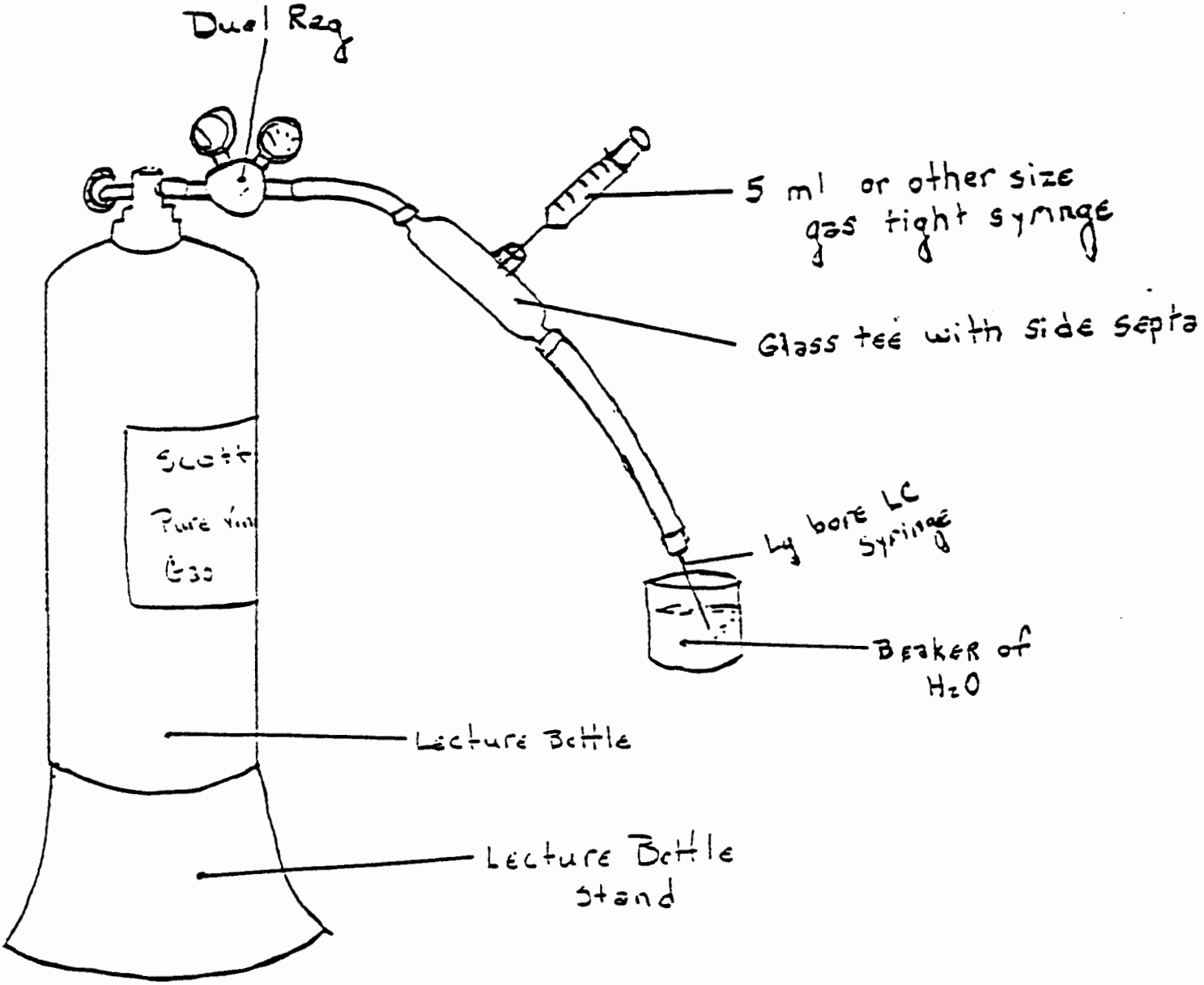
5.6.1 When making a standard in methanol, follow this procedure.

5.6.1.1 Set up the lecture bottle for your target with a double or single control pressure regulator connected to a tee (see Figure 3), followed by a section of tubing and large bore needle.

5.6.1.2 Fill a 10 mL volumetric to the mark with methanol, and allow all surfaces above the meniscus to dry (about 10 minutes). Weigh flask and solvents; record weight.

5.6.1.3 Adjust flow from the needle immersed in water to just bubbling to purge the tee. Using a 5 mL gas-tight syringe draw out 5 mL of pure gas. Dispense this slowly just above the methanol (most gases will dissolve in methanol readily).

Figure 3


Dual Reg
5 ml or other size gas tight syringe
Glass tee with side septa
Lg bore LC syringe
Beaker of H_2O
Lecture Bottle
Lecture Bottle stand

5.6.1.4 Reweigh flask and contents and calculate concentration of stock.

5.6.1.5 Transfer the stock and subsequent dilutions into vials with zero head space and Teflon lined caps, and label appropriately. Store these standards in the refrigerator.

Notes: Set up the lecture bottle apparatus in the hood. The efficiency of this method can be calculated by using the ideal gas law.

5.6.2 When making up a gas standard in a solvent with low vapor pressure like carbon disulfide, use the following procedure.

5.6.2.1 Pipet a known volume of CS_2 into a screw top vial cap with a Teflon lined septum of "Mini-Nert" valve and refrigerate.

5.6.2.2 Set up the lecture bottle as directed in 5.6.1. Draw out 1 to 5 mL of pure gas with a gas tight syringe, and inject this into the vial of CS_2 through the septum of Mini-Nert valve. Using the ideal gas law calculate the concentration of the target in the CS_2.

For example, 5 mL dichlorodifluoromethane in 10 mL

$n = (1\ \text{atm}^{*})(0.005\ \text{L})/(0.08250\ \text{L-atm/deg-mole}^{+})(298\ ^{o}\text{K})$

$n = 0.0002034$ mole X 102.92 (MW) = 0.02093 g = 20.93 mg

Concentration = 20.93 mg/10 mL = 2.093 mg/mL = 2093 ug/mL

+ = 0.08205 factor is an adjustment for room temperature. vs. STP.
* = Can change for actual pressure

5.6.2.3 Transfer the stock and diluted working standards into zero head space vials with Mini-Nert values. Store these standards in the refrigerator.

5.7 Quality Control

5.7.1 Always have the standard calculations checked by another analyst before using the standard.

5.7.2 Run a reference standard as a sample against your standard curve. This standard should be 1) purchased from an approved source, 2) made by another analyst independently from another source of neat standard, 3) made independently from neat standard.

5.7.3 Analyze the new standard against the old if this is a replacement standard. Insert documentation of the results in the standard comparison log book.

5.7.4 Remake volatile stock standards every 3 months or sooner if there are signs of compound loss or degradation.

5.7.5 Remake volatile working standards every week or sooner if there are signs of compound loss or degradation.

5.7.6 Remake semi-volatile, pesticide, PCB, and other extractable stocks every six months or sooner if there are signs of compound loss or degradation.

5.7.7 Remake extractable working standards every week or sooner if there are signs of compound loss or degradation.

5.7.8 Always store standards at -20 ^{o}C +/- 2 ^{o}C

5.7.9 Standards are prepared and stored in the solvents listed in Table 1.

TABLE 1

SOLVENTS FOR PREPARATION AND STORAGE

Solution Class	Solvent
Volatiles	Methanol
Semivolatile (BNA)	1:1 Acetone/methylene chloride
Organochlorine pesticides and PCB's	Hexane
Phenoxy acid herbicides	Hexane
Organophosphate pesticides	Hexane
Alcohols and other water solubles	Water
Petroleum hydrocarbons	Freon 113

This table applies to calibration standards, surrogates, matrix spiking mixes, and internal standards.

ROSS Zone Analytical Services, Inc.	SOP Standard Operating Procedure	SOP No: GX-004-00 Initial Date: 2/1/91 Page 1 of 7 Date Revised:

Title:

Preventive Maintenance in the GC Laboratory

Prepared by:	Approved by:	Date:	QC Approval:
Lawrence D. Anderson	C. Caldwell	5/7/91	CJC

1.0 PURPOSE

To outline procedures to be followed for the maintenance and upkeep of the gas chromatographs (GC's) the detectors, and the autosamplers.

2.0 SCOPE

This SOP describes the process of periodic maintenance used on the instruments, detectors, and autosamplers to assure proper performance and quality data.

3.0 REFERENCES

3.1 RASI Laboratory Quality Assurance Manual.

3.2 Varian 3400 Gas Chromatograph Operator's Manual, Vol. II.

3.3 Instruction Manual for Model P1-52-02 Photoionization Detector.

3.4 The HALL Book, Tracor Instruments.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Gas Chromatographs

5.1.1 The septum inside the GC injection port is to be changed after each project or at a time determined by the analyst so that the calibration of the instrument is within required specifications.

5.1.2 Each column inside a GC should be constantly watched during an analysis for possible deteriorating performance, such as excessive breakdown, high baseline, or non-resolution of target peaks. If such performance is detected, the column should be pulled from the GC, the head of the column repacked and the glasswool plug replaced for a packed column. If this type of performance is seen on a megabore column it usually means that the column should be replaced before other sample analyses takes place.

5.1.3 Supply gases are checked every day to make sure that flow to the GC's and detectors are at appropriate levels.

5.1.4 Gas line traps are changed when baseline levels and noise are excessive or when determined by the analyst that calibration is outside required specifications.

5.2 Detectors

5.2.1 Electron Capture Detectors

Preventive maintenance on the ECD detector is difficult. However, if an ECD is performing poorly here are several suggestions. First try baking the detector out at about 400°C. This is normally all that is necessary to clean out a detector. If that doesn't work try running hydrogen gas through the lines and into the detector for about 3 minutes. Refer to Varian 3400 Operator's Manual, Vol. II.

5.2.2 Flame Ionization Detector

FIDs collect a lot of soot from the burning process of the detector, so periodically (every 6 months) or sooner the FID is taken apart so the flame tip can be cleaned. The flame tip is first cleaned of soot and dirt with an emery board. A wire brush may help. Then sonicate the flame tip with methylene chloride, hexane, and methanol in that order. It is also a good idea to use emery paper on the electrodes going to the FID tower. Refer to Varian 3400 Operators Manual , Vol.II.

When assembling the detector install a new aluminum seal washer onto the shoulder of the detector base. This should be done each time the detector is installed. For best results, tighten the screws to the tower a little at a time so that equal pressure is applied to the seal. Refer to the Varian 3400 Operators Manual, Vol. II.

5.2.3 Photoionization Detector

CAUTION: When disassembling the detector, use disposable rubber gloves or tweezers to avoid contamination of internal components.

The PID is cleaned when the baseline level and the noise are excessive and when it is determined by the analyst that calibration is outside the required specifications. To clean the 10.2 eV UV lamp first turn off the lamp and line power. Remove the four lamp housing screws from the rim of the detector assemble and carefully remove lamp housing. Lift the light source from detector. Check the light source window for deposits, films, or discoloration by looking at a reflected image on the window. Then use a clean Kimwipe or other non-linting tissue dampened with water to remove any water soluble film. Next, add a drop of cleaning compound (supplied by HNU) to the window surface. Use a clean Kimwipe and scour the surface in a circular motion to remove film. Be careful no to scratch the window. Rinse

the window with an 80°C solution of mild dish washing liquid in water and rinse finally with 80°C distilled water. Dry the window in air or with lint free tissue before reassembling the detector. To reassemble the PID, position the disc and clamp window seal in place and place the lamp in position over the lamp window seal making sure the seal is properly positioned to insure against leaks. Carefully place the lamp housing over the lamp and insert and tighten the screws. Once reassembled, check the flowrate at the PID exhaust. Refer to PID instruction manual.

5.2.4 Thermioic Specific Detector

This detector is also known as the nitrogen-phosphorus detector. When the TSD exhibits low sensitivity or high background noise, cleaning is necessary. Visual inspection may also indicate a need for cleaning since the ceramic bead, collector, and flame tip are all parts that are prone to deposits.

Bead cleaning: deposits can be removed from the bead by gently scraping the bead surface with a sharp tool or abrasive paper. Support the bead while cleaning to prevent bending the lead and cracking the ceramic coating on the leads.

Flame Tip and Internal Parts Cleaning: handle internal parts with tweezers and place on a clean, uncontaminated surface. Clean deposits from the surface of the collector with water or emery paper. Remove deposits which form on top of the flame tip by scraping. If the flame tip is plugged, clear by inserting wire into flame tip orifice. Flush the cleaned components with methanol and air dry.

When reassembling the detector, install a new seal washer onto the shoulder of the base. This should be done each time the detector is installed. For best results, tighten the screws to the tower a little at a time so that equal pressure is applied to the seal. Refer to the Varian 3400 Operators Manual, Vol. II.

5.2.5 HALL Electrolytic Conductivity Detector

Preventative maintenance for the HALL detector consists of checking to make sure the solvent in the solvent reservoir is at the correct level. The flow from the solvent reservoir must also be checked periodically. A changing flow rate may indicate a leak or clog in the transfer lines. The flow rate should be about 0.5 ml/min. Refer to the HALL book.

5.3 Infrared Spectrophotometer

Freon can cause fogging of the two front plane mirrors. This fogging will cause excessive noise in the baseline and can be seen when an air check is performed. When this problem arises the mirrors must be cleaned with a cotton swab and methanol or deionized water with soap not containing lanolin.

5.4 Autosamplers

5.4.1 CTC A200S Autosamplers

Close attention is paid to an autosampler when high levels of targets are passed through it to see that no cross-contamination is taking place. Corrective action is taken if such a situation arises. The first injection made by the autosampler is always watched to see if the injection sequence is working properly.

5.5 Preventative Maintenance Records

5.5.1 Records of preventative maintenance and repairs must be kept in all situations. For this, a logbook has been designed and placed beside each instrument. If an instrument has two detectors that can be

used simultaneously, one logbook will accompany that instrument.

5.5.2 The check list (Figure 1) is filled out by first designating the instrument ID such as V, or A, or K. Next, the week the check list is kept is recorded. The top portion of the list is a quick reference to the availability of the instrument. If, for example, an analyst changes the septum and replaces the gas line trap on the instrument, the analyst initials the box pertaining to the action that day.

5.5.3 The bottom portion of the list is filled out when routine maintenance and repairs are made to the instrument. A detailed description of the action performed on the instrument is written down with the analyst's initials and date(s) when the repairs took place.

5.5.4 Preventative maintenance documentation is reviewed monthly by the Group Leader or Team Leader.

WEEKLY PREVENTATIVE MAINTENANCE CHECK LIST

INSTR. ID_____ WEEK OF _____

ITEM REQUIRED		Frequency	MON	TUE	WED	THU	FRI	SAT	SUN
CHANGE SEPTUM	FRONT	A,B,&							
	BACK								
CHANGE A/S SYRINGE		F,B,&							
ECD WIPE TEST		C							
CHANGE GAS TRAPS		B,E							
REPLACE COL PACKING		B,D							
CLEAN DETECTOR		B,D							
CLEAN IR MIRRORS		B,F							
MEOH CLEAN P/T		B,H							

A. AFTER EACH PROJECT
B. DETERMINED BY ANALYST SO THAT CALIBRATION IS ACCEPTABLE
C. ONCE EVERY THREE YEARS - ECD ONLY
D. WHEN BASE LINE LEVEL AND NOISE ARE EXCESSIVE
E. ONCE EVERY THREE MONTHS
F. IR ONLY
H. PURGE AND TRAP ONLY
&. WHICHEVER IS MORE FREQUENT

DATE	INT.	PREVENTATIVE MAINTENANCE / ROUTINE MAINTENANCE

DATE	INT.	REPAIRS MADE TO INSTRUMENT

ROSS	SOP Standard Operating Procedure	SOP No: GX-005-00 Inital Date: 2/1/91 Page 1 of 4 Date Revised:

Title:

Chlorinated Herbicide Analysis (EPA SW-846, Method 8150).

Prepared by:	Approved by:	Date:	QC Approval:
L. Anderson	CHCaldwell	6/3/91	CHC

1.0 PURPOSE

This SOP describes the GC/ECD analysis for chlorinated herbicides.

2.0 APPLICATION

This procedure provides guidelines to the determination of chlorinated herbicides.

3.0 REFERENCES

EPA Test Method 8150A (SW-846)

4.0 ASSOICATED SOPs

SOP No. OP-009-00, Extraction of Acid Herbicides from Solid Samples.

SOP No. OP-013-00, Extraction of Acid Herbicides from Water Samples.

5.0 PROCEDURE

5.1 Samples are analyzed using a Varian 3400 GC under the following conditions:

Detector: Electron Capture
Column: 30 m x .53 mm ID RTx-5 capillary column 1 micron film thickness.
Injector Temperature: 250°C
Detector Temperature: 320°C
ECD Range: 1

Approximate Gas Flows

Carrier Gas, Helium: 8 mL/min.
Make-up Gas, Argon/5% methane: 20 mL/min.

Temperature Program

Initial Temp: 160°C Hold: 2 min. Ramp: 8°C/min.
Final Temp: 280°C Hold: 3 min
Total Run Time: 20.0 min.

5.2 Stock Standards

Herbicide stock standard is bought from Supelco and is diluted 1:100 in hexane by putting 0.50 mL into 50.0 mL hexane. This stock contains the esters of 2,4-D, 2,4,5-TP (Silvex), and 2,4,5-T.

5.3 Working Standards

Prepare medium high, medium, medium low, and low level standards of each mix by diluting as below:

Standard	Dilution	Conc.
High	80 ul stock into 10.0 ml	0.16 ug/ml
Mid High	60 ul stock into 10.0 ml	0.12 ug/ml
Mid	40 ul stock into 10.0 ml	0.08 ug/ml
Mid Low	20 ul stock into 10.0 ml	0.04 ug/ml
Low	10 ul stock into 10.0 ml	0.02 ug/ml

5.4 Surrogates

Triclopyr is used to monitor the performance for the extraction, cleanup (when used), the analytical system, and the effectiveness of the method. Triclopyr must be derivitized as a sample to calculate a response factor. Recoveries are calculated by the equation:

$$\%R = \frac{X}{T}$$

Where,

X = measured concentration value found.

T = true value spiked.

5.5 Calibration

A five level curve is run first in the sequence, followed by blanks, then samples. A continuing calibration check is run every ten samples. This calibration check can be any level of calibration standards. The percent difference between the areas of the analytes of the first standard and the continuing check must be within 15%. If one of the analytes is not in calibration and that analyte is contained in the sample(s) that followed that calibration check, a new calibration check must be run followed by any samples that contained the analyte(s). If a calibration check fails a second time then a new calibration curve must be run for that analyte.

5.6 Analysis

Targets are located in the sample chromatogram by comparing the retention times of the suspect peaks to the retention data from the standards. The concentrations of analyte are determined by following these calculations:

Solid Matrix:

$$\text{mg/Kg} = \frac{\text{Area Counts (sample)}}{\text{RF}} * \frac{\text{final vol.}}{\text{sample wgt. (g)}}$$

Water:

$$\text{mg/L} = \frac{\text{Area Counts (sample)}}{\text{RF}} * \frac{\text{final vol.}}{\text{sample vol. (ml)}}$$

Where:

$$\text{RF} = \frac{\text{Area count (std)}}{\text{std. conc.}}$$

The concentrations of the ester must be converted to the concentration of the acid because of the different molecular weights. The ratio of the molecular weights of the acid to the ester is multiplied to the ester result as shown in the equation below:

$$\text{ug/L ester} * \frac{\text{acid molecular wt.}}{\text{ester molecular wt.}} = \text{ug/L acid}$$

Compound	Conversion factor (acid MW/ester MW)
2,4-D	0.948
2,4,5-T	0.940
2,4,5-TP	0.951

6.0 QUALITY CONTROL

This method requires the laboratory to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of lab capability and the analysis of spiked samples as a continuing check on performance. These records of initial laboratory capability and analyst ability are on file in the QA Coordinator's office.

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: Chlorinated Herbicide Analysis

SOP Number: GX-005-00

Date change implemented : 5/7/91

Change initiated by: ABA

Operational approval:

QA Approval:

Section to be changed: 5.4 Surrogates

Change(s): 2,4-DB (4(2,4-Dichlorophenoxy)Butyric Acid) is used to monitor the performance for the extraction, cleanup, the analytical system and the effectiveness of the method.

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: Chlorinated Herbicide Analysis

SOP Number: GX-005-0D

Date change implemented : 8/7/91

Change initiated by: QOA

Operational approval:

QA Approval:

Section to be ~~changed~~ ADDED: Practical Quantitation Limits Section 5.7

(1) Change(s): PQL's are listed below for water (assuming 1.0 liter extraction amount), Soils (assuming 50 g extract amount), and ~~TECP~~ TCLP samples (50.0 mL extraction amount).

