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Quality Assurance Project Plan Requirements for Sampling and Analysis

Environmental Restoration Project

March 1996

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