PQLs (ug/L or ug/Kg)

	H_2O	Soil	TCLP
2,4-D	12.0 ug/L	240.0 ug/Kg	240.0 ug/L
2,4,5-TP	0.17	34.0	34.0
2,4,5-T	0.20	40.0	40.0

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: Chlorinated Herbicide Analysis

SOP Number: GX-005

Date change implemented : 5/1/92

Change initiated by: CCaldwell

Operational approval: CCaldwell

QA Approval:

Section to be changed: 3.0 Add: "Methods for Organochlorine Pesticides and Chlorophenoxy acid Herbicides in Drinking Water ..." p. 20-35

Change(s):

5.5 Add: Calibration curves for drinking water analyses must be validated with a standard mix of different origin than that of the curve. Agreement (RPD) must be within 15%

ROSS ANALYTICAL SERVICES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

Approved by	Date

Terminated by	Date

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title: Documentation of Run Logs for GC Analysis			
Prepared by: L. Anderson	Approved by:	Date: 6/8/95	QA Approval:

1.0 PURPOSE

The purpose of this procedure is to assist in the documentation of instrument run logs in the GC Lab.

2.0 APPLICATION

This procedure applies to all GC analysis.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Almost all analysis are performed using a automated sequence from the CHROM PERFECT ANALYTICAL DATA SYSTEM. These sequence tables are used by a program that searches for the data files and formats the data into instrument run logs.

5.2 A copy of an instrument run log is found in Section 6.0, Figure 1. File location of the raw data is given along with the date and time of the injection. The method name used to process the data is stated as well as the type of analysis. The injection names and injection-specific information such as weights, dilutions, amount injected, and internal standard amounts are also listed.

5.3 Analysts must generate these instrument run logs every day and have them checked by another analyst for accuracy and completeness. All instrument sequences and raw data are backed up on tape and could be retrieved to duplicate the run logs, if necessary.

5.4 If the analysis is not collected using a sequence, there are forms available in the GC lab to fill out a run log by hand. A copy of the form is found in Section 6.0, Figure 2.

6.0 FIGURES

Figure 1

Example - Automated Instrument Run Log

```
Sequence: C:\CP\DATA1\G0510.SEQ Data Directory: C:\CP\GH\
Date & Time Printed: 05-12-1995 17:53:06   Analyst: GJN
=================================================================
BOX 5-1 = ECD Instrument V3400H: Rtx-5; 30M x 0.32mm ID
          Carrier: Helium   Make-up: Argon/Methane

BOX 5-1 = ECD Instrument V3400H: Rtx-5; 30M x 0.32mm ID
          Carrier: Helium   Make-up: Argon/Methane

g0510.b,gpest.m,GJN,1,2,0,1000,1000
g0510b.b,gpestb.m,GJN,1,2,0,1000,1000
=================================================================
File: G0510.01R     Analysis Date & Time: 05-10-1995 23:40:34  Box: 5-0
Method: GPEST.MET    CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  1
Sample: PIBLKNG,PIBLKNG,1,1,,,3,,INSTBLANK,,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
-----------------------------------------------------------------
File: G0510B.01R    Analysis Date & Time: 05-10-1995 23:40:34  Box: 5-1
Method: GPESTB.MET   CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  1
Sample: PIBLKNG,PIBLKNG,1,1,,,3,,INSTBLANK,,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
=================================================================
File: G0510.02R     Analysis Date & Time: 05-11-1995 00:20:16  Box: 5-0
Method: GPEST.MET    CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  2
Sample: PEMNE,PEMNE,1,1,,,3,,PEM,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
-----------------------------------------------------------------
File: G0510B.02R    Analysis Date & Time: 05-11-1995 00:20:16  Box: 5-1
Method: GPESTB.MET   CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  2
Sample: PEMNE,PEMNE,1,1,,,3,,PEM,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
=================================================================
File: G0510.03R     Analysis Date & Time: 05-11-1995 01:01:38  Box: 5-0
Method: GPEST.MET    CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  3
Sample: GPC#02D BLANK,GPC#02D BLANK,1,,,,3,,QCCHECK,7,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
-----------------------------------------------------------------
File: G0510B.03R    Analysis Date & Time: 05-11-1995 01:01:38  Box: 5-1
Method: GPESTB.MET   CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  3
Sample: GPC#02D BLANK,GPC#02D BLANK,1,,,,3,,QCCHECK,7,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
=================================================================
File: G0510.04R     Analysis Date & Time: 05-11-1995 01:39:37  Box: 5-0
Method: GPEST.MET    CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  4
Sample: GPC#02D PEST,GPC#02D PEST,1,,,,3,,GPCCAL,7,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
-----------------------------------------------------------------
File: G0510B.04R    Analysis Date & Time: 05-11-1995 01:39:37  Box: 5-1
Method: GPESTB.MET   CLP ORGANO-CHLORINE PESTICIDE ANALYSIS    Cycle:  4
Sample: GPC#02D PEST,GPC#02D PEST,1,,,,3,,GPCCAL,7,,,
Weight: 1000.000  Inject:    2.000  Dilution:    1.000  IS Amt:    0.000
=================================================================
          Sequence: C:\CP\DATA1\G0510.SEQ   Page  1  of  7
```

Analyst _____ Reviewer _____

Logbook Page: 574

Figure 2

Example - Manual Instrument Run Log

INSTRUMENT LOG

CLIENT _____ ANALYSIS _____ DATE _____

W.O. # _____ ANALYST _____ CHECKER _____

INJECTION VOLUME _____ INSTRUMENT _____ DETECTOR _____

CLIENT ID	FILENAME	INJECTION TIME	NOTES

Ross Analytical Services, Inc.
STANDARD OPERATING PROCEDURE

Title:	Determination of Volatile Organic Compounds by GC/MS by Methods 8240B and 8260A			
Prepared by:	Approved by:	Date: 10/21/96	QA Approval:	

1.0 PURPOSE

The purpose of this procedure is to describe the determination of volatile organic compounds using GC/MS.

2.0 APPLICATION

This procedure may be used for the analysis of water, sediment, soil, sludge, and waste samples for the organic compounds listed in Table 1 and other related compounds. Information from SW-846, Method 8240B and 8260A was used to design this procedure.

This method is divided into the following sections: sample preparation including medium level extraction, screening, and analysis. The screening section contains a technique for estimation of levels of organic materials present in samples. The analysis section contains the GC/MS analytical methods for organics. The purge and trap technique, including related sample preparation, is included in the analysis section. Interferences are described in Section 7.4.

Most RCRA/CERCLA VOA analyses, except those done by CLP methodology, are performed using this method.

3.0 REFERENCES

3.1 EPA SW-846, 3rd Edition, Update II, September 1994, Methods 8240B and 8260A

3.2 RASI Laboratory Quality Assurance Manual

4.0 ASSOCIATED SOPs

MV-006, Analysis of Low Level Purgeable Organic Compounds by GC/MS Using Method 8260A

QC-001, Nonconformance and Corrective Action

QC-006, GC/MS Data Review and Validation

QC-013, Analyst Proficiency Requirements

5.0 EQUIPMENT/APPARATUS

5.1 Micro syringes - 10 uL and larger, 0.006 inch ID needle

5.2 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device

5.3 Syringe - 5 mL, gas tight with shut-off valve

5.4 Balance - Analytical, capable of accurately weighing ±0.0001 g and top-loading balance capable of weighing ±0.1 g

5.5 Glassware

5.5.1 Water purge vessels

5.5.2 Soil purge vessels

5.5.3 Bottle - 15 mL, screw cap, with Teflon cap liner

5.5.4 Volumetric flasks - Class A with ground glass stoppers

5.6 Purge and trap

The purge and trap device consists of a Tekmar model ALS2016 autosampler interfaced with a Tekmar model LSC2000 thermal desorber.

5.6.1 The sample sparger for water samples is designed to accept 5 mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less then 3 mm at the origin. The sample purger, illustrated in Figure 1, meets these design criteria. These purge vessels are available from Tekmar. Alternate sample purge devices may be utilized provided equivalent performance is demonstrated. An example of a suitable impinger for low level soil samples is illustrated in Figure 2.

For low level soil samples and some water samples, the purge vessel is covered with a heated jacket capable of maintaining the samples at 40°C ±1°C. If a heated purge is utilized, all standards, blanks, LCSs, and samples are heated, too.

5.6.2 The trap must be at least 25 cm long with an inside diameter of at least 0.105 inch. RASI uses a Vocarb 3000 or a OI Trap 10 multibed purge trap for volatiles. See Figure 3 for product information.

5.6.3 The desorber should be capable of rapidly heating the trap to 240°C. The trap should be able to be heated to 250°C during bakeout mode. The desorber schematic, illustrated in Figure 4, meets these criteria.

5.6.4 The transfer line from the purge and trap unit is coupled directly to the injection port of the gas chromatograph.

5.7 GC/MS

5.7.1 Gas chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

5.7.2 Column - A 75 m x 0.53 mm DB-624 fused silica megabore column, with a 3 um mid polarity, stationary phase.

5.7.3 Mass spectrometer - Scanning from 35 to 300 AMU every second utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the following criteria in Table 2 when 50 ng of p-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

5.7.4 GC/MS interface - A glass jet separator is used to interface the gas chromatograph and mass spectrometer.

5.7.5 Data system - A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is also available to integrate the abundance in any EICP between specified time or scan number limits.

6.0 STANDARDS AND SOLUTIONS

6.1 Reagents

6.1.1 Reagent water

Reagent water is taken from a 5 gallon glass bottle of LabConco-purified, nitrogen-purged water.

6.1.2 Methanol

B&J Purge and Trap Methanol

6.2 Standards

Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at -10 to -20°C in screw-cap amber bottles with Teflon liners.

6.2.1 Stock standard solutions

Stock standard solutions are purchased commercially.

Prepare fresh working standards every week for gases or for reactive compounds such as styrene. All other stock standards must be replaced after three months or sooner if comparison with check standards indicates a problem.

6.2.2 Calibration standards

Prepare daily calibration standards from the working calibration standards at five concentration levels or at the mid-level, if checking a continuing calibration, in methanol for each target parameter and each surrogate. The concentration levels are 10, 20, 50 (continuing calibration level),100, and 200 ug/L."

6.2.3 Surrogate standard spiking solution

Stock standard solutions for toluene-d8, 4-bromofluorobenzene (BFB), and 1,2-dichloroethane-d4 in methanol are also purchased commercially at concentrations of 2500 ug/mL.

A secondary dilution standard is prepared at 50 ug/mL. The addition of 5 uL of this standard to each 5 mL sample yields a surrogate concentration of 50 ug/L.

6.2.4 Internal standard spiking solution

Stock standard solutions of bromochloromethane, 1,4-difluorobenzene and chlorobenzene-d_5 in methanol are purchased commercially at 2500 ug/mL.

A secondary dilution standard is prepared at a concentration of 50 ug/mL containing each internal standard compound. The addition of 5 uL of this standard solution to 5 mL of sample or calibration standard yields a internal standard concentration of 50 ug/L.

6.2.5 Matrix Standard and Laboratory Control Sample Spiking Solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 50 ng/uL:

Matrix spike compounds
1,1-Dichloroethene
Trichloroethene
Chlorobenzene
Toluene
Benzene

Matrix spikes also serve as duplicates; therefore, add an aliquot of this solution to each of two portions from one sample chosen for spiking. It also serves as the laboratory control standard.

6.2.6 BFB Standard

Prepare a 50 ng/uL solution of BFB in methanol.

7.0 PROCEDURE

7.1 Sample Storage and Holding Times

7.1.1 Procedure for Sample Storage - The sample must be protected from light and refrigerated at 4°C (± 2°C) from the time of receipt until analysis. Samples are stored in a refrigerator used solely for volatiles samples.

7.1.2 Required Holding Times - The holding time for aqueous and non-aqueous samples alike is 14 days from date of collection. Aqueous samples must be preserved with HCl. If samples are not preserved with HCl, data generated from sample analyses must be flagged as being estimated values.

It is common belief that aqueous samples have a seven day holding time, if not preserved. This is not supported by the EPA methods.

7.2 Purging of Water Samples

An inert gas is bubbled through a 5 mL sample contained in a specially designed purging chamber (Figure 1) at either ambient temperature or at 40°C +/- 1°C. If a heated purge is used, all standards, blanks, LCSs, and samples are processed at 40°C +/- 1°C. The purgeables are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column (Figure 4) where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging. All water samples are, by definition, low level.

7.3 Purging of Soil/Sediment and Nonaqueous Waste Samples

7.3.1 Low level

An inert gas is bubbled through a mixture of a 5 g sample (weighed to the nearest 0.1 g) and 10 mL reagent water contained in a specially designed purging chamber (Figure 2) at 40°C. The purgeables are transferred from the aqueous phase to the vapor phase, and the analysis is continued as in 7.2.

7.3.2 Medium level

A measured amount of solid (4 g $\pm$ 0.1 g) is slurried with 10 mL of methanol. An aliquot of the methanol supernatant is added to 5 mL of reagent water. An inert gas is bubbled through this solution in the water purging chamber (Figure 1) at ambient temperature. The purgeables are transferred from the aqueous phase to the vapor phase, and the analysis is continued as in 7.2.

7.3.3 Nonaqueous liquids

Nonaqueous liquids are diluted in methanol to an appropriate concentration. An aliquot of the methanol is added to 5 mL of water in the purge chamber and analyzed.

7.4 Interferences

7.4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. If methanol dilutions are to be analyzed, a methanol blank must also be analyzed. No samples can be analyzed until the system is demonstrated to be free of contamination.

7.4.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling. A holding blank prepared from reagent water and carried through the holding period and the analysis protocol serves as a check for such contamination. One holding blank per week should be analyzed. Data are reviewed by the Quality Assurance Manager.

7.4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sampling syringe must be

rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

The laboratory where volatile analysis is performed should be kept as free of solvents as possible.

7.5 Purge Device and GC/MS Operating Conditions

Assemble the purge and trap device and connect it to the injection port of the gas chromatograph. Condition the trap at 250°C in the purge mode with an inert gas flow of at least 40 mL/minute.

Gas chromatograph conditions are as follows:

Temperature program:	-10°C initial temperature. Hold for 3 minutes then ramp to 240°C at 8°C/minute.
Injection port temperature:	220°C
Purge flow:	40 mL/minute
Carrier gas:	Helium
Desorb (carrier) flow:	10 mL/minute
Interface oven temperature:	250°C

Mass spectrometer conditions are as follows:

Electron energy:	70 volts (nominal)
Mass range:	35-300
Scan time:	To give at least 5 scans per peak and not to exceed 1 second per scan

7.6 Calibration

7.6.1 Initial Calibration

7.6.1.1 Tune the GC/MS system to meet the tune criteria in Table 2, Section 9.0, by injecting BFB. Analyze each calibration standard, adding 5 uL of internal standard spiking solution directly into the syringe. Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate response factors (RF) for each compound using Equation 1.

Eq. 1 $$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the internal standard

C_x = Concentration of the compound to be measured

7.6.1.2 The average response factor (RFi*) must be calculated for all compounds, including surrogates, using the 5 RF values calculated for each compound from the initial (5 point) calibration. The average response factor is calculated using Equation 2.

Eq. 2 $$RFi^* = \frac{\Sigma\,(RFi) \text{ from 1 to 5}}{5}$$

Where,

RFi* = Average response factor for compound i
RFi = Initial response factor for compound i
Note: 5 is for the five different levels

A system performance check must be made before this calibration curve is used. Five system performance check compounds (SPCCs) are checked to ensure that a minimum response factor is achieved. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average response factor is 0.300 for all SPCCs except bromoform whose factor must be greater than 0.10. These compounds are used to check for both compound instability and for degradation caused by contamination in lines or actives sites in the system.

Examples of possible problem issues include the following:

Bromoform is one of the compounds which is most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly effected by the turning of BFB at ion m/z 174/176. Increasing the m/z 174/176 ratio to m/z 95 ratio may improve bromoform response.

Chloromethane is the most likely compound to be lost if the purge flow is too fast.

Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

7.6.1.3 Using the average response factors (RFi*) from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all compounds. Calculate the %RSD using Equation 3.

Eq. 3 $$\%RSD = SD/RFi^* \quad * 100$$

Where,

SD = Standard deviation of the average response factors for compound i
RFi* = Same as in 7.6.1.2

The %RSD for each individual calibration check compound (CCC) must be less than 30%. The CCCs are listed below,

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene, and
Vinyl chloride.

If a %RSD greater than 30% is measured for any one CCC, then corrective action to eliminate a system leak and/or column reactive sites is required before reattempting calibration.

7.6.1.4 Linearity

7.6.1.4.1 If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation.

Or,

If the %RSD of any compound is 15% or less, you may construct a calibration curve using a first or second order regression fit of the five calibration points.

However,

7.6.1.4.2 If the %RSD of any compound is greater than 15%, construct a calibration curve using a first or higher order regression fit of the five calibration points for that compound. Select the lowest order regression which yields a coefficient of determination (r^2) of ≥ 0.99; however, note that there may be a limited number of compounds which have a lower r^2 value and the curve remains acceptable..

In practice, if the %RSD of any compound is greater than 15%, all compounds are quantitated from a regression fit curve.

7.6.2 Daily GC/MS Calibration

7.6.2.1 A check of the calibration curve must be performed once every 12 hours. This standard contains 50 ug/L of all of the analytes of concern. The minimum response factors for the system performance check compounds chloromethane, 1,1-dichloroethane, 1,1,2,2-tetrachloroethane, and chlorobenzene must be 0.300 (0.10 for bromoform). If these criteria are met, the response factor of all compounds are calculated and reported.

7.6.2.2 Before sample analysis begins, the percent drift of the continuing calibration check compounds (CCCs) is calculated using Equation 4 listed below. The CCCs are1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride.

Eq. 4 % Drift (%D) = $\frac{C_I - C_C}{C_I} \times 100$

Where:

C_I - Calibration check compound standard concentration

C_C - Measured concentration

The maximum percent drift allowed for each CCC is 20%. If this criterion is not met (>20% percent drift) for any one CCC, corrective action must be taken. If no source of problem can be determined after corrective action has been taken, a new five point calibration must be generated. This criterion must be met before quantitative sample analysis begins.

NOTE: If no CCCs are required target analytes, then only the required analytes must meet the maximum 20% drift criterion.

Only after both the minimum response factors and %D criteria are met can sample analysis begin. Though the 20% maximum drift criterion does not always apply to all targets, if many targets have > 20% drift, this is a warning that system performance is deteriorating.

7.6.2.3 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hours) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer system must be inspected for malfunction and correction made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

7.7 Sample Analysis

7.7.1 Water Samples

7.7.1.1 The holding time for water samples 14 days from sample collection, if preserved. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.7.1.2 Operating conditions for the gas chromatograph are listed in Section 7.5.

7.7.1.3 After achieving the key ion abundance criteria using BFB, calibrate the system with either the initial calibration procedure or continuing calibration procedure as appropriate.

7.7.1.4 Adjust the purge gas (helium) flow rate to 40 mL/minute. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

7.7.1.5 Remove a 5 mL syringe barrel from the oven and allow to cool to room temperature. Open the sample, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the sample for future analysis so if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as when the analyst has determined that the first sample has been analyzed properly. Filling one 20 mL syringe would allow the use of only one syringe. If a second analysis is needed from the 20 mL syringe, it must be analyzed within 24 hours. Care must also be taken to prevent air from leaking into the syringe.

7.7.1.6 Previous analyses, screening, or previous experience with a sample may indicate that a dilution is necessary. Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

Calculate the approximate volume of reagent water which will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

Inject the proper aliquot from the syringe into the volumetric flask. Dilute the flask to the mark with reagent water. Cap the flask, invert, and shake three times.

Fill a 5 mL syringe with the diluted sample.

If this is an intermediate dilution, use it and repeat the above procedure to achieve larger dilutions.

7.7.1.7 Add 5 uL of the surrogate spiking solution (Section 6.2.3) and 5 uL of the internal standard spiking solution (Section 6.2.4) through the valve bore of the syringe, then close the valve.

Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber.

7.7.1.8 Close both valves and purge the sample for 11.0 $\pm$ 0.1 minutes at ambient temperature.

NOTE: Water samples may be analyzed using a heated (40°C) purge provided that data are on file demonstrating equivalent or superior performance and that standards, blanks, LCSs, and samples are analyzed using this method.

7.7.1.9 At the conclusion of the purge time, switch the device to the desorb mode, and begin the gas chromatograph temperature program. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 240°C while backflushing the trap with helium at 10 mL/minute for two minutes.

7.7.1.10 While the trap is being desorbed into the gas chromatograph, empty the purging chamber of operating in manual mode. Wash the chamber with a minimum of two 5 mL flushes of reagent water to avoid carryover of pollutant compounds.

7.7.1.11 After desorbing the sample for two minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 250°C. After approximately ten minutes, turn off the trap heater open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.7.1.12 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the calibration curve.

7.7.1.13 For water samples to be matrix spiked, add 5 uL of the matrix spike solution (Section 6.2.5) to the 5 mL of sample purged. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike compound. Prepare MS/MSD pairs at the frequency described in Section 8.6.

7.7.1.14 At the conclusion of analysis, measure and record the pH of the sample to determine if it was properly preserved. <u>If a sample is found to be unpreserved, then flag data as being estimates</u>.

7.7.2 <u>Soil/Sediment/Nonaqueous Waste Samples</u>

7.7.2.1. Low Level Soil Method

The holding time for soil and other non-aqueous samples is 14 days. The low level soil method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards under the same conditions as the samples. A specially designed purge vessel is used for these analyses (See Figure 2). Screen the sample before analysis.

The GC/MS system should be set up as previously discussed. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and sample. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low level-method. Follow the initial and daily calibration instructions given, taking notice that a 40°C purge must be used.

To prepare the reagent water containing the surrogates and internal standards, remove the plunger from a 5 mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5 mL. Add 5 uL each of the surrogate spiking solution and the internal standard solution to the syringe through the valve. The addition of 5 uL of the surrogate spiking solution to 5 g of soil/sediment is equivalent to 50 ug/Kg of each surrogate standard.

The sample (for volatile organics) consists of the <u>entire</u> contents of the sample container (do not discard any supernatant liquids). Mix the contents of the sample container with a narrow metal spatula. Using a top loading balance, weigh an appropriate amount (0.5 -

5g) of sample into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

Add 5 mL of reagent water to the purge vessel, cap, shake to disperse the sample, and attach it to the ALS-2016 unit. Spike a syringe containing 5 mL of reagent water with internal standards and surrogates, and inject through the valve into the sample. Attach the heating jacket to the purge vessel in order to bring the sample to 40°C ± 1°C, then purge the sample for 11.0 ± 0.1 minutes.

Proceed with the analysis as for water samples. Use 10 mL of the sample reagent water for the reagent blank.

For matrix spikes of low level solids/sediments add 5 uL of the matrix spike solution to the 10 mL of water. The concentration for a 5 g sample would be equivalent to 50 ug/Kg of each matrix spike standard.

7.7.2.2. Medium Level Soil/Other Solids Method

The medium level method is based on extracting the sample with methanol. An aliquot of the methanol extract is added to the reagent water containing the surrogate and internal standards and purged at ambient temperature. All samples with a dilution factor greater than 10 (e.g., those which require an aliquot <0.5g) should be analyzed by the medium level method. If saturated peaks or concentrations exceeding the calibration range occurred or would occur when a 0.5 g sample was analyzed, the medium level method must be used.

The GC/MS system should be set up as previously discussed. This should be done prior to the addition of the methanol extract to reagent water. Initial and continuing calibrations are performed by adding standards in methanol to reagent water purging at ambient temperature.

NOTE: Samples may be analyzed using a heated (40°C) purge provided that data are on file demonstrating superior performance and that standards, blanks, LCSs, and samples are analyzed accordingly.

The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Using a top loading balance, weigh 4 g (wet weight) of sample into a tared 15 mL vial. Note and record the actual weight to the nearest 0.1 g.

Quickly add 9.0 mL of methanol, then 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 minutes. Note: Perform rapidly to avoid loss of volatile organics. Perform in a laboratory free of solvent fumes.

Using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be disposed of. Transfer approximately 1 mL of the reagent methanol to a GC vial for use as the method blank for each case or set of 20 or fewer samples, whichever is more frequent. These extracts may be stored in the dark at 4°C (±2°C) prior to analysis.

The volume of methanol added to the 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

Dilute an aliquot of the methanol extract if necessary and then take 100 uL for analysis.

Remove the plunger from a 5 mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5 mL to allow volume for the addition of sample and standards. Add 5 uL of the internal standard solution.

Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber. A water purge vessel is used for analysis of methanol dilutions (Figure 1).

Proceed with the analysis. Analyze all reagent blanks on the same instrument as the samples. The standards should also contain 100 uL of methanol to simulate the sample conditions.

For a matrix spike the medium level samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution, and 1.0 mL of matrix spike solution. This results in a 6,200 ug/Kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100 uL aliquot of this extract to 5 mL of water for purging.

7.8 Data Interpretation and Calculations

7.8.1 Qualitative Analysis

The compounds listed in the in Table 1 shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications:

(1) Elution of the sample component at the same GC relative retention time as the standard component,

(2) Correspondence of the sample component and standard component mass spectra.

7.8.1.1 For establishing correspondence of the relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

7.8.1.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the same GC/MS may be used for identification purposes, only if the GC/MS meets the daily tuning requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRTs.

7.8.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

The relative intensities of ions must agree within plus or minus 30 percentage points between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent).

Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives.

7.8.1.4 If a compound cannot be verified by all of the criteria above, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the analyst shall report that identification and proceed with quantification.

7.8.1.5 A library search is performed for non-target sample components for the purpose of tentative identification where requested. For this purpose, the 1990 release of the NIST Mass Spectral Library (or more recent release), containing approximately 50,000 spectra, is used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Up to 20 non-surrogate, non-target organic compounds of greatest apparent concentration are tentatively identified via a forward search of the NIST mass spectral library. (Substances with responses less than 10% of the internal standard are not required to be searched in this fashion.) Only after visual comparison of sample spectra with the nearest library searches does the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ± 20 percentage points. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

If in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights or carbon numbers can be distinguished, include them.

7.8.2 Quantitative Analysis

7.8.2.1 Target components identified shall be quantified by the internal standard method. The internal standards used are listed in Table 5. The EICP area of the characteristic ions of analytes listed in Tables 3 and 4 are used.

7.8.2.2 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and correction made as appropriate. When corrections are made, reanalysis of sample analyzed while the system was malfunctioning is necessary.

If after reanalysis, the EICP area for all internal standards are inside the required limits (-50% to +100% of that internal standard in the mid low standard analyses for that day), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, only submit data from the analysis with EICPs within the control limits.

If the reanalysis of the sample does not solve the problem, e.g., the EICP area are outside the control limits for both analyses report the initial analysis only and note the presence of matrix interferences.

7.8.2.3 Linearity is assumed for all targets which had ≤ 15% RSD in the initial calibration. For these targets, their average response factors may be used for quantitation (Section 7.8.3); otherwise, the calibration curve must be used (Section 7.8.4).

7.8.2.4 Xylenes (o,m, & p - isomers) are to be reported as xylenes (total). Since m- and p-xylene co-elute, the xylenes must be quantitated as m/p-xylene. The concentration of all xylene isomers must be added together to give the total.

1,2-dichloroethene (trans and cis stereoisomers) are usually reported as 1,2-dichloroethene (total). The concentrations of both isomers must be added together to give the total.

7.8.3 Calculations For Targets Whose %RSD ≤15%.

7.8.3.1 Water

Eq. 5 Concentration ug/L = $\frac{(A_x)(I_s)}{(A_{is})(RFi^*)(V_o)}$

Where:

A_x	=	Area of the characteristic ion for the compound to be measured.
A_{is}	=	Area of the characteristic ion of the specific internal standard from Table 4.
I_s	=	Amount of internal standard added in nanograms (ng).
V_o	=	Volume of water purged in milliliters (mL) (take into account any dilutions).
RFi*	=	Average response factor for the compound being measured.

7.8.3.2 Sediment/Soil Waste (medium level)

Eq 6. Concentration in ug/g (Dry weight basis) = $\frac{(A_x)(I_s)(V_t)}{(A_{is})(RFi^*)(V_i)(W_s)(D)}$

Note: For analyses to be reported on an as-received basis, omit the division by "D".

Where:

A_x, I_s, A_{is}, RFi*	=	Same as for water, above
V_t	=	Volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made)
V_i	=	Volume of extract added (uL) for purging
D	=	$\frac{100 - \%\ moisture}{100}$
W_s	=	Weight of sample extracted (g) or purged

Note: For analyses to be reported on an as-received basis, omit the division by "D".

An estimated concentration for non-target components tentatively identified is calculated by the internal standard method. For quantitation, the nearest internal standard free of interference is used.

The formula for calculating non-target concentrations is the same as above. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A average response factor (RFi*) of one is to be assumed. The value from this quantitation shall be qualified as estimated and this estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

Target analytes are usually reported to two significant figures if ≥ 10 ug/L or 10 ug/Kg. Targets present at <10 ug/L or ug/Kg, and tentatively identified compounds, are usually reported to one significant figure.

7.8.4 Quantitation For Targets Whose %RSD >15%

When a regression fit is used, the concentration is taken directly from the curve and multiplied by any dilution factor and divided by the percent solids.

8.0 QUALITY CONTROL

8.1 Tuning

8.1.1 The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant tune. The time period ends after twelve (12) hours has elapsed according to the system clock.

8.1.2 Tune to meet criteria as shown in Table 2.

Note: All instruments conditions must be identical to those used in sample analysis, except that a different temperature may be used.

8.1.3 BFB criteria must be met before any standards, samples, or blanks are analyzed. Any samples analyzed when tuning criteria have not been met will require reanalysis.

8.2 Calibration

8.2.1 After tuning criteria have been met but prior to the analysis of samples and required blanks, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound standards. Once the system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

8.2.2 Initial calibration of volatile target compounds is required at a minimum of 5 concentration levels (normal concentration levels are 10, 20, 50, 100, and 200 ug/L). This will result in 50-1000 total ng analyzed. Surrogate and internal standards are used with each of the calibration standards. Analyze all method blanks and standards under the same conditions as the sample.

8.2.3 System Performance Check Compounds (SPCC)

8.2.3.1 The initial calibration is valid only after the minimum response factors for SPCCs have been met. Only after both these criteria are met can sample analysis begin.

8.2.3.2 A calibration standard(s) containing all volatile target compounds, including all required surrogates, must be analyzed each twelve hours during analysis. The SPCCs must meet the minimum response factor criteria.

8.2.4 Calibration Check Compounds

After the system performance check is met, the %Drift of the CCCs listed in the following table must be checked to ensure the validity of the initial calibration.

Calibration Check Compounds

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

If the percent drift for each target compound is less than 20.0%, the initial calibration is assumed to be valid. If the criteria are not met (>20.0% drift), for any one calibration check compound, corrective action must be taken.

Problems similar to those listed under SPCC could affect these criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five point calibration must be generated. These criteria must be met before sample analysis begins.

If no CCCs are required target analytes, then only the required analytes must meet the 20% drift criterion.

8.3 Method Blanks

A method blank is a volume of deionized, distilled laboratory water which is carried through the entire analytical scheme. The method blank volume must be approximately equal to the sample volumes or sample weights being processed.

8.3.1 For the analysis of volatile target compounds, a method blank analysis must be performed once for each 12 hour time period. A method blank must always follow any standard analysis.

8.3.2 Per SW-846, an acceptable method blank should not be higher than the highest of either:

(1) The method detection limit, or

(2) Five percent of the regulatory limit for that analyte, or

(3) Five percent of the measured concentration in the sample.

However, per Method 8240B, the laboratory is permitted to adopt additional quality assurance practices for use with this method. As such, RASI has adopted the following industry-wide protocol for method blank acceptance. It is our belief that this protocol is achievable, understandable, and produces data which is acceptable.

A method blank for volatile analysis must contain less than five times (5X) the EQL of methylene chloride, acetone, and 2-butanone.

For all other target compounds, the method blank must contain less than the EQL of any single target analyte.

If a laboratory method blank exceeds these criteria, the analyst must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (e.g., contaminated) must be reextracted/repurged and reanalyzed. The result of blank analyses will be reported in text form.

All sample concentration data will be reported uncorrected for blanks.

8.4 Laboratory Control Sample (LCS)

8.4.1 A LCS is prepared by spiking a water blank with the spike solution described in Section 6.2.5.

8.4.2 A LCS will be analyzed at a frequency of once per tune.

8.4.3 LCSs must pass the acceptance criteria listed in Section 9.0, Table 6. If the LCS fails, perform corrective action and reanalyze.

8.5 Surrogates

Surrogate standard determinations are performed on all samples and blanks. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples.

Eq. 7 $\% \text{ Surrogate Recovery} = \frac{Q_d}{Q_a}$

Where:

Q_d = Quantity determined by analysis
Q_a = Quantity added to sample

8.5.1 Surrogate Recovery Limits Are As Follows:

REQUIRED SURROGATE SPIKE RECOVERY LIMITS

Surrogate Compound	Water	Low/Medium Soil
Toluene-d_8	88-110	81-117
4-Bromofluorobenzene	86-115	74-121
1,2-Dichloroethane	76-114	70-121

Surrogate spike recovery limits for TCLP leachates, and wastes are determined statistically from actual laboratory data, and are subject to change quarterly.

8.5.2 Method Blank Surrogate Spike Recovery

The laboratory must take the actions listed below if the recovery of any surrogate compound in the volatiles fraction of the method blank is outside of the required surrogate spike recovery limits.

Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; also check instrument performance.

Reanalyze the blank or extract if steps above fail to reveal the cause of the noncompliant surrogate recoveries.

If the blank is a methanol extract for medium level soil samples, reextract and reanalyze the blank if reanalysis alone fails to reveal the cause of the noncompliant surrogate recoveries.

If all measures above fail to correct the problem, the analytical system must be considered out of control. The problem must be corrected before continuing. This may mean recalibrating the instrumentation but it may also mean more extensive action. The specific corrective action is left up to the GC/MS operator. When surrogate recovery(ies) in the blank is outside of the required windows, all samples associated with that blank must be reanalyzed.

8.5.3 Sample Surrogate Spike Recovery

The laboratory must take the action listed below if the recovery of any one surrogate compound in the volatiles fraction of the sample is outside of the control limits.

Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; also check instrument performance.

If the above steps fail to reveal a problem, then reanalyze the sample or extract. If reanalysis of the sample or extract solves the problem, then the problem was within the laboratory's control. Therefore, <u>only</u> <u>submit</u> data from analyses with surrogate spike recoveries <u>within</u> limits.

If the sample was a soil extracted with methanol and the steps above fail to solve the problem, then reextract and reanalyze the sample. If the reextraction and reanalysis solves the problem, then the problem was instrumental. <u>Only submit</u> data from the extraction and analysis with surrogate spike recoveries <u>within</u> the control windows.

If the reextraction and/or reanalysis of the samples does not solve the problem; e.g., surrogate recoveries are outside the control limits for both analyses, then report the initial analysis and describe the confirmation in text.

If the sample with surrogate recoveries outside the limits is the sample used for the matrix spike and matrix spike duplicate, and the surrogate recoveries of the matrix spike and matrix spike duplicate show the same pattern (e.g., outside the limits), then the sample, matrix spike, and matrix spike duplication do <u>not</u> require reanalysis. Document in the narrative the similarity in surrogate recoveries.

8.6 <u>Matrix Spike/Matrix Spike Duplicate Analyses (MS/MSD)</u>

8.6.1 Frequency of Analysis

Perform an MS/MSD analysis on at least every 20th sample of each matrix type (water, soil, and waste). One TCLP (ZHE) leachate of each waste type (as determined by the client) is spiked with all targets. No MS/MSD pairs are performed on TCLP leachates.

8.6.2 Matrix Spike % Recovery - Calculate the percent recovery for each spike target using the equation below.

Eq. 8 Matrix Spike Percent Recovery = $\frac{SSR - SR}{SA} \times 100$

Where:

SSR = Spike sample result
SR = Sample result
SA = Spike added for spiking mix

If the percent recovery is outside the range listed in Section 9.0, Table 6, flag the data noting that matrix interferences are present in these samples.

8.6.3 Relative Percent Difference (RPD) - Calculate the RPD for each spike target using the equation below.

Eq. 9 $RPD = \frac{D_1 - D_2}{(\bar{D}_1 + \bar{D}_2)/2} \times 100$

Where:

RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value (duplicate)

If the RPD is greater than that listed in Section 9.0, Table 6, flag this in the narrative.

9.0 TABLES/FIGURES

Table 1

Normal Target Compounds
Estimated Quantitation Limits (EQLs)

Volatiles	CAS Number	Quantitation limits Water*, µg/L	Low soil/sediment*,** µg/Kg
Chloromethane	74-87-3	10	10
Bromomethane	74-83-9	10	10
Vinyl chloride	75-01-4	10	10
Chloroethane	75-00-3	10	10
Methylene chloride	75-09-2	5	5
Acetone	67-64-1	10	10
Carbon disulfide	75-15-0	5	5
1,1-Dichloroethene	75-35-4	5	5
1,1-Dichloroethane	75-34-3	5	5
1,2-Dichloroethene (total)	540-59-0	5	5
Chloroform	67-66-3	5	5
1,2-Dichloroethane	107-06-2	5	5
2-Butanone	78-93-3	10	10
1,1,1-Trichloroethane	71-55-6	5	5
Carbon tetrachloride	56-23-5	5	5
Vinyl acetate	108-05-4	20	20
Bromodichloromethane	75-27-4	5	5
1,2-Dichloropropane	78-87-5	5	5
cis-1,3-Dichloropropene	10061-01-5	5	5
Trichloroethene	79-01-6	5	5
Dibromochloromethane	124-48-1	5	5
1,1,2-Trichloroethane	79-00-5	5	5
Benzene	71-43-2	5	5
trans-1,3-Dichloropropene	10031-02-6	5	5
Bromoform	75-25-2	5	5
4-Methyl-2-pentanone	108-10-1	10	10
2-Hexanone	591-78-6	10	10
Tetrachloroethene	127-18-4	5	5
Toluene	108-88-3	5	5
1,1,2,2-Tetrachloroethane	79-34-5	5	5
Chlorobenzene	108-90-7	5	5
Ethyl benzene	100-41-4	5	5
Styrene	100-42-5	5	5
Xylenes (total)	1330-20-8	5	5

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediments are based on wet weights. The quantitation limits calculated on a dry weight basis will be higher than those listed.

Table 1 (Continued)

Additional Appendix IX Target Compounds
Estimated Quantitation Limits (EQLs)

Volatiles	CAS Number	Quantitation limits Water*, µg/L	Low soil/sediment*,** µg/Kg
Acetonitrile	75-05-8	10	10
Acrolein	107-02-8	20	20
Acrylonitrile	107-13-1	10	10
Ethyl methacrylate	97-83-2	5	5
Allyl chloride	107-05-1	5	5
Iodomethane	74-88-4	10	10
Methacrylonitrile	126-98-7	20	20
Methylene bromide	74-95-3	5	5
Methyl methacrylate	80-62-6	5	5
Propionitrile	107-12-0	20	20
1,1,1,2-Tetrachloroethane	630-20-6	5	5
Chloroprene	126-99-8	5	5
1,2-Dibromo-3-chloropropane	96-12-8	10	10
1,2-Dibromoethane	106-93-4	5	5
trans-1,4-Dichloro-2-butene	110-57-6	5	5
Dichlorodifluoromethane	75-71-8	10	10
Trichlorofluoromethane	75-69-4	5	5
1,2,3-Trichloropropane	96-18-4	5	5

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediments are based on wet weights. The quantitation limits calculated on a dry weight basis will be higher than those listed.

Table 2

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15 - 40 percent of the base peak
75	30 - 60 percent of the base peak
95	Base peak, 100 percent relative abundance
96	5 - 9 percent of the base peak
173	Less than 2 percent of mass 174
174	Greater than 50 percent of the base peak
175	5 - 9 percent of mass 174
176	Greater than 95 percent but less than 101 percent of mass 174
177	5 - 9 percent of mass 176

Table 3

Characteristic Ions For Surrogate and Internal Standards For Volatile Organic Compounds

Parameter	Primary ion	Secondary ion(s)
Surrogate standards		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane	65	120
Toluene d-8	98	70, 100
Internal standards		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene d-5	117	82, 119

Table 4, Characteristic Ions for Volatile Compounds

Parameter	Primary ion*	Secondary ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethane	96	61, 98
1,1-Dichloroethene	63	65, 83, 85, 998, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	72	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon Tetrachloride	117	119, 121
Vinyl acetate	43	86
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	-
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total xylenes	106	91
Dichlorodifluoromethane	85	85, 87, 101, 103
Acetonitrile	41	41, 40, 39
Methyl iodide (iodomethane)	142	142, 127, 141
Trichlorofluoromethane	101	103, 66
Propionitrile	54	54, 52, 55, 40
Allyl chloride	76	76, 41, 39, 78
Methacrylonitrile	41	41, 67, 39, 52, 66
Dibromomethane	93	93, 174, 95, 172, 176

* The primary ion should be used unless interferences are present; in which case, a secondary ion may be used.

Table 4, Characteristic Ions for Volatile Compounds (Continued)

Parameter	Primary ion*	Secondary ion(s)
Chloroprene	53	53, 88, 90, 51
1,2-Dibromoethane	107	107, 109, 93, 188
Methyl methacrylate	69	69, 41, 100, 39
1,1,1,2-Tetrachloroethane	131	131, 133, 117, 119, 95
1,2,3-Trichloropropane	110	75, 77, 112, 97
1,4-Dichloro-2-butene	75	75, 53, 77, 124, 89
Ethyl methacrylate	69	69, 41, 99, 86, 114
1,2-Dibromo-3-chloropropane	157	157, 75, 155, 77
Acrolein	56	55, 58
Acrylonitrile	53	52, 51

* The primary ion should be used unless interferences are present; in which case, a secondary ion may be used.

Table 5

Volatile Internal Standards With Corresponding Target Analytes Assigned For Quantitation

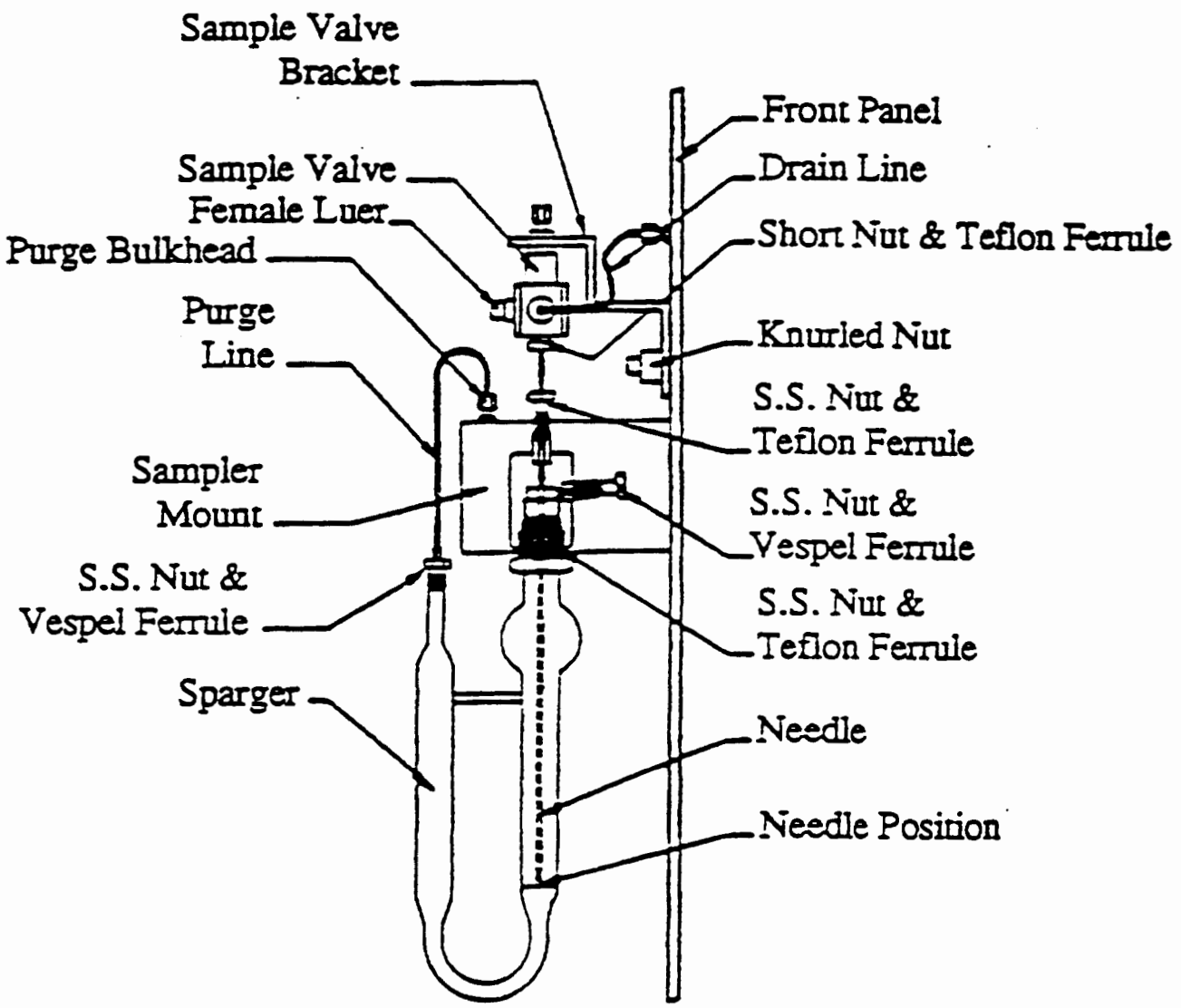
Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d5
Chloromethane	2-Butanone	2-Hexanone
Bromomethane	1,1,1-Trichloroethene	4-Methyl-2-pentanone
Vinyl Chloride	Carbon tetrachloride	Tetrachloroethene
Chloroethane	Vinyl acetate	1,1,2,2-Tetrachloroethane
Methylene chloride	Bromodichloromethane	Toluene
Acetone	1,2-Dichloropropane	Chlorobenzene
Carbon Disulfide	trans-1,3-Dichloropropene	Ethylbenzene
1,1-Dichloroethene	Trichloroethene	Styrene
1,1-Dichloroethane	Dibromochloromethane	Xylene (total)
1,2-Dichloroethene (total)	1,1,2-Trichloroethane	Bromofluorobenzene (surrogate)
Chloroform	Benzene	Toluene-d_8 (surrogate)
1,2-Dichloroethane	cis-1,3-Dichloropropene	Ethyl methacrylate
1,2-Dichloroethane-d_4 (surrogate)	Bromoform	1,2,3-Trichloropropane
Acrolein	Methacrylonitrile	Methyl methacrylate
Acrylonitrile	1,2-Dibromoethane	1,1,2-Tetrachloroethane
Dichlorodifluoromethane	Chloroprene	1,2-Dibromo-3-chloropropane
Iodomethane	Dibromomethane	
Trichlorofluoromethane		
Acetonitrile		
Proprionitrile		
Allyl chloride		

Table 6

Matrix Spike/LCS Recovery Limits

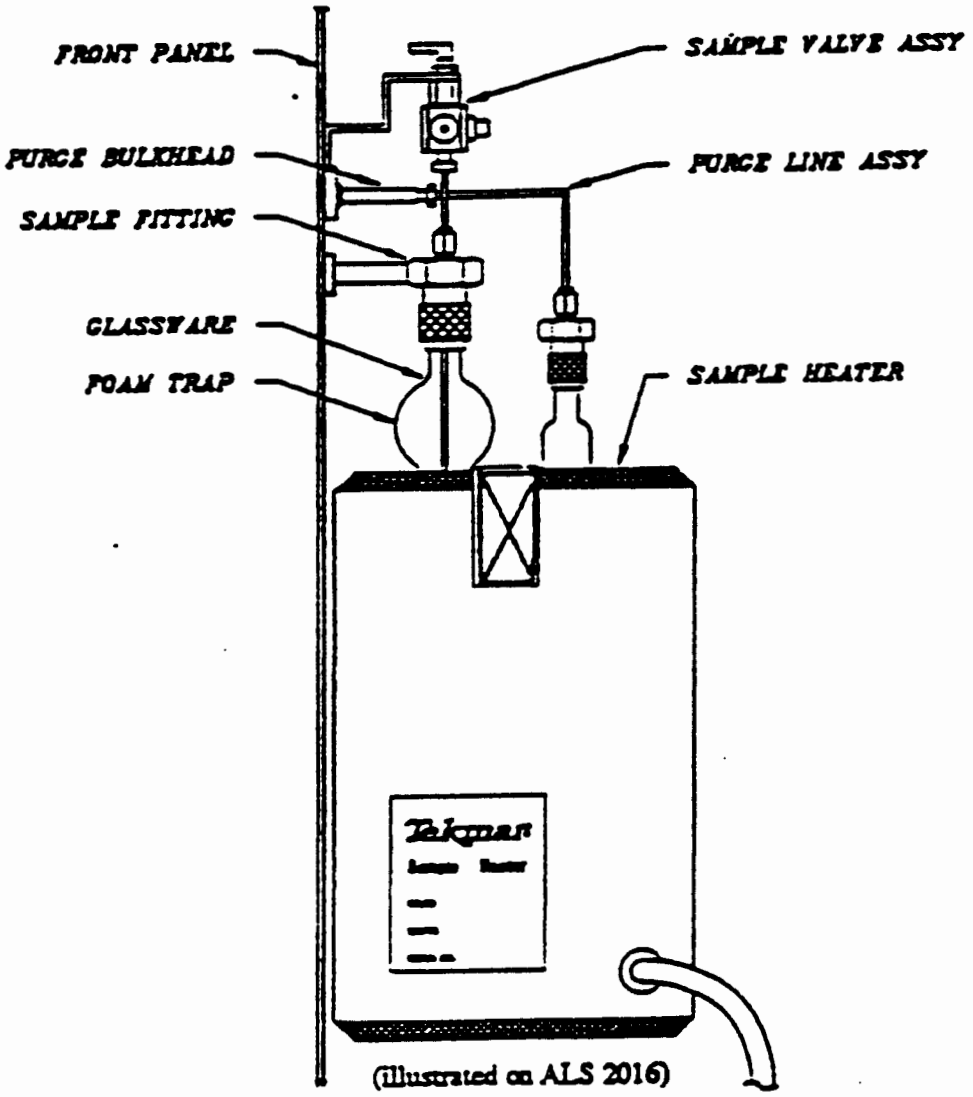
Matrix Spike Compound	Water/Soil	RPD
1,1-Dichloroethene	66-122	40
Trichloroethene	63-117	40
Chlorobenzene	69-127	40
Toluene	71-133	40
Benzene	68-126	40

Figure 1
Needle Sparger for LSC2000



Needle Sparger for the LSC 2000

Figure 2
Low Level Soils Impinger



(Illustrated on ALS 2016)

Figure 3
Trap Specifications (VOCARB 3000 Multibed Purge Trap for Volatiles)

SAVE THIS DATA SHEET!
It Contains Important Information About This Product.

Tekmar® Purge Trap

This trap was designed for use with Tekmar LSC-1, LSC-2, LSC-3, LSC-2000 and LSC-4000 purge and trap instruments. The Swagelok® nut attached to the trap has been stamped with a letter designation for identification. Use the chart below for future identification of this trap as well as other purge traps available from Supelco. Custom traps can be obtained through our Order Processing Department.

Trap	Trap Contents	Cat. No.
A	Tenax®	2-1058
B	Tenax/silica gel	2-1060
C	Tenax/silica gel/charcoal	2-1061
D	Tenax/charcoal	2-1062
E	SP™-2100/Tenax/silica gel/charcoal	2-0294
F	SP-2100/Tenax/silica gel	2-0293
G	SP-2100/Tenax	2-0295
H	Carbopack™ B/Carbosieve™ S-III*	2-0321
I (VOCARB™ 4000)	Carbopack C & B/Carboxen™ 1000 & 1001	2-0308
J (BTEXTRAP™)	Carbopack C & B	2-1064
K (VOCARB 3000)	Carbopack B/Carboxen 1000 & 1001	2-1066
Z	custom order	———

Installation

See instrument users manual for proper trap installation instructions. Since it is necessary to periodically replace the trap, you must use a removable ferrule on the inlet (top) end of the trap. We recommend 1/8" flexible graphite ferrules (e.g., Supelex™ M-4, Cat. No. 2-2491). Once you have installed the trap, check both fittings for leaks while operating in the purge mode and the desorb mode.

Operating Conditions

All traps must be preconditioned prior to use. This not only eliminates contamination, but also assures uniform performance during the lifetime of the trap. We recommend using conditions specified in the chart on the back of this form when setting time and temperature parameters. Purge times and sample volumes are dependent on compounds of interest and/or the specific method being followed.

Recommended Conditions
Temp. (°C) / Time (min.)

Trap	Desorb	Bake	Precondition
A	180/4	180/7	180/30
B	180/4	180/7	180/30
C	180/4	180/7	180/30
D	180/4	180/7	180/30
E	180/4	180/7	180/30
F	180/4	180/7	180/30
G	180/4	180/7	180/30
H	230/6	250/5	260/60
I (VOCARB 4000)	250/10	260/4	270/60
J (BTEXTRAP)	250/6	260/4	270/60
K (VOCARB 3000)	250/6	260/4	270/60

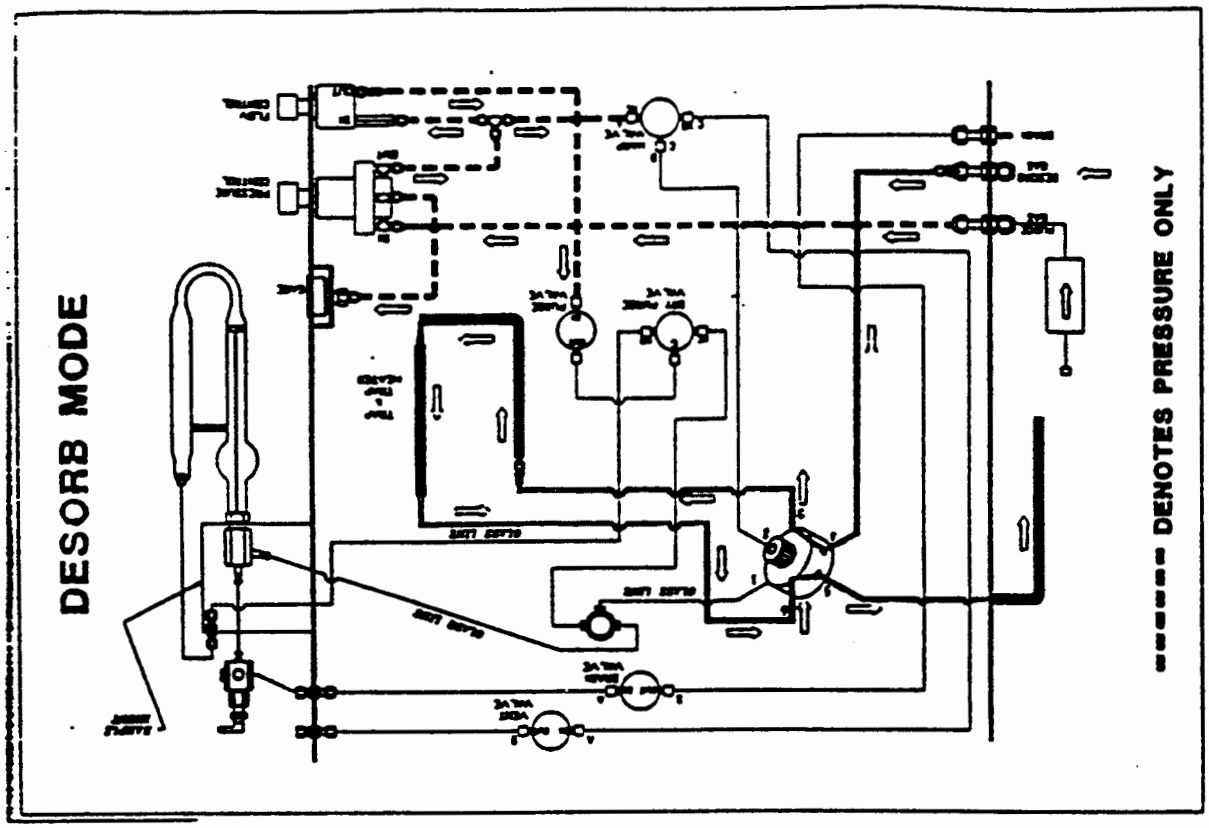
Trademarks

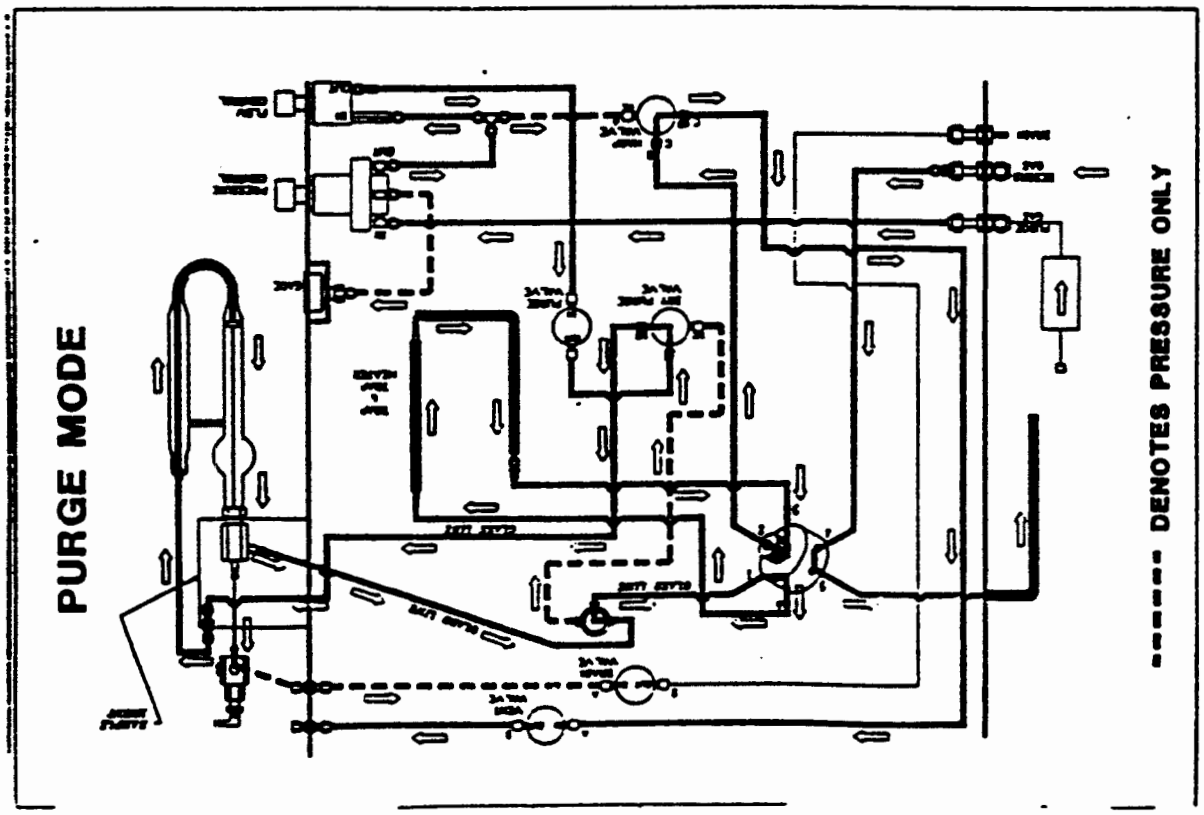
Carbopack — Supelco, Inc.
Carbosieve — Supelco, Inc.
Carboxen — Supelco, Inc.
SP — Supelco, Inc.
Supelex — Supelco, Inc.
Swagelok — Crawford Fitting Co.
Teknar — Teknar Co.
Tenax — Enka Research Institute Arnhem
VOCARB — Supelco, Inc.

*German Pat. No. 1935600. Patent holder — Badische Anilin-& Soda-Fabrik Aktiengesellschaft.

SUPELCO
Bellefonte, PA

Figure 4
Purge Mode, Desorb Mode Illustration





ROSS Roy Anderson Services, Inc.	SOP Standard Operating Procedure	SOP No: MV-003- Initial Date: 2/1/91 Page 1 of 10 Date Revised:

Title:

Preventive Maintenance in the Gas Chromatography/Mass Spectrometry Lab

Prepared by:	Approved by:	Date:	QC Approval:
Patricia Farrell	CHCaldwell	5/7/91	CHC

1.0 PURPOSE

This procedure is to be followed to prolong operation of the analytical instrumentation and to minimize and correct defects before they result in serious damage or failure.

2.0 APPLICATION

This procedure applies to the instruments used in the Gas Chromatography/Mass Spectrometry Lab.

3.0 REFERENCES

The manuals from the instrument's manufacturer are the source of the "How-to" details for performing preventive maintenance, routine maintenance, and repairs on all the instruments within the applicable laboratory. A library of instrument manuals is kept in the GC/MS lab.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Gas Chromatographs

5.1.1 Once a month dust off instrument externally and internally or when repairs are made.

5.1.2 Replace septa with new septa as warranted, when the injection port liner is replaced, or after 50 septum punctures, whichever is soonest.

5.1.3 Replace Injection Port Liner, if applicable, as warranted when the column is "clipped".

5.1.4 Cut about a foot off of a capillary column when the peak tailing becomes excessive. (See Figure 5)

5.1.5 Replace the capillary column when the chromatographic resolution of the column is no longer suitable for the application.

5.1.6 Check zone temperatures via the computer feedback daily or whenever the temperatures have been changed.

5.1.7 Check the operating gases daily for sufficient volume and pressure.

5.1.8 Check the injection port pressure/flow rate and any split flow rates after any connection change on a column, or at a minimum, once a month.

5.2 Autosamplers

5.2.1 Replace wash cup septa once a week or more often if warranted.

5.2.2 Dispose of waste solvent once a week or more often if warranted.

5.2.3 Perform the internal diagnostics instrument test on the Varian GCs one a month while visually checking the motion of the autosampler for proper action.

5.2.4 Visually check the injection syringe for leaks at the needle connection, daily.

5.3 Mass Spectrometers

5.3.1 General

5.3.1.1 Replace/clean all air filters once a quarter.

5.3.1.2 Dust off the instrument externally and internally once a month.

5.3.1.3 Check the operation of all cooling fans once a quarter.

5.3.2 Electronics

5.3.2.1 Dust off and reseat all printed circuit boards semi-annually or when any maintenance is performed on that sub-assembly, whichever is more often.

5.3.2.2 Reseat all cable connectors whenever any work is performed within the sub-assembly. Reseat all easily accessed cable connectors at least semi-annually.

5.3.2.3 Readjust RF resonance on the Finnigan MS after each source cleaning or whenever the front flange is removed from the manifold, whichever is more often.

5.3.3 Vacuum/Pneumatics System

5.3.3.1 Change the mechanical roughing pump oil semi-annually.

5.3.3.2 Check the oil level in the mechanical roughing pumps and the tubomolecular pumps monthly.

5.3.3.3 Purge the mechanical pumps monthly for about 15 minutes.

5.3.3.4 Check the condition of the manifold seals each time the flange is removed from the manifold.

5.3.3.5 Check the flexible tubing quarterly or when work is performed on the pumps, whichever is more often.

5.3.3.6 Check that the calibration gas valve and introduction system are functional weekly.

5.3.3.7 Leak check vacuum system if excessive pressures (> 0.025 TORR) are observed on the vacuum gauges or if excessive air is present in spectrum. When evaluating the air/water spectrum, a leak-tight system will have the N_2 peak (m/z 28) $\leq$ the water peak (m/z 18).

5.3.4 Data Systems

5.3.4.1 Clean heads on tape units quarterly.

5.3.4.2 Thoroughly clean printers including ribbon transport system semi-annually.

5.3.4.3 Replace printer ribbons as needed, to maintain enough contrast so that no special effort needs to be taken in reproducing copies of the output due to insufficient contrast.

5.3.4.4 Backup your system software at least quarterly, preferably monthly.

5.4 PURGE AND TRAP EQUIPMENT

5.4.1 Leak check all accessible fittings on the LSC-2000, ALS-2016, and their connections to the gas chromatograph, on a monthly basis.

5.4.2 Methanol flush the LSC-2000 and ALS-2016 quarterly or when needed as indicated by carryover contamination, plugged or restricted flow, or suspected adsorption problems.

5.4.3 Check flow rates when any line (other than the front panel connections) has been disconnected or connected.

5.5 THERMAL DESORPTION AND CRYOTRAPPING EQUIPMENT

5.5.1 Check temperatures of each zone monthly or more often if stability of the analysis is a problem.

6.0 DOCUMENTATION

6.1 Each analyst will keep records for the instrument or instruments assigned to him or her.

6.2 A checklist will be used to record the date and nature of the preventive maintenance performed. (See Figures 1-4).

6.3 Daily/weekly checklists will be stored in the Maintenance Log Book kept with each Mass Spectrometer. Monthly, quarterly, and semi-annually checklists will be stored in a notebook in the possession of the Technical Support Specialist.

6.4 Preventive maintenance documentation will be reviewed monthly by the Group Leader of the GC/MS Lab and at least quarterly by the Technical Director.

7.0 DEFINITION

7.1 When a preventive maintenance action is specified to be performed on a daily basis, it is intended to mean each day that the piece of analytical instrumentation is used, and is not required on the days that the instrument is not used.

Weekly and Daily Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Week Beginning: _____

Preventive Maintenance Action	Weekly	M	T	W	T	F	S	S
Gas Chromatograph								
Check zone temperatures via computer feedback								
Check supply gases for sufficient volume & pressure								
Autosampler								
Check or replace wash cup septa								
Check or dispose of waste solvent								
Visually check injection syringe for leakage								
Mass Spectrometer								
Check calibration gas valve & response level								

Record of routine or conditional preventive maintenance	M	T	W	T	F	S	S
Replacement of GC septa							
Replacement of GC injection port liner							
Clipped GC capillary column							
Replaced GC column							
Cleaned MS source & rods, dipped RF & EC							
Replaced desiccant in vent air drier							
MS manifold seals checked							
MS system leaked checked							
Printer ribbon replaced							
Purge & trap equipment flow rates checked							

Notes & Comments:

Monthly Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Gas Chromatograph	
☐ Check the gas pressures and flow rates	_____
Autosampler	
☐ Perform Varian instrument test while watching autosampler for proper action	_____
Vacuum Pumps	
☐ Check the oil level in the mechanical pumps and in the turbomolecular pumps	_____
☐ Purge the mechanical pumps	_____
Purge & Trap Equipment	
☐ Leak check all accessible fittings	_____
Thermal Desorption and Cyrotrapping Equipment	
☐ Check temperatures of each zone	_____

Notes & Comments:

Quarterly Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Gas Chromatograph	
☐ Dust off instrument internally & Externally	_____
Mass Spectrometers	
☐ Dust off instrument internally & Externally	_____
☐ Replace or clean the air filters	_____
☐ Check the cooling fans	_____
Vacuum Pumps	
☐ Check the integrity of the vacuum tubing	_____
Data Systems	
☐ Clean heads on tape units	_____
☐ Backup the operating software	_____
Purge & Trap Equipment	
☐ Methanol flush LSC & ALS	_____

Notes & Comments:

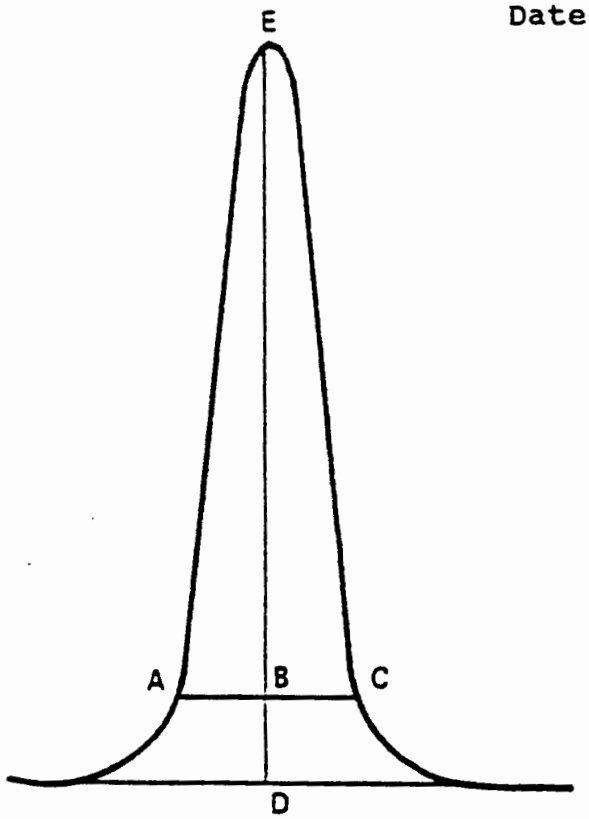
Semi-Annual Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Electronics	
☐ Dust off and reseat all printed circuit boards	_____
☐ Reseat all cable connectors	_____
☐ Check the operation of all solenoid valves	_____
Vacuum Pumps	
☐ Change the mechanical roughing pump oil	_____
Data Systems	
☐ Thoroughly clean printer	_____

Notes & Comments:



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Figure 5. Tailing factor calculation.

TO CALCULATE THE TAILING FACTOR YOU MUST FIRST DRAW TWO IMAGINARY LINES;
1) ONE THAT DIVIDS THE PEAK FROM APEX TO BASELINE (LINE DE).
2) ONE THAT GOES HORIZONTALLY ACROSS THE PEAK AT 10% OF IT'S HEIGHT. (LINE AC)
THE CALCULATION IS THEN SIMPLY MEASURING THE TWO LENGTHS AB AND BC.
N IS A REAL NUMBER THAT IS THE QUOTIENT (SEE EX. CALC)
THE VALUE FOR N SHOULD BE <3.

SOP MODIFICATION FORM

Use this form for minor modifications of SOP's. Reference the section to be changed and fully describe the change(s). If needed for maximum clarity, rewrite the whole section rather than listing several changes to it. If a single procedural change means changes in many sections of the SOP, it is best to amend and reissue the SOP. Otherwise, incorporate the changes into the body of the SOP at its annual review.

The original of this form is filed with the original of the SOP. Copies are issued to everyone who has a copy of the SOP.

SOP Name: PREVENTIVE MAINTENANCE IN THE GC/MS LAB

SOP Number: MV-003-00

Date change implemented : 10-15-91

Change initiated by: P Hamill

Operational approval:

QA Approval:

==========

Section to be changed: FIGURES 1-4

Change(s): THESE 4 FORMS WERE CONSOLIDATED INTO 1 MORE USABLE FORM. (ATTACHED)

THE SOP DOES NOT NEED TO BE RE-WRITTEN TO MAKE THIS CHANGE.

GC/MS PREVENTIVE MAINTENANCE CHECKLIST

INSTRUMENT:_____ ANALYST:_____ WEEK BEGINNING:_____

FREQUENCY							PREFORMEI	
						GAS CHROMATOGRAPH		
D	W	M	Q	S	A		YES	NO
*						Check zone temperatures via computer feedback.		
	*					Check supply gases for sufficient volume & pressure.		
	*					Replace GC septa.		
					*	Replace injection port liner.		
					*	Clip GC column.		
					*	Replace GC column.		
		*				Run instrument self test.		
						MASS SPECTROMETER		
D	W	M	Q	S	A		YES	NO
	*					Calibrate with FC-43.		
*						Check zone temperatures via computer feedback.		
	*					Check system vacuum via computer feedback.		
					*	Clean mass spectrometer source & rods. Dip RF.		
		*				Check oil level in mechanical pumps.		
			*			Replace oil pads in turbo.		
				*		Replace filters on mechanical pumps.		
				*		Replace oil in mechanical pumps.		
		*				Clean air filters to electronics.		
						TEKMAR PURGE & TRAP		
D	W	M	Q	S	A		YES	NO
*						Flush sparge vessels with reagent water.		
					*	Change out sparge vessels.		
	*					Check purge flow rate.		
	*					Check temperature zones.		
			*			Leak-check all assessable fittings.		
					*	Solvent back-flush transfer lines.		
						CTC AUTOSAMPLER		
D	W	M	Q	S	A		YES	NO
	*					Replace wash vial septa.		
	*					Replenish wash solvent.		
	*					Dispose of waste solvent.		
*						Check injection syringe.		
*						Confirm correct operation of autosampler.		
						PRINTER		
D	W	M	Q	S	A		YES	NO
					*	Change printer ribbon.		
*						Check paper supply.		
*						Check paper alignment.		
		*				Clean printer (paper dust, etc.)		

D=Daily W=Weekly M=Monthly Q=Quarterly S=Semi Annually A=As Needed

ROSS Ross Analytical Services, Inc.	SOP Standard Operating Procedure	SOP No: MV-004-00 Initial Date: 2/1/91 Page 1 of 4 Date Revised:

Title:

Training and Qualification in the GC/MS Laboratory

Prepared by:	Approved by:	Date:	QC Approval:
Patricia Farrell	CA Caldwell	5/7/91	CAC

1.0 PURPOSE

To describe the training program followed within the GC/MS laboratory.

2.0 APPLICATION

This procedure applies to all new employees in the GC/MS laboratory and employees performing analyses different from previous ones. It is used to provide guidelines for a training program and a method of documenting employee training.

3.0 REFERENCES

RASI Laboratory Quality Assurance Manual

4.0 ASSOCIATED SOPs

Associated SOP No. AD-006-00, Personnel Training and Qualifications.

5.0 PROGRAM

Individuals newly hired to work in the GC/MS laboratory shall be trained as follows:

5.1 First Day - General Orientation

5.1.1 On the first day of work the new employee will fill out all required paper work for the personnel office. This usually takes one to two hours. During this time the employee will be issued an employee handbook and given the opportunity to briefly read the book and ask questions concerning benefits and related personnel policies.

5.1.2 After completing the required paper work, the new employee will be taken on a "get acquainted" tour of the laboratory by the GC/MS supervisor or his associate. During this tour the following objectives will be met:

1. Introduction to other workers in the organic lab.

2. Introduction to the administrative assistant who will order lab coats.

3. Familiarization with general lab layout and emergency exits and equipment.

5.1.3 After the tour the following materials will be issued to the employee:

1. A copy of the Laboratory Quality Assurance Manual.

2. A copy of the Laboratory Safety Manual.

3. Copies of pertinent GC/MS lab SOPs.

These materials are the property of RASI and must be returned if the employee leaves the company.

5.1.4 After issuing these materials the employee will be asked to read the safety manual. Any questions will be answered by his assigned supervisor.

5.1.5 RASI employees transferred to the GC/MS lab will be issued the appropriate SOP's.

5.2 Training

5.2.1 New employees, trainees and experienced analysts learning new analyses are assigned to work with a qualified and experienced analyst. During this initial training period, the new employee reads instrument operation manuals and appropriate SOP's to familiarize himself with the general theory of instrument operation and analysis. He will also observe the analyst as he analyzes samples and processes data, and ask any questions which arise.

5.2.2 When the new employee has become acquainted with the general theory and operation of the instrument, the analyst in charge will begin to allow him, under constant supervision, to begin taking a gradual hand in operation. It is understood that this training does not follow a predetermined timetable, but proceeds based on the ability and previous experience of the new analyst. Some will proceed more slowly than others. This process will continue until either the new analyst is judged ready to proceed by himself, or is deemed unable to handle the complexity of the task. All work done to this point must have been under the direct supervision of an experienced analyst who has been in frequent contact with the laboratory supervisor with progress reports on the status of the trainee.

5.2.3 When the experienced analyst feels that a trainee is ready to begin analyses on his own, he will inform the laboratory supervisor who will arrange for performance testing for the new analyst. Performance tests consist of the analysis of blind spike samples as detailed in the methods to be tested. The new analyst will be required to perform all phases of the analysis, including instrument tuning and calibration, making of standards, set-up and analysis, data processing, and reporting of results.

5.2.4 Completed analytical results are reported directly to the laboratory supervisor, who will compare the results to the theoretical values and tabulate the results. An operator is deemed competent in a particular field of analyses when greater then 90 percent of the results obtained deviated by less than plus/minus 25 percent of the theoretical values for the samples analyzed.

5.2.5 If the operator is unable to meet these criteria, he will first re-examine the data along with his "teacher" to determine if any errors in calculations were made. If no mistakes are found, the new analyst will re-analyze spiked samples under the direct supervision of a qualified analyst. If errors are found, they are corrected, and the trainee will analyze a new set of performance evaluation samples. Only after successful analysis of unknown samples will the new analyst be allowed to proceed on his own.

5.3 Documentation

After an analyst has shown himself competent in performing a type of analyses, the laboratory supervisor will prepare a summary of the performance evaluation results, along with an explanation that the analyst has successfully completed his training and is deemed competent in that type of analyses. This documentation will be placed in the employee's training file for future reference and documentation. The performance evaluation applies only to the type of analyses for which the analyst was evaluated. For example, demonstrated proficiency in volatile analyses does not show competence in the analysis of semi-volatiles (base-neutral/acids).

Ross Analytical Services, Inc.

STANDARD OPERATING PROCEDURE

Title: Analysis of Semivolatile Compounds by GC/MS for RCRA/CERCLA by Method 8270B			
Prepared by:	Approved by:	Date: 10/21/96	QA Approval:

1.0 PURPOSE

This method describes procedures for the analysis of base-neutral and acidic compounds from various matrices using a GC/MS with a capillary column.

2.0 APPLICATION

This procedure covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography according to Method 8270B. This procedure is designed to analyze water, soil, sediment and waste samples for organic compounds listed in Section 10.0, Table 1 and other related compounds. It involves GC/MS analysis to determine if semivolatile (BNA) organic compounds are present in a sample.

This procedure applies to all semivolatile analyses performed in the RASI laboratory for RCRA/CERCLA purposes, except those done specifically by CLP protocol. This procedure is also used for confirmation analyses of pesticides/PCBs identified by GC/EC, as concentrations permit.

Interferences are discussed in Section 9.0.

3.0 REFERENCES

U.S. EPA SW-846, 3rd Edition, Update II, Method 8270B

U.S. EPA SW-846, 3rd Edition, Update II, Method 3510B

U.S. EPA SW-846, 3rd Edition, Update II, Method 3520B

U.S. EPA SW-846, 3rd Edition, Update II, Method 3540A

U.S. EPA SW-846, 3rd Edition, Update II, Method 3550A

U.S. EPA SW-846, 3rd Edition, Update II, Method 3580A

4.0 ASSOCIATED SOPs

OP-001 B/N/A Extraction of Water Samples Using SW-846 Methods 3510A, 3520A

OP-011 Ultrasonic Extraction of BNAs from Low Level Soils, Solid Wastes, and Sludges

OP-025 BNA Liquid Waste Dilution

OP-038 B/N/A Soxhlet Extraction for Wastes by Method 3540A

QC-013 Analyst Proficiency Requirements

5.0 EQUIPMENT/MATERIALS

5.1 Gas chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for splitless injection and all required accessories including syringes, analytical columns, and gases.

5.2 Column - 30 m x 0.32 mm ID bonded-phase silicone coated fused silica capillary column (Supelco SPB-5 or equivalent) with a film thickness of 1.0 micron.

5.3 Mass Spectrometer - Capable of scanning from 35-600 amu every one second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all required criteria when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet.

5.4 Data system - A computer system is interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software also allows integrating the abundance in any EICP between specific time or scan number limits.

5.5 Syringes - 10 uL and larger

5.6 GC/MS interface-Any GC to MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria. For narrow-bore capillary columns, the interface is capillary-direct into the mass spectron source.

5.7 Volumetric flasks, Class A -Appropriate sizes with ground glass stoppers.

5.8 Balance-Analytical, 0.001g.

5.9 Bottles- glass with Teflon-lined screw tops.

6.0 STANDARDS AND SOLUTIONS

All standards are stored in amber bottles with Teflon liners. Stock calibration standards are prepared at least annually and stored in a freezer with a temperature range of -10 °C to -20 °C. Stock standards and mixes purchased from suppliers are stored at -10 °C to -20 °C in amber bottles with Teflon liners for a maximum of one year. Daily continuing calibration standards are prepared every week and stored in amber bottles with Teflon liners at -10 to -20 °C.

6.1 Internal standards - 1,4 dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. An internal standard solution is prepared by dissolving 200 mg of each compound in 50 mL of methylene chloride. It may be necessary to use 5 to 10 percent carbon disulfide in this solution. A few minutes of ultrasonic mixing may help to dissolve all the constituents. The resulting solution contains each standard at a concentration of 4000 ng/uL. A 10 uL portion of this solution is added to each 1 mL of sample extract. This gives a concentration of 40 ng/uL of each constituent.

6.2 Calibration standards - Prepare calibration standards at a minimum of five concentration levels (20, 50, 80, 120, and 160 ng/2 uL). Each calibration standard contains each compound of interest, surrogate standards, and internal standards. Multiple mixes are usually required for Appendix IX and other extended target lists.

Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at -10 °C to -20 °C in screw-cap amber bottles with Teflon liners.

Fresh stock standards are prepared every twelve months at a minimum. The continuing calibration standard is prepared weekly and stored at -10 °C to -20 °C.

6.3 DFTPP tuning standard - Prepare a 25 ng/uL DFTPP standard containing 50 ng/uL of pentachlorophenol and benzidine in methylene chloride. Store at -10 °C to -20 °C.

6.4 Surrogate and both matrix and LCS spiking solutions - Surrogate and spiking solutions are prepared at concentrations of 200 ug/mL for acids and 100 ug/mL for base neutral compounds in acetone. See Section 10.0, Tables 7 and 8, for a listing of acid and base/neutral surrogate and LCS/matrix spiking compounds, respectively.

7.0 PROCEDURE

7.1 Sample Storage and Holding Times.

7.1.1 Procedure for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until extraction and analysis.

After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C (±2°C) until disposal.

7.1.2 Holding Times

The extract must be analyzed within 40 days of extraction.

7.2 Instrumental Set-up and Operating Conditions

7.2.1 The following instrumental parameters are required for all performance tests and for all sample analyses:

Electron energy: 70 volts (nominal)

Mass range: 35-500 amu

Scan time: Not to exceed 1 second per scan

7.2.2 The normal GC operating conditions to be used are as follows:

Initial column temperature: Hold at 50 °C for 4 minutes

Column temperature program: Ramp temperature 50°-290°C at 10°C/minute

Final column temperature: Hold at 290°C until elution of benzo (g,h,i) perylene

Injector temperature: 250°C

Transfer line temperature: 300°C

Source temperature: 200°C

Injector:	Grob-type, splitless
Sample volume:	2 uL
Carrier gas:	Helium at 3.5 mL/minute

7.3 Calibration

7.3.1 Initial Calibration

7.3.1.1 The GC/MS system must be tuned to meet the criteria listed in Section 10.0, Table 2 for a 50 ng injection (2 uL) of decafluorotriphenyl phosphine (DFTPP) tuning standard. No sample analyses can begin until all these criteria are met. Pentachlorophenol and benzidine are added to this standard to monitor column and inlet performance. Benzidine and pentachlorophenol should be present at their normal responses and their peaks should not tail. Poor response or peak shape may mean that the injection port needs cleaning or the column must be clipped. These criteria must be demonstrated each 12 hour shift.

7.3.1.2 The internal standards selected permit most components of interest in a chromatogram to have retention times of 0.80 to 1.20 relative to the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantification (see Section 10.0, Table 3). If interferences are noted, use the next most intense ion as the secondary ion, e.g., for 1,4-dichlorobenzene-d_4 use m/z 152 for quantification.

7.3.1.3 Inject 2 uL of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound including the surrogate compounds. Calculate response factors (RF) for each compound using Equation 1.

Eq. 1 $RF = (Ax * Cis) / (Ais * Cx)$

Where:

Ax = Area of the characteristic ion for the compound to be measured

Ais = Area of the characteristic ion for the specific internal standard from Section 10.0, Table 4

Cis = Concentration of the internal standard (ng/uL)

Cx = Concentration of the compound to be measured (ng/uL)

7.3.1.4 The average response factor (RFi*) must be calculated for all compounds, including surrogates, using the 5 RF values calculated for each compound from the initial (5 point) calibration. The average response factor is calculated using Equation 2.

Eq. 2 $$RFi^* = \frac{\Sigma\,(RFi) \text{ from 1 to 5}}{5}$$

Where,

RFi* = Average response factor for compound i

RFi = Initial response factor for compound i

Note: 5 is for the five different levels

A system performance check must be made before this calibration curve is used. Four system performance check compounds (SPCCs) are checked to ensure that a minimum response factor is achieved. These compounds are N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The minimum acceptable average response factor is 0.050 for all SPCCs. These compounds are used to check for both compound instability and for degradation caused by contamination in lines or actives sites in the system. These compounds are usually the first to show poor performance.

7.3.1.5 Using the average response factors (RFi*) from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all compounds. Calculate the %RSD using Equation 3.

Eq. 3 $$\%RSD = SD/RFi^* \quad * 100$$

Where,

SD = Standard deviation of the average response factors for compound i

RFi* = Same as in 7.3.1.4

The %RSD for each individual calibration check compound (CCC) must be less than 30%. The CCCs are listed below,

Phenol	1,4-dichlorobenzene
2-Nitrophenol	2,4-Dichlorophenol
Hexachlorobutadiene	4-Chloro-3-methylphenol
2,4,6-Trichlorophenol	Acenaphthene
N-nitrosodiphenylamine	Fluoranthene
Di-n-octylphthalate	Benzo(a)pyrene
Pentachlorophenol	

If a %RSD greater than 30% is measured for any one CCC, then corrective action to eliminate column reactive sites is required before reattempting calibration. Clean or replace the injector line and/or replace capillary column, then repeat the calibration procedure.

7.3.1.6 Linearity

7.3.1.6.1 If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation.

Or,

If the %RSD of any compound is 15% or less, you may construct a calibration curve using a first or second order regression fit of the five calibration points.

However,

7.3.1.6.2 If the %RSD of any compound is greater than 15%, construct a calibration curve using a first or higher order regression fit of the five calibration points for that compound. Select the lowest order regression which yields a coefficient of determination (r^2) of ≥ 0.99; however, note that there may be a limited number of compounds which have a lower r^2 value and the curve remains acceptable.

In practice, if the %RSD of any compound is greater than 15%, all compounds are quantitated from a regression fit curve.

7.3.2 Working Calibration Curve Verification

7.3.2.1 The GC/MS system must be tuned to meet the criteria listed in Section 10,Table 2, for a 50 ng injection(2ul) of DFTPP tuning standard. No sample analysis can begin until all of these criteria are m the beginning of each 12 hour shift.

7.3.2.2 A calibration standard at mid-concentration level(80 ng/ul) containing all semivolatile analytes and surrogates of interest must be analyzed after the DFTPP tune check during each 12 hour shift. Compare the instrument response factor from the daily standard with the SPCC (7.3.2.3) and CCC (7.3.2.4) criteria. **Both criteria must pass to be acceptable**.

7.3.2.3 System Performance Check Compounds (SPCCs): A system performance check must be made every 12 hour shift. For each SPCC compound in the daily calibration check, a minimum response factor of 0.050 must be met before sample analysis can begin.

7.3.2.4 Calibration Check Compounds (CCCs) : After the system performance check is met, the CCCs are used to check the validity of the initial calibration. The CCCs are checked using the equation below.

$$\% \text{ Drift } (\% D) = \frac{C_I - C_C}{C_I} \times 100$$

Where:

C_I - Calibration check compound standard concentration

C_C - Measured concentration

The maximum percent drift allowed for any CCC is 20%. If this criteria is not met(>20% drift) for any one CCC, corrective action must be taken. If no source of the problem can be determined after corrective action is taken then a new five point calibration curve must be generated. This criteria must be met befc any sample analysis can begin.

NOTE: If no CCCs are required target analytes, then only the required analytes must meet the maximum 20% drift criterion.

Only after both the minimum response factors and % drift criteria are met can sample analysis begin. Though the 20% maximum drift criteria does not apply to all targets, if many targets have a >20% drift, this is a warning that system performance is deteriorating.

7.3.2.5 Internal standard responses and retention times in all standards must be evaluated during or immediately after acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the system must be inspected for malfunctions and corrections made accordingly. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to 100%) from the last daily calibration standard check, the mass spectrometer system must be inspected for malfunction and corrections made as needed. When corrections are made, reanalysis of any samples analyzed while the system was malfunctioning is necessary.

7.4 Analysis

Make any extract dilutions indicated by characterization prior to the addition of internal standards. If any further dilutions of water or soil/sediment extracts are made, additional internal standards must be added to maintain the required 20 ng/uL of each internal standard in the extract volume. If the concentration of any compound exceeds the initial calibration range, the extract must be diluted and reanalyzed. Secondary ion quantitation is <u>only</u> allowed when there are sample interferences with the primary ion. If secondary ion quantitation is performed document the reasons.

7.5 Data Interpretation and Calculations

7.5.1 Qualitative Analysis

7.5.1.1 The compounds listed in the target list (Section 10.0, Table 1) shall be identified by an analyst competent in the interpretation of mass spectra by comparing the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: 1) elution of the sample component at the GC relative retention time as the standard component and 2) correspondence of the sample component and standard component mass spectra.

7.5.1.2 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within $\pm$0.06 RRT units of the RRT of the standard component. For reference the standard must be run on the same 12 hour "clock" as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

7.5.1.3 For comparison of standard and sample component mass spectra, mass spectra obtained on the same GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, if the GC/MS meets the DFTPP daily tuning requirements. These standard spectra may be obtained from the run used to obtain reference RTTs.

7.5.1.4 The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of ions specified in (1) must agree within plus or minus 30 percentage points between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.)

Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For most work, report only compounds detected above the Estimated Quantitation Limit.

If a compound cannot be verified by all of the criteria above, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then report that identification and proceed with quantification in 7.5.2.

7.5.1.5 If the library search is to be done, up to 20 non-surrogate, non-target organic compounds of the greatest apparent concentrations for the combined base/neutral/acid fraction are tentatively identified via a forward search of the NIST mass spectral library. (Substances with responses less than 10% of the nearest internal standard are not required to be searched). Only after visual comparison of sample spectra with the nearest library searches does the mass spectral interpretation specialist assign a tentative identification.

NOTE: Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

7.5.1.6 Guidelines for making tentative identification:

Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ±20 percentage points. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. NOTE: Data system library reduction programs can sometimes create these discrepancies.

7.5.1.7 If in the technical judgement of the mass interpretation spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral

specialist should give additional classification of the unknown compound, if possible (e.g., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound).

7.5.2 Quantitation

7.5.2.1 Target components identified are quantified by the internal standard method. The internal standard used is the one nearest the retention time to that of a given analyte. The EICP area of characteristic ions of analytes listed in Section 10.0, Table 3 are used.

7.5.2.2 Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required. The samples or standards with EICP areas outside the limits must be re-analyzed, and treated accordingly as shown below. If the corrections are made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that <u>does</u> meet the EICP criteria. After corrections are made, the analyst is required to reanalysis samples which were analyzed while the system was malfunctioning.

7.5.2.3 If after re-analysis, the EICP areas for all internal standards are within limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, report <u>only</u> data from the analysis with EICPs within limits.

7.5.2.4 If the re-analysis of the sample shows the same problem, e.g., the EICP areas are outside limits for both analyses, report the initial analysis only and note the presence of matrix interferences.

7.5.2.5 If the %RSD of a compound is 15% or less, then the concentration in the extract may be determined using the average RF from the initial calibration data (see Section 7.5.3, Calculations below). Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless a average response factor is calculated using the secondary ion. The regression fit of the initial calibration response factors may also be used when the %RSD ≤ 15%.

7.5.2.6 If the %RSD of a compound is > 15%, then the regression fit of the initial calibration response factors must be used for the determination of its concentration.

7.5.3 Calculations

7.5.3.1 Calculate the concentration in the sample using the average response factor (RFi*) in the following equations:

<u>Water</u>

$$\text{Concentration ug/L} = \frac{(Ax)(Is)(Vt)}{(Ais)(RFi^*)(Vo)(Vi)}$$

Where:

Ax	=	Area of the characteristic ion for the compound to be measured
Ais	=	Area of the characteristic ion for the internal standard
Is	=	Amount of internal standard injected in nanograms (ng)
Vo	=	Volume of water extracted in milliliters (mL)
Vi	=	Volume of extract injected (uL)
Vt	=	Volume of total extract (use 1000 uL or a factor of this when dilutions are made).
RFi*	=	Average response factor

Soil/Sediment

$$\text{Concentration ug/Kg} = \frac{(Ax)(Is)(Vt)}{(Ais)(RFi^*)(Vi)(Ws)(D)}$$

(Dry weight basis)

Where:

RFi*, Ax, Is, Ais	=	Same as given for water, above.
Vt	=	Volume of low level total extract (use 1000 uL or a factor of this when dilutions are made. The 1000 uL is derived from concentrating the 9.5 mL extract to 0.95 mL).
or, Vt	=	Volume of medium level extract (use 1000 uL or a factor of this when dilutions are made. The 1000 uL is derived from concentration 5 mL of the 10 mL extract to 0.5 mL).
Vi	=	Volume of extract injected (uL)

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

Ws	=	Weight of sample extracted (grams)

NOTE: For analyses to be reported on an as-received basis, omit the division by "D".

7.5.3.2 Generally, report only sample results which are greater than the EQL.

7.5.3.3 An estimated concentration for non-target components tentatively identified is determined by the internal standard method. For quantification, the nearest internal standard free of interferences used.

The formula for calculating concentrations is the same as for the target. Total area counts (or peak heights) from the total ion chromatograms are used for both the compound to be measured and the internal standard. A response factor of one is assumed. The value from this quantitation is qualified as estimated. This estimated concentration is calculated for all tentatively identified compounds as well as those identified as unknowns.

8.0 QUALITY CONTROL

8.1 A method detection limit (MDL) study must be conducted annually, see SOP QC-010.

8.2 Samples must be analyzed within 40 days from time of extraction.

8.3 Tuning

8.3.1 The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of a compliant tune. The time period ends after twelve (12) hours has elapsed according to the system clock.

8.3.2 Tune to meet criteria as shown in Section 10, Table 2.

Note: All instruments conditions must be identical to those used in sample analysis, except that a different temperature may be used.

8.3.3 DFTPP criteria must be met before any standards, samples, or blanks are analyzed. Any samples analyzed when tuning criteria have not been met will require reanalysis.

8.4 Calibration

8.4.1 After tuning criteria have been met but prior to the analysis of samples and required blanks, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound standards. Once the system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

8.4.2 Initial calibration of semivolatile target compounds is required at a minimum of five concentration levels (normal levels are 10, 25, 40, 60, and 80 ng/2 uL). Surrogate and internal standards are used with each of the calibration standards. Analyze all method blanks and standards under the same conditions as the sample.

8.4.3 System Performance Check Compounds (SPCC)

8.4.3.1 The initial calibration is valid only after the minimum response factors for SPCCs have been met.

8.4.3.2 A calibration standard(s) containing all semivolatile target compounds, including all required surrogates, must be analyzed each twelve hours during analysis. The SPCCs must meet the minimum response factor criterion of 0.050 before continuing.

8.4.4 Calibration Check Compounds

After the system performance check is met, the %Drift of the CCCs listed Section 7.3.1.5 must be checked to ensure the validity of the initial calibration.

If the percent drift for each target compound is less than 20.0%, the initial calibration is assumed to b valid. If the criteria are not met (>20.0% drift), for any one calibration check compound, corrective action must be taken.

Problems similar to those listed under SPCC could affect these criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five point calibration must be generated. These criteria must be met before sample analysis begins.

If no CCCs are required target analytes, then only the required analytes must meet the 20% drift criterion.

8.5 Method Blanks

One method blank must be extracted per extraction batch or 20 samples, whichever is more frequent. To be acceptable, a method blank must pass all recovery ranges for surrogates, and must not contain targets at greater than the established EQLs or reporting limits.

8.6 Laboratory Control Sample (LCS)

8.6.1 A LCS is prepared by spiking a water blank with the spike solution described in Section 6.4.

8.6.2 A LCS is analyzed at a frequency of once per batch or 20 samples, whichever is more frequent.

8.6.3 LCSs must pass the acceptance criteria listed in Section 10.0, Table 6. If the LCS fails, the batch of samples must be reprepared and reanalyzed.

8.7 Surrogates

Surrogates standard determinations are performed on all blanks, LCSs, and samples. Equation 7 is used to determine the percent recovery of each surrogate.

Eq. 7 $$\% \text{ Surrogate Recovery} = \frac{Q_d}{Q_a}$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample

Surrogate recovery limits are listed in Section 10.0, Table 9, for water and soil/sediment samples. Limits for other matrices are produced on a quarterly basis by the QA Department.

Surrogate recoveries must be with control limits for all blanks, LCSs, and samples.

If surrogate recoveries are outside the established limits for blanks and LCSs, then the batch must be reextracted and reanalyzed.

If surrogate recoveries are outside the established limits for a sample, then the sample must be reextracted and reanalyzed unless there is sufficient information available indicating that matrix interferences are the cause (e.g., an MS/MSD performed on this sample have the same problem, or the chromatogram shows presence of high interferences).

8.8 Matrix Spike/Matrix Spike Duplicate Analyses (MS/MSD)

8.8.1 Frequency of Analysis

An MS/MSD pair is prepared and analyzed on at least every 20th sample of each matrix type (water, soil, and waste). One TCLP (ZHE) leachate of each waste type (as determined by the client) is spiked with all targets. No MS/MSD pairs are performed on TCLP leachates.

8.8.2 Matrix Spike % Recovery - Calculate the percent recovery for each spike target using the equation below.

Eq. 8 $$\text{Matrix Spike Percent Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added for spiking mix

If the percent recovery is outside the range listed in Section 10.0, Table 6, flag the data noting that matrix interferences are present in these samples.

8.8.3 Relative Percent Difference (RPD) - Calculate the RPD for each spike target using the equation below.

Eq. 9 $$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where:

RPD = Relative Percent Difference

D_1 = First Sample Value

D_2 = Second Sample Value (duplicate)

If the RPD is greater than that listed in Section 10.0, Table 6, flag this in the narrative.

9.0 INTERFERENCES

Problems have been associated with the following compounds covered by this method.

Dichlorobenzidine and 4-chloroaniline can be subject to oxidative losses during solvent concentration. This is especially true in the soil/sediment method when concentrating the methylene chloride/acetone extraction solvent.

Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction on acetone solution, and photochemical decomposition.

N-nitrosodiphenylamine decomposes in the gas chromatographic inlet forming diphenylamine and, consequently, cannot be separated from diphenylamine native to the sample.

Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

10.0 TABLES

TABLE 1

Compound List and
Estimated Quantitation Limits (EQL)

Semivolatiles	CAS Number	Estimated Quantitation Limits * Water ug/L	Low Soil/Sediment ug/Kg
Normal Laboratory Target List			
Phenol	108-95-2	10	330
Bis(2-chloroethyl) ether (2,2'-Oxybis(1-chloropropane))	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
Bis(2-Chloroisopropyl) ether	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	10	330
Bis(2-Chloroethoxy) methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	10	330
Dimethylphthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
2,6-Dinitrotoluene	606-20-2	10	330
3-Nitroaniline	99-09-2	10	330
Acenaphthene	83-32-9	10	330

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

TABLE 1
(continued)

Semivolatiles	CAS Number	Estimated Quantitation Limits * Water ug/L	Low Soil/Sediment ug/Kg
2,4-Dinitrophenol	51-28-5	10	330
4-Nitrophenol	100-02-7	10	330
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenol phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	10	330
4,6-Dinitro-2-methylphenol	534-32-1	10	330
N-Nitrosodiphenylamine	86-30-6	10	330
4-Bromopheny phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	10	330
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Di-n-butylphthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Pyrene	129-00-0	10	330
Butylbenzylphthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	10	330
Benzo(a)anthracene	56-55-3	10	330
Chrysene	218-01-9	10	330
Bis(2-ethylhexyl) phthalate	117-81-7	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Additional Target List			
2-Acetylaminofluorene	53-96-3	20	670
Aminoazobenzene	60-09-3	10	330
Aramite	140-57-8	20	670
Chlorobenzilate	510-15-6	10	670

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

TABLE 1
(continued)

Semivolatiles	CAS Number	Estimated Quantitation Limits * Water ug/L	Low Soil/Sediment ug/Kg
Diallate (cis or trans)	2303-16-4	10	330
Dibenzo(a,e)pyrene	192-65-4	10	330
Dimethoate	60-51-5	20	670
3,3' -Dimethoxybenzidine	119-90-4	100	3300
Dimethylaminoazobenzene	60-11-7	10	330
7,12 -Dimethylbenz(a)anthracene	57-97-6	10	330
3,3 -Dimethylbenzidine	119-93-7	10	330
1,2 -Dinitrobenzene	528-29-0	40	1300
1,3 -Dinitrobenzene	99-65-0	20	670
1,4 -Dinitrobenzene	100-25-4	40	1300
2,6 -Dinitrotoluene	606-20-2	10	330
Dinoseb	88-85-7	20	670
Ethyl methanesulfonate	62-50-0	20	670
Hexachloropropene	1888-71-7	10	330
Isosafrole	120-58-1	10	330
Methapyrilene	91-80-5	100	3300
3-Methylcholanthrene	56-49-5	10	330
Methyl methanesulfonate	66-27-3	10	330
3-Methylphenol	108-39-4	10	330
1,4-Naphthoquinone	130-15-4	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
5-Nitro-o-toluidine	99-55-8	10	330
4-Nitroquinoline-l-oxide	56-57-5	40	1300
N-Nitrosodibutylamine	924-16-3	10	330
N-Nitrosodiethylamine	55-18-5	20	670
N-Nitrosopiperidine	100-75-4	20	670
N-Nitrosopyrrolidine	930-55-2	40	1300
Pentachlorobenzene	680-93-5	10	330
Pentachloronitrobenzene	82-68-8	20	670
Phenacetin	62-44-2	20	670
Phthalic anhydride (as Phthalic acid)	85-44-9	100	3300
Pronamide	23950-58-5	10	330
Pyridine	110-86-1	10	330
Resorcinol	108-46-3	100	3300
Safrole	94-59-7	10	330
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

TABLE 1
(continued)

Semivolatiles	CAS Number	Estimated Quantitation Limits * Water ug/L	Low Soil/Sediment ug/Kg
2,3,4,6-Tetrachlorophenol	58-90-2	10	330
Thiophenol (Benzenethiol)	108-98-5	20	330
o-Toluidine	95-53-4	10	330
1,3,5-Trinitrobenzene	99-35-4	10	330

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

TABLE 2

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion abundance criteria
51	30 - 60 percent of mass 198
68	less than 2 percent of mass 69
70	less than 2 percent of mass 69
127	40 - 60 percent of mass 198
197	less than 1 percent of mass 198
198	base peak, 100 percent relative abundance
199	5 - 9 percent of mass 198
275	10 - 30 percent of mass 198
365	greater than 1 percent of mass 198
441	present but less than mass 443
442	greater than 40 percent of mass 198
443	17 - 23 percent of mass 442

TABLE 3

Characteristic Ions for Semivolatile Compounds

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
Bis(2-chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
Bis(2-chloroisopropyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
Benzoic acid	122	105, 77
Bis(2-chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenol phenyl ether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167

TABLE 3
(continued)

Characteristic Ions for Semivolatile Compounds

Parameter	Primary Ion	Secondary Ion(s)
4-Bromophenyl phenyl ether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
Bis(2-ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octyl phthalate	149	-
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
2-Picoline	93	66, 92
Aniline	93	66, 65
N-Nitrosomethylethylamine	88	42,88,43,56
Thiophenol (Benzenethiol)	110	110,66,109,84
Methyl methanesulfonate	80	80,79,65,95
N-Nitrosodiethylamine	102	102,42,57,44,56
Ethyl methanesulfonate	79	79,109,97,45,65
Hexachloropropene	213	213,211,215,117,106,141
N-Nitrosopyrrolidine	100	100,41,42,68,69
Acetophenone	105	71,105,51,120
o-Toluidine	106	106,107,77,51,79
3-Methylphenol	107	107,108,77,79,90
N-Nitrosopiperidine	114	42,114,55,56,41
1-Chloronaphthalene	162	127,164
2,6-Dinitrotoluene	165	63,89
Phthalic anhydride	104	104,76,50,148
Isosafrole	162	162,131,104,77,51
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	158,104,102,76,50,130
N-Nitrosodibutylamine	84	84,57,41,116,158
Resorcinol	110	110,81,82,53,69
Diphenylamine	169	168,167

TABLE 3
(continued)

Characteristic Ions for Semivolatile Compounds

Parameter	Primary Ion	Secondary Ion(s)
1,2,4,5-Tetrachlorobenzene	216	216,214,179,108,143,218
1-Naphthylamine	143	143,115,89,53
5-Nitro-o-toluidine	152	77,152,79,106,94
1,4-Dinitrobenzene	168	168,75,50,76,92,122
Diallate (cis or trans)	86	86,234,43,70
Pentachlorobenzene	250	250,252,108,248,215,254
Pentachloronitrobenzene	237	237,142,214,249,295,265
4-Nitroquinoline-1-oxide	174	174,101,128,75,116
2,3,4,6-Tetrachlorophenol	232	232,131,230,166,234,168
Benzidine	184	92,185
Phenacetin	108	180,179,109,137,80
Dimethoate	87	87,93,125,143,229
Pronamide	173	173,175,145,109,147
Dinoseb	211	211,163,147,117,240
Dimethylaminoazobenzene	225	225,120,77,105,148,42
3,3'Dimethylbenzidine	212	212,106,196,180
Methapyrilene	97	97,50,191,71
Aramite	185	185,191,319,334,197,321
Chlorobenzilate	251	251,139,253,111,141
7,12-Dimethybenz(a)anthracene	256	256,241,239,120
2-Acetylaminofluorene	181	181,180,223,152
3,3'-Dimethoxybenzidine	244	244,201,229
1,2,4,5-Dibenzopyrene	302	302,151,150,300
1,2-Diphenylhydrazine	77	105,182
N-Nitrosodimethylamine	42	74,44

TABLE 4

Characteristic Ions for Surrogates and
Internal Standards for Semivolatile Compounds

Surrogates	Primary Ion	Secondary Ion(s)
Phenol-d_6	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene d-5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122, 212
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115, 150
Naphthalene-d_8	136	68
Acenapthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

The internal standards are added to all calibration standards and all sample extracts just prior to analysis by GC/MS. A 10 uL aliquot of the internal standard solution should be added to a 1 mL aliquot of calibration standards.

TABLE 5

Semivolatile Internal Standards with Corresponding Analytes
Assigned for Quantitation

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl)ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl)ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenol
1,3-Dichlorobenzene	2,4-Dichlorophenol	Phenyl ether
1,4-Dichlorobenzene	2,6-Dichlorophenol	Dibenzofuran
1,2-Dichlorobenzene	a,a-Dimethyl phenethylamine	Diethyl phthalate
Ethyl methanesulfonate	2,4-Dimethyphenol	Dimethylphthalate
2-Fluorophenol (surrogate)	Hexachlorobutadiene	2,4-Dinitrophenol
Hexachloroethane	Isophorone	2,4-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	2,6-Dinitrotoluene
2-Methylphenol	Nitrobenzene-d6 (surrogate)	Fluorene
4-Methylphenol	2-Nitrophenol	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Naphthalene	4-Nitroaniline
N-Nitroso-di-n-propylamine	Nitrobenzene	3-Nitroaniline
Phenol	N-Nitrosodibutylamine	2-Fluorobiphenyl (surrogate)
Phenol-d6 (surrogate)	N-Nitrosopiperidine	1-Naphthylamine
2-Picoline	1,2,4-Trichlorobenzene	2-Naphthylamine
		2-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surrogate)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

Phenanthrene-d10	Chrysene-d12	Perylene-d12
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i) perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methyphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
1,2-Diphenylhydrazine	p-Dimethylaminoazobenzene	7,12-Dimethylbenz(a)anthracene
Fluoranthene	Pyrene	Di-n-octyl phthalate
Hexachlorobenzene	Terphenyl-d14 (surrogate)	Indeno(1,2,3-cd)pyrene
N-Nitrosodiphenylamine		3-Methylcholanthrene
Pentachlorophenol		
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

TABLE 6

QC Acceptance Criteria for MS/MSD and LCS

Compound	Method Percent Recovery Limits	MS/MSD RPD Limits
Phenol	5-112	40
2-Chlorophenol	23-134	40
1,4-Dichlorobenzene	20-124	40
N-Nitroso-di-n-propylamine	D-230	40
1,2,4-Trichlorobenzene	44-142	40
4-Chloro-3-methylphenol	22-147	40
Acenaphthene	47-145	40
4-Nitrophenol	D-132	40
2,4-Dinitrotoluene	39-139	40
Pentachlorophenol	14-176	40
Pyrene	52-115	40

D = detection

TABLE 7

Surrogate Table

Base/Neutral Compounds	Final Concentration
2-Fluorobiphenyl	50 ug/mL
Nitrobenzene-d5	50 ug/mL
Terphenyl-d14	50 ug/mL

Acid Compounds	Final Concentration
2-Fluorophenol	100 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Phenol-d6	100 ug/mL

TABLE 8

Matrix Spiking Table

Base/Neutral Compounds	Final Concentration
1,2,4-Trichlorobenzene	50 ug/mL
Acenaphthene	50 ug/mL
2,4-Dinitrotoluene	50 ug/mL
Pyrene	50 ug/mL
N-Nitroso-di-n-propylamine	50 ug/mL
1,4-Dichlorobenzene	50 ug/mL

Acid Compounds	Final Concentration
Pentachlorophenol	100 ug/mL
Phenol	100 ug/mL
2-Chlorophenol	100 ug/mL
4-Chloro-3-methylphenol	100 ug/mL
4-Nitrophenol	100 ug/mL

TABLE 9

Surrogate Recovery Limits for Water and Soil/Sediment Samples

Surrogate Compound	Low/High Water	Low/High Soil/Sediment
Nitrobenzene-d5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
p-Terphenyl-d14	33-141	18-137
Phenol-d5	10-100	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122

ATTACHMENT C
GMP WATER QUALITY DATA TO DATE

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		83600.0000		84900.0000		80100.0000
SULFATE	mg/l		5360.0000		5540.0000		4935.0000
TOTAL DISS SOLIDS	mg/l		77500.0000		64900.0000		67800.0000
TOTAL SUSP SOLIDS	mg/l	<	10.0000		19.0000		30.5000
DENSITY	g/mL		1.0530		1.0460		1.0475
pH	SU		7.1000		7.2850		7.2350
ALKALINITY	mg/l		47.0000		52.5000		50.1000
BROMIDE	mg/l		45.0000		35.4000		41.1500
CHLORIDE	mg/l		34750.0000		33250.0000		37100.0000
FLUORIDE	mg/l	<	2.0000		1.4400		1.0650
IODIDE	mg/l	<	2.0000	<	2.0000	<	2.0000
NITROGEN, NO3 (AS N)	mg/l	<	0.0100	<	0.2000	<	0.1000
TOTAL ORGANIC CARBON	mg/l		1.4800		1.3100		1.0250
TOTAL ORGANIC HALOGENS	mg/l		0.0255		0.0365		0.0434
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		13.8000		11.5500		12.4000
CALCIUM	mg/l		1685.0000		1700.0000		1705.0000
IRON	mg/l	<	1.0000	<	0.5000	<	0.5000
LITHIUM	mg/l		0.4155		0.3580		0.4065
MAGNESIUM	mg/l		1095.0000		1045.0000		1001.0000
POTASSIUM	mg/l		485.5000		470.5000		461.0000
SILICA	mg/l		9.3350		10.3000		10.3000
SODIUM	mg/l		19950.0000		18350.0000		18650.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l			<	1.5000	<	1.5000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0055	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.0400		0.0240		0.0260
BERYLLIUM	mg/l	<	0.0200	<	0.0025	<	0.0025
CADMIUM	mg/l			<	0.0025	<	0.0025
CHROMIUM	mg/l	<	0.0025	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l			<	0.0130	<	0.2500
LEAD	mg/l	<	0.0130	<	0.0130	<	0.0130
MERCURY	mg/l	<	0.0002	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0100	<	0.0130	<	0.0130
SILVER	mg/l	<	0.0025	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l			<	0.0250	<	0.0250
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l			<	10.0000	<	10.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000	<	10.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l		1.6500	<	5.0000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	20.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l				17.8000	<	10.0000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.5000	<	0.2500
DISULFOTON	ug/l			<	0.5000	<	0.2500
FAMPHUR	ug/l			<	0.5000	<	0.2500
METHYL PARATHION	ug/l			<	0.5000	<	0.2500
PARATHION	ug/l			<	0.5000	<	0.2500
PHORATE	ug/l			<	0.5000	<	0.2500

TABLE D-18C-1
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-1

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.5000	<	0.2500
THIONAZIN	ug/l			<	0.5000	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.5000	<	0.2500
2,4-D	ug/l			<	1.0000	<	1.0000
2,4,5-T	ug/l			<	1.0000	<	1.0000
2,4,5- TP	ug/l			<	1.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0270	<	0.0380
HEXACHLORODIBENZOFURANS	ng/l			<	0.0090	<	0.0190
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0180	<	0.1100
PENTACHLORODIBENZOFURANS	ng/l			<	0.0150	<	0.0500
2,3,7,8-TCDD	ng/l			<	0.0160	<	0.0490
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0160	<	0.0490
TETRACHLORODIBENZOFURANS	ng/l			<	0.0088	<	0.0310

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		80000.0000		81250.0000		78000.0000
SULFATE	mg/l		5505.0000		5625.0000		5705.0000
TOTAL DISS SOLIDS	mg/l		67600.0000		70500.0000		65000.0000
TOTAL SUSP SOLIDS	mg/l		41.0000	<	10.0000		40.5000
DENSITY	g/mL		1.0550		1.0403		1.0395
pH	SU		7.3300		7.1250		7.3400
ALKALINITY	mg/l		28.8750		48.1000		48.1000
BROMIDE	mg/l		33.5500		31.1000		37.0500
CHLORIDE	mg/l		38500.0000		35100.0000		36000.0000
FLUORIDE	mg/l	<	2.0000		1.3100	<	1.0000
IODIDE	mg/l	<	2.0000	<	2.0000	<	2.0000
NITROGEN, NO3 (AS N)	mg/l	<	0.1000		0.2700	<	0.1000
TOTAL ORGANIC CARBON	mg/l	<	4.0000		1.3400		2.2200
TOTAL ORGANIC HALOGENS	mg/l		44.1000		0.4574		0.0474
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		17.2000		14.0500		15.5000
CALCIUM	mg/l		1455.0000		1445.0000		1420.0000
IRON	mg/l	<	1.0000	<	0.5000	<	0.5000
LITHIUM	mg/l	<	0.3890		0.3460		0.4090
MAGNESIUM	mg/l		963.0000		938.0000		880.0000
POTASSIUM	mg/l		448.0000		415.5000		447.5000
SILICA	mg/l		7.6600		10.4000		9.6400
SODIUM	mg/l		18750.0000		18300.0000		19000.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l			<	1.5000	<	1.5000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0100	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.0400		0.0201		0.0211
BERYLLIUM	mg/l	<	0.0200	<	0.0025	<	0.0025
CADMIUM	mg/l			<	0.0025	<	0.0025
CHROMIUM	mg/l	<	0.0025	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l			<	0.0130	<	0.2500
LEAD	mg/l	<	0.0130	<	0.0130	<	0.0130
MERCURY	mg/l	<	0.0010	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0100	<	0.0130	<	0.0130
SILVER	mg/l		0.0025	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l			<	0.0250	<	0.0250
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l			<	10.0000	<	10.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000	<	10.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	20.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l			<	10.0000	<	10.0000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.2500	<	0.2500
DISULFOTON	ug/l			<	0.2500	<	0.2500
FAMPHUR	ug/l			<	0.2500	<	0.2500
METHYL PARATHION	ug/l			<	0.2500	<	0.2500
PARATHION	ug/l			<	0.2500	<	0.2500
PHORATE	ug/l			<	0.2500	<	0.2500

TABLE D-18C-2
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-2

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.2500	<	0.2500
THIONAZIN	ug/l			<	0.2500	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.2500	<	0.2500
2,4-D	ug/l			<	1.0000	<	1.0000
2,4,5-T	ug/l			<	1.0000	<	1.0000
2,4,5- TP	ug/l			<	1.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0580	<	0.0740
HEXACHLORODIBENZOFURANS	ng/l			<	0.0320	<	0.0510
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0940	<	0.0830
PENTACHLORODIBENZOFURANS	ng/l			<	0.0730	<	0.0740
2,3,7,8-TCDD	ng/l			<	0.0360	<	0.0530
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0360	<	0.0530
TETRACHLORODIBENZOFURANS	ng/l			<	0.0410	<	0.0580

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		193500.0000		200500.0000		118500.0000
SULFATE	mg/l		6705.0000		6800.0000		7650.0000
TOTAL DISS SOLIDS	mg/l		218500.0000		211500.0000		217205.0000
TOTAL SUSP SOLIDS	mg/l		72.5000	<	10.0000		15.0000
DENSITY	g/mL		1.1350		1.1435		1.1540
pH	SU		7.1150		6.8200		6.8550
ALKALINITY	mg/l		44.0000		35.0000		35.6000
BROMIDE	mg/l		102.5000		83.4000		70.6000
CHLORIDE	mg/l		134000.0000		144500.0000		128500.0000
FLUORIDE	mg/l	<	2.0000	<	10.0000	<	1.0000
IODIDE	mg/l	<	2.0000	<	2.0000		2.1800
NITROGEN, NO3 (AS N)	mg/l	<	0.1000		0.1950	<	0.1000
TOTAL ORGANIC CARBON	mg/l	<	1.3600		1.4100		1.2800
TOTAL ORGANIC HALOGENS	mg/l		0.1565		0.0394		54.9500
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		49.3000		49.4000		40.7000
CALCIUM	mg/l		1385.0000		1465.0000		1280.0000
IRON	mg/l	<	4.0000	<	0.5000	<	0.5000
LITHIUM	mg/l	<	0.8000		0.9065		0.7995
MAGNESIUM	mg/l		2160.0000		1985.0000		1925.0000
POTASSIUM	mg/l		1345.0000		1710.0000		1405.0000
SILICA	mg/l		4.5700		4.2000		3.9550
SODIUM	mg/l		77900.0000		73950.0000		72450.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l				5.6000	<	1.5000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0100	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.1600		0.0283		0.0260
BERYLLIUM	mg/l	<	0.0800		0.0054	<	0.0025
CADMIUM	mg/l			<	0.0025	<	0.0025
CHROMIUM	mg/l	<	0.0026	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l				0.0460	<	0.0250
LEAD	mg/l	<	0.0130	<	0.0130	<	0.0130
MERCURY	mg/l	<	0.0010	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0100	<	0.0130	<	0.0130
SILVER	mg/l	<	0.0025	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l			<	0.0250	<	0.0250
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l				21.0000		26.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000	<	10.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	20.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l				10.0000		10.8000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.2500	<	0.2500
DISULFOTON	ug/l			<	0.2500	<	0.2500
FAMPHUR	ug/l			<	0.2500	<	0.2500
METHYL PARATHION	ug/l			<	0.2500	<	0.2500
PARATHION	ug/l			<	0.2500	<	0.2500
PHORATE	ug/l			<	0.2500	<	0.2500

TABLE D-18C-3
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-3

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.2500	<	0.2500
THIONAZIN	ug/l			<	0.2500	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.2500	<	0.2500
2,4-D	ug/l			<	1.0000	<	1.0000
2,4,5-T	ug/l			<	1.0000	<	1.0000
2,4,5- TP	ug/l			<	1.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0120	<	0.0190
HEXACHLORODIBENZOFURANS	ng/l			<	0.0035	<	0.0120
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0880	<	0.0240
PENTACHLORODIBENZOFURANS	ng/l			<	0.0065	<	0.0270
2,3,7,8-TCDD	ng/l			<	0.0046	<	0.0220
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0046	<	0.0220
TETRACHLORODIBENZOFURANS	ng/l			<	0.0031	<	0.0160

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.2500	<	0.2500
THIONAZIN	ug/l			<	0.2500	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.2500	<	0.2500
2,4-D	ug/l			<	1.0000	<	1.0000
2,4,5-T	ug/l			<	1.0000	<	1.0000
2,4,5- TP	ug/l			<	1.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0340	<	0.0230
HEXACHLORODIBENZOFURANS	ng/l			<	0.0180	<	0.0070
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0620	<	0.0150
PENTACHLORODIBENZOFURANS	ng/l			<	0.0390	<	0.0140
2,3,7,8-TCDD	ng/l			<	0.0180	<	0.0140
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0180	<	0.0140
TETRACHLORODIBENZOFURANS	ng/l			<	0.0130	<	0.0078

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.2500	<	0.2500
DISULFOTON	ug/l			<	0.2500	<	0.2500
FAMPHUR	ug/l			<	0.2500	<	0.2500
METHYL PARATHION	ug/l			<	0.2500	<	0.2500
PARATHION	ug/l			<	0.2500	<	0.2500
PHORATE	ug/l			<	0.2500	<	0.2500

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l			<	10.0000		17.1000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	10.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l		10.0000		15.8000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0080	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.1600		0.0258		0.0240
BERYLLIUM	mg/l	<	0.0800	<	0.0260	<	0.0025
CADMIUM	mg/l			<	0.0025		0.0410
CHROMIUM	mg/l	<	0.0025	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l			<	0.0130	<	0.0130
LEAD	mg/l	<	0.0130	<	0.0130		0.5250
MERCURY	mg/l	<	0.0010	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0100	<	0.0130	<	0.0130
SILVER	mg/l	<	0.0025	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l			<	0.0250	<	0.0250
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l				12.4000	<	10.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000	<	10.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-4
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-4

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		106500.0000		126500.0000		118500.0000
SULFATE	mg/l		7075.0000		6240.0000		7260.0000
TOTAL DISS SOLIDS	mg/l		108000.0000		106500.0000		122500.0000
TOTAL SUSP SOLIDS	mg/l		57.0000	<	10.0000	<	10.0000
DENSITY	g/mL		1.0800		1.0755		1.0665
pH	SU		7.1650		7.5800		7.2350
ALKALINITY	mg/l		41.0000		29.5000		39.5500
BROMIDE	mg/l		54.1500		49.7500		50.2500
CHLORIDE	mg/l		60950.0000		61350.0000		58350.0000
FLUORIDE	mg/l	<	2.0000		2.3400		2.5800
IODIDE	mg/l	<	2.0000	<	2.0000	<	2.0000
NITROGEN, NO3 (AS N)	mg/l	<	0.1000	<	0.1000	<	0.1000
TOTAL ORGANIC CARBON	mg/l	<	1.1850		2.0050		1.6500
TOTAL ORGANIC HALOGENS	mg/l	<	0.0395		0.0934		0.0466
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		33.5500		27.8500		27.2500
CALCIUM	mg/l		1680.0000		1560.0000	<	1595.0000
IRON	mg/l	<	4.0000	<	0.5000	<	0.5000
LITHIUM	mg/l	<	0.8000		0.4760		0.6040
MAGNESIUM	mg/l		1250.0000		1085.0000		1145.0000
POTASSIUM	mg/l		748.0000		695.5000		757.5000
SILICA	mg/l		6.0750		6.2550	<	7.4400
SODIUM	mg/l		35150.0000		33000.0000		33100.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l			<	1.5000	<	1.5000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		43150.0000		44500.0000		45050.0000
SULFATE	mg/l		5375.0000		5815.0000		5090.0000
TOTAL DISS SOLIDS	mg/l		43950.0000		33250.0000		32400.0000
TOTAL SUSP SOLIDS	mg/l	<	10.0000	<	10.0000	<	10.0000
DENSITY	g/mL		1.0290		1.0220		1.0185
pH	SU		7.8000		7.6700		7.5800
ALKALINITY	mg/l		51.0000		41.6000		50.1000
BROMIDE	mg/l		21.5000		19.7500		46.2000
CHLORIDE	mg/l		14900.0000		15850.0000		15950.0000
FLUORIDE	mg/l	<	3.0000		2.9100		2.2500
IODIDE	mg/l	<	2.0000	<	2.0000	<	2.0000
NITROGEN, NO3 (AS N)	mg/l	<	0.1000		0.2850	<	0.1000
TOTAL ORGANIC CARBON	mg/l		1.9450		1.3600		1.6200
TOTAL ORGANIC HALOGENS	mg/l		0.0537		0.0635		0.0353
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		30.2000		28.2000		28.3000
CALCIUM	mg/l		984.5000		1020.0000		1020.0000
IRON	mg/l	<	1.0000	<	0.5000	<	0.5000
LITHIUM	mg/l		0.3570		0.3805		0.3705
MAGNESIUM	mg/l		433.0000		422.0000		424.0000
POTASSIUM	mg/l		286.0000		289.0000		291.5000
SILICA	mg/l		10.9500		10.2500		11.0500
SODIUM	mg/l		8890.0000		9500.0000		9135.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l			<	1.5000		3.8000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0130	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.0400		0.0185		0.0150
BERYLLIUM	mg/l	<	0.0200	<	0.0025	<	0.0025
CADMIUM	mg/l			<	0.0025	<	0.0025
CHROMIUM	mg/l	<	0.0130	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l			<	0.0130	<	0.0130
LEAD	mg/l	<	0.0130	<	0.0130	<	0.0130
MERCURY	mg/l	<	0.0010	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0130	<	0.0130	<	0.0130
SILVER	mg/l	<	0.0130	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l			<	0.0250	<	0.0250
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l				10.0000	<	10.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000	<	10.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	10.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l				13.0000	<	10.0000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.2500	<	0.2500
DISULFOTON	ug/l			<	0.2500	<	0.2500
FAMPHUR	ug/l			<	0.2500	<	0.2500
METHYL PARATHION	ug/l			<	0.2500	<	0.2500
PARATHION	ug/l			<	0.2500	<	0.2500
PHORATE	ug/l			<	0.2500	<	0.2500

TABLE D-18C-5
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-5

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.2500	<	0.2500
THIONAZIN	ug/l			<	0.2500	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.2500	<	0.2500
2,4-D	ug/l			<	1.0000	<	1.0000
2,4,5-T	ug/l			<	1.0000	<	1.0000
2,4,5- TP	ug/l			<	1.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0580	<	0.0180
HEXACHLORODIBENZOFURANS	ng/l			<	0.0320	<	0.0130
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0940	<	0.0130
PENTACHLORODIBENZOFURANS	ng/l			<	0.0730	<	0.0100
2,3,7,8-TCDD	ng/l			<	0.0360	<	0.0100
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0360	<	0.0100
TETRACHLORODIBENZOFURANS	ng/l			<	0.0410	<	0.0100

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		27150.0000		18750.0000		21800.0000
SULFATE	mg/l		5465.0000		4700.0000		4430.0000
TOTAL DISS SOLIDS	mg/l		21600.0000		17200.0000		16400.0000
TOTAL SUSP SOLIDS	mg/l	<	14.7500	<	10.0000	<	10.0000
DENSITY	g/mL		0.9995		1.0028		1.0120
pH	SU		7.8050		7.6350		7.6200
ALKALINITY	mg/l		52.5000		51.5000		47.6000
BROMIDE	mg/l		10.9500	<	2.0000		10.5400
CHLORIDE	mg/l		15800.0000		6200.0000		6148.0000
FLUORIDE	mg/l	<	2.0000		2.2500		2.5850
IODIDE	mg/l	<	1.5100	<	2.0000	<	2.0000
NITROGEN, NO3 (AS N)	mg/l	<	0.1000	<	0.1000	<	0.1000
TOTAL ORGANIC CARBON	mg/l		1.1350		1.0450		1.5450
TOTAL ORGANIC HALOGENS	mg/l		0.0455		0.0554		0.0624
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		16.7500		15.6000		14.5000
CALCIUM	mg/l		725.0000		661.0000		702.5000
IRON	mg/l	<	1.0000	<	0.1870	<	0.5000
LITHIUM	mg/l		0.2605		0.2385		0.2545
MAGNESIUM	mg/l		251.5000		213.5000		200.5000
POTASSIUM	mg/l		183.0000		194.5000		151.5000
SILICA	mg/l		10.4500		10.6500		11.8900
SODIUM	mg/l		6060.0000		5455.0000		4460.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l			<	1.5000	<	1.5000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0040	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.0400		0.0100		0.0110
BERYLLIUM	mg/l	<	0.0200	<	0.0025	<	0.0030
CADMIUM	mg/l			<	0.0025	<	0.0030
CHROMIUM	mg/l	<	0.0026	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l			<	0.0130	<	0.0250
LEAD	mg/l	<	0.0130	<	0.0130	<	0.0130
MERCURY	mg/l	<	0.0010	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0040	<	0.0130	<	0.0130
SILVER	mg/l	<	0.0029	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l			<	0.0250	<	0.0250
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l			<	10.0000	<	10.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000		140.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l	<	6.5000	<	5.0000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	20.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l				26.0000	<	10.0000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.2500	<	0.2500
DISULFOTON	ug/l			<	0.2500	<	0.2500
FAMPHUR	ug/l			<	0.2500	<	0.2500
METHYL PARATHION	ug/l			<	0.2500	<	0.2500
PARATHION	ug/l			<	0.2500	<	0.2500
PHORATE	ug/l			<	0.2500	<	0.2500

TABLE D-18C-6
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.2500	<	0.2500
THIONAZIN	ug/l			<	0.2500	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.2500	<	0.2500
2,4-D	ug/l			<	2.0000	<	1.0000
2,4,5-T	ug/l			<	2.0000	<	1.0000
2,4,5- TP	ug/l			<	2.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.1400	<	0.1000
HEXACHLORODIBENZOFURANS	ng/l			<	0.0550	<	0.0570
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0960	<	0.1000
PENTACHLORODIBENZOFURANS	ng/l			<	0.0790	<	0.1000
2,3,7,8-TCDD	ng/l			<	0.0460	<	0.0990
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0460	<	0.0990
TETRACHLORODIBENZOFURANS	ng/l			<	0.0340	<	0.0400

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SPECIFIC CONDUCTANCE	umhos/cm		4968.0000		4311.0000		4550.7500
SULFATE	mg/l		1905.0000		1800.0000		1980.0000
TOTAL DISS SOLIDS	mg/l		11000.0000		3870.0000		4240.0000
TOTAL SUSP SOLIDS	mg/l	<	91.0000	<	10.0000	<	10.0000
DENSITY	g/mL		0.9772		1.0015		1.0000
pH	SU		7.6600		7.2500		7.6400
ALKALINITY	mg/l		111.0000		101.5000		104.7000
BROMIDE	mg/l	<	2.0000	<	2.0000		2.1250
CHLORIDE	mg/l		1040.0000		511.0000		6723.0000
FLUORIDE	mg/l	<	3.0000		1.4400		1.0650
IODIDE	mg/l	<	2.0000	<	2.0000	<	2.0000
NITROGEN, NO3 (AS N)	mg/l		7.6200		4.0050		2.8050
TOTAL ORGANIC CARBON	mg/l		1.1000		1.7800		1.1450
TOTAL ORGANIC HALOGENS	mg/l		0.0880		0.0663		0.0442
ORTHOPHOSPHATE (AS P)	mg/l	<	0.0200	<	0.0200	<	0.0200
BORON	mg/l		0.4290		0.3760	<	0.5000
CALCIUM	mg/l		681.0000		566.0000		646.5000
IRON	mg/l	<	0.4000	<	0.1375	<	0.5000
LITHIUM	mg/l		0.0950		0.0905	<	0.2000
MAGNESIUM	mg/l		181.0000		155.5000		155.5000
POTASSIUM	mg/l		4.8200		3.8850		5.0550
SILICA	mg/l		24.2700		24.7200		22.8400
SODIUM	mg/l		347.0000		282.0000		314.0000
FREON-113	mg/l	<	0.0050	<	0.0050	<	0.0050
CYANIDE	mg/l			<	0.0100	<	0.0100
SULFIDE	mg/l				5.3800	<	1.5000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
ANTIMONY	mg/l			<	0.0130	<	0.0130
ARSENIC	mg/l	<	0.0060	<	0.0130	<	0.0130
BARIUM	mg/l	<	0.0200		0.0090		0.0100
BERYLLIUM	mg/l	<	0.0100	<	0.0025	<	0.0030
CADMIUM	mg/l			<	0.0025	<	0.0030
CHROMIUM	mg/l	<	0.0025	<	0.0250	<	0.0250
COBALT	mg/l			<	0.0130	<	0.0130
COPPER	mg/l			<	0.0130	<	0.0250
LEAD	mg/l	<	0.0125	<	0.0130		0.0170
MERCURY	mg/l	<	0.0002	<	0.0020	<	0.0020
NICKEL	mg/l			<	0.0250	<	0.0250
SELENIUM	mg/l	<	0.0060	<	0.0130		0.0200
SILVER	mg/l	<	0.0028	<	0.0130	<	0.0130
THALLIUM	mg/l			<	0.0130	<	0.0130
TIN	mg/l			<	0.0250	<	0.0250
VANADIUM	mg/l				0.0500		0.0530
ZINC	mg/l			<	0.0500	<	0.0500
ACETONE	ug/l			<	10.0000	<	10.0000
ACETONITRILE	ug/l			<	50.0000	<	50.0000
ACROLEIN	ug/l			<	10.0000	<	10.0000
ACRYLONITRILE	ug/l			<	10.0000	<	10.0000
BENZENE	ug/l			<	5.0000	<	5.0000
DICHLOROBROMOMETHANE	ug/l			<	5.0000	<	5.0000
BROMOFORM	ug/l			<	5.0000	<	5.0000
METHYL BROMIDE	ug/l			<	10.0000	<	10.0000
2-BUTANONE	ug/l			<	10.0000	<	10.0000
CARBON DISULFIDE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
CARBON TETRACHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
CHLOROBENZENE	ug/l			<	5.0000	<	5.0000
CHLOROPRENE	ug/l			<	5.0000	<	5.0000
CHLOROETHANE	ug/l			<	10.0000	<	10.0000
CHLOROFORM	ug/l			<	5.0000	<	5.0000
METHYL CHLORIDE	ug/l			<	10.0000	<	10.0000
ALLYL CHLORIDE	ug/l			<	5.0000	<	5.0000
DIBROMOCHLOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMO-3-CHLOROPROPANE	ug/l			<	5.0000	<	5.0000
1,2-DIBROMOETHANE	ug/l			<	5.0000	<	5.0000
TRANS-1,4-DICHLORO-2-BUTENE	ug/l			<	5.0000	<	5.0000
DICHLORODIFLUOROMETHANE	ug/l			<	10.0000	<	10.0000
1,1-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
TRANS-1,2-DICHLOROETHYLENE	ug/l			<	5.0000	<	5.0000
1,2-DICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
CIS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
TRANS-1,3-DICHLOROPROPENE	ug/l			<	5.0000	<	5.0000
ETHYLBENZENE	ug/l			<	5.0000	<	5.0000
ETHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000
2-HEXANONE	ug/l			<	10.0000	<	10.0000
METHYL IODIDE	ug/l			<	5.0000	<	5.0000
METHACRYLONITRILE	ug/l			<	5.0000	<	5.0000
METHYLENE BROMIDE	ug/l			<	5.0000	<	5.0000
METHYLENE CHLORIDE	ug/l	<	5.0000	<	5.0000	<	5.0000
METHYL METHACRYLATE	ug/l			<	5.0000	<	5.0000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
4-METHYL-2-PENTANONE	ug/l			<	10.0000	<	10.0000
PROPIONITRILE	ug/l			<	20.0000	<	20.0000
STYRENE	ug/l			<	5.0000	<	5.0000
1,1,1,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
1,1,2,2-TETRACHLOROETHANE	ug/l			<	5.0000	<	5.0000
TETRACHLOROTEHYLENE	ug/l			<	5.0000	<	5.0000
TOLUENE	ug/l			<	5.0000	<	5.0000
1,1,1-TRICHLOROETHANE	ug/l	<	5.0000	<	5.0000	<	5.0000
1,1,2-TRICHLOROETHANE	ug/l			<	5.0000	<	5.0000
TRICHLOROETHYLENE	ug/l	<	5.0000	<	5.0000	<	5.0000
TRICHLOROFLUOROMETHANE	ug/l			<	5.0000	<	5.0000
1,2,3-TRICHLOROPROPANE	ug/l			<	5.0000	<	5.0000
VINYL ACETATE	ug/l			<	10.0000	<	20.0000
VINYL CHLORIDE	ug/l			<	10.0000	<	10.0000
XYLENE	ug/l			<	10.0000	<	10.0000
1,4-DIOXANE	ug/l			<	410.0000	<	410.0000
ISOBUTYL ALCOHOL	ug/l			<	320.0000	<	320.0000
ACENAPHTHENE	ug/l			<	10.0000	<	10.0000
ACENAPHTHYLENE	ug/l			<	10.0000	<	10.0000
ACETOPHENONE	ug/l			<	10.0000	<	10.0000
2-ACETYLAMINOFLUORENE	ug/l			<	10.0000	<	10.0000
4-AMINOBIPHENYL	ug/l			<	10.0000	<	10.0000
ANILINE	ug/l			<	10.0000	<	10.0000
ANTHRACENE	ug/l			<	10.0000	<	10.0000
ARAMITE	ug/l			<	10.0000	<	10.0000
1,2-BENZANTHRACENE	ug/l			<	10.0000	<	10.0000
3,4-BENZOFLUORANTHENE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
BENZO[K]FLUORANTHENE	ug/l			<	10.0000	<	10.0000
BENZO[GHI]PERYLENE	ug/l			<	10.0000	<	10.0000
BENZO[A]PYRENE	ug/l			<	10.0000	<	10.0000
BENZYL ALCOHOL	ug/l			<	20.0000	<	20.0000
BIS(2-CHLOROETHOXY)METHANE	ug/l			<	10.0000	<	10.0000
BIS(2-CHLOROETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-CHLORO-1-METHYLETHYL)ETHER	ug/l			<	10.0000	<	10.0000
BIS(2-ETHYLHEXYL)PHTHALATE	ug/l			<	10.0000	<	10.0000
4-BROMOPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
BENZYL BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
4-CHLOROANILINE	ug/l			<	10.0000	<	10.0000
CHLOROBENZILATE	ug/l			<	10.0000	<	10.0000
P-CHLORO-M-CRESOL	ug/l			<	10.0000	<	10.0000
2-CHLORONAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-CHLOROPHENOL	ug/l			<	10.0000	<	10.0000
4-CHLOROPHENYL PHENYL ETHER	ug/l			<	10.0000	<	10.0000
CHRYSENE	ug/l			<	10.0000	<	10.0000
DIALLATE	ug/l			<	10.0000	<	10.0000
DIBENZ[A,H]ANTHRACENE	ug/l			<	10.0000	<	10.0000
DIBENZOFURAN	ug/l			<	10.0000	<	10.0000
DI-N-BUTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,2-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,3-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
1,4-DICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
DCB	ug/l			<	20.0000	<	20.0000
2,4-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,6-DICHLOROPHENOL	ug/l			<	10.0000	<	10.0000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DIETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
P-(DIMETHYLAMINO)AZOBENZENE	ug/l			<	10.0000	<	10.0000
7,12-DIMETHYLBENZ[A]ANTHRACENE	ug/l			<	10.0000	<	10.0000
O-TOLIDINE	ug/l			<	10.0000	<	10.0000
A,A-DIMETHYLPHENETHYLAMINE	ug/l			<	10.0000	<	10.0000
2,4-DIMETHYLPHENOL	ug/l			<	10.0000	<	10.0000
DIMETHYL PHTHALATE	ug/l			<	10.0000	<	10.0000
1,3-DINITROBENZENE	ug/l			<	10.0000	<	10.0000
4,6-DINITRO-O-CRESOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROPHENOL	ug/l			<	50.0000	<	50.0000
2,4-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
2,6-DINITROTOLUENE	ug/l			<	10.0000	<	10.0000
DINOSEB	ug/l			<	10.0000	<	10.0000
DI-N-OCTYL PHTHALATE	ug/l			<	10.0000	<	10.0000
DIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
ETHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
FLUORANTHENE	ug/l			<	10.0000	<	10.0000
FLUORENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROBUTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROCYCLOPENTADIENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROETHANE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPHENE	ug/l			<	10.0000	<	10.0000
HEXACHLOROPROPENE	ug/l			<	10.0000	<	10.0000
INDENO(1,2,3-CD)PYRENE	ug/l			<	10.0000	<	10.0000
ISOPHORONE	ug/l			<	10.0000	<	10.0000
ISOSAFROLE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
METHAPYRILENE	ug/l			<	10.0000	<	10.0000
3-METHYLCHOLANTHRENE	ug/l			<	10.0000	<	10.0000
METHYL METHANESULFONATE	ug/l			<	10.0000	<	10.0000
2-METHYLNAPHTHALENE	ug/l			<	10.0000	<	10.0000
2-MEYTHLPHENOL	ug/l			<	10.0000	<	10.0000
3+4-METHYLPHENOL	ug/l			<	10.0000	<	10.0000
NAPHTHALENE	ug/l			<	10.0000	<	10.0000
1,4-NAPHTHOQUINONE	ug/l			<	200.0000	<	200.0000
1-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
2-NAPHTHYLAMINE	ug/l			<	10.0000	<	10.0000
O-NITROANILINE	ug/l			<	50.0000	<	50.0000
M-NITROANILINE	ug/l			<	50.0000	<	50.0000
P-NITROANILINE	ug/l			<	50.0000	<	50.0000
NITROBENZENE	ug/l			<	10.0000	<	10.0000
2-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROPHENOL	ug/l			<	10.0000	<	10.0000
4-NITROQUINOLINE-1-OXIDE	ug/l			<	20.0000	<	20.0000
N-NITROSODI-N-BUTYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIMETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPHENYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSODIPROPYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMETHYLETHYLAMINE	ug/l			<	10.0000	<	10.0000
N-NITROSOMORPHOLINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPIPERIDINE	ug/l			<	10.0000	<	10.0000
N-NITROSOPYRROLIDINE	ug/l			<	10.0000	<	10.0000
5-NITRO-O-TOLUIDINE	ug/l			<	10.0000	<	10.0000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
PENTACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROETHANE	ug/l			<	10.0000	<	10.0000
PENTACHLORONITROBENZENE	ug/l			<	10.0000	<	10.0000
PENTACHLOROPHENOL	ug/l			<	50.0000	<	50.0000
PHENACETIN	ug/l			<	10.0000	<	10.0000
PHENANTHRENE	ug/l			<	10.0000	<	10.0000
PHENOL (TOTAL)	ug/l			<	10.0000	<	10.0000
P-PHENYLENEDIAMINE	ug/l			<	10.0000	<	10.0000
2-PICOLINE	ug/l			<	10.0000	<	10.0000
PRONAMIDE	ug/l			<	10.0000	<	10.0000
PYRENE	ug/l			<	10.0000	<	10.0000
PYRIDINE	ug/l			<	10.0000	<	10.0000
SAFROLE	ug/l			<	10.0000	<	10.0000
1,2,4,5-TETRACHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,3,4,6-TETRACHLOROPHENOL	ug/l			<	10.0000	<	10.0000
O-TOLUIDINE	ug/l			<	10.0000	<	10.0000
1,2,4-TRICHLOROBENZENE	ug/l			<	10.0000	<	10.0000
2,4,5-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
2,4,6-TRICHLOROPHENOL	ug/l			<	10.0000	<	10.0000
1,3,5-TRINITROBENZENE	ug/l			<	10.0000	<	10.0000
ALDRIN	ug/l			<	0.0500	<	0.0500
ALPHA-BHC	ug/l			<	0.0500	<	0.0500
BETA-BHC	ug/l			<	0.0500	<	0.0500
DELTA-BHC	ug/l			<	0.0500	<	0.0500
LINDANE	ug/l			<	0.0500	<	0.0500
CHLORDANE	ug/l			<	0.1000	<	0.1000
TDE	ug/l			<	0.1000	<	0.1000

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
DDE	ug/l			<	0.1000	<	0.1000
DDT	ug/l			<	0.1000	<	0.1000
DIELDRIN	ug/l			<	0.1000	<	0.1000
ENDOSULFAN I	ug/l			<	0.0500	<	0.0500
ENDOSULFAN II	ug/l			<	0.1000	<	0.1000
ENDOSULFAN SULFATE	ug/l			<	0.1000	<	0.1000
ENDRIN	ug/l			<	0.1000	<	0.1000
ENDRIN ALDEHYDE	ug/l			<	0.1000	<	0.1000
HEPTACHLOR	ug/l			<	0.0500	<	0.0500
HEPTACHLOR EPOXIDE	ug/l			<	0.0500	<	0.0500
ISODRIN	ug/l			<	0.0500	<	0.0500
KEPONE	ug/l			<	0.2500	<	0.2500
METHOXYCHLOR	ug/l			<	0.5000	<	0.5000
TOXAPHENE	ug/l			<	2.0000	<	2.0000
AROCLOR 1016	ug/l			<	1.0000	<	1.0000
AROCLOR 1221	ug/l			<	2.0000	<	2.0000
AROCLOR 1232	ug/l			<	1.0000	<	1.0000
AROCLOR 1242	ug/l			<	1.0000	<	1.0000
AROCLOR 1248	ug/l			<	1.0000	<	1.0000
AROCLOR 1254	ug/l			<	1.0000	<	1.0000
AROCLOR 1260	ug/l			<	1.0000	<	1.0000
DIMETHOATE	ug/l			<	0.5000	<	0.2500
DISULFOTON	ug/l			<	0.5000	<	0.2500
FAMPHUR	ug/l			<	0.5000	<	0.2500
METHYL PARATHION	ug/l			<	0.5000	<	0.2500
PARATHION	ug/l			<	0.5000	<	0.2500
PHORATE	ug/l			<	0.5000	<	0.2500

TABLE D-18C-7
GMP WATER QUALITY DATA TO DATE
FOR
WQSP-6a

Parameter	Unit		Value		Value		Value
			Round		Round		Round
			1		2		3
SULFOTEPP	ug/l			<	0.5000	<	0.2500
THIONAZIN	ug/l			<	0.5000	<	0.2500
O,O,O-TRIETHYL PHOSPHOROTHIOATE	ug/l			<	0.5000	<	0.2500
2,4-D	ug/l			<	1.0000	<	1.0000
2,4,5-T	ug/l			<	1.0000	<	1.0000
2,4,5- TP	ug/l			<	1.0000	<	1.0000
HEXACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0230	<	0.0350
HEXACHLORODIBENZOFURANS	ng/l			<	0.0076	<	0.0130
PENTACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0430	<	0.0490
PENTACHLORODIBENZOFURANS	ng/l			<	0.0140	<	0.0360
2,3,7,8-TCDD	ng/l			<	0.0110	<	0.0330
TETRACHLORODIBENZO-P-DIOXINS	ng/l			<	0.0110	<	0.0330
TETRACHLORODIBENZOFURANS	ng/l			<	0.0070	<	0.0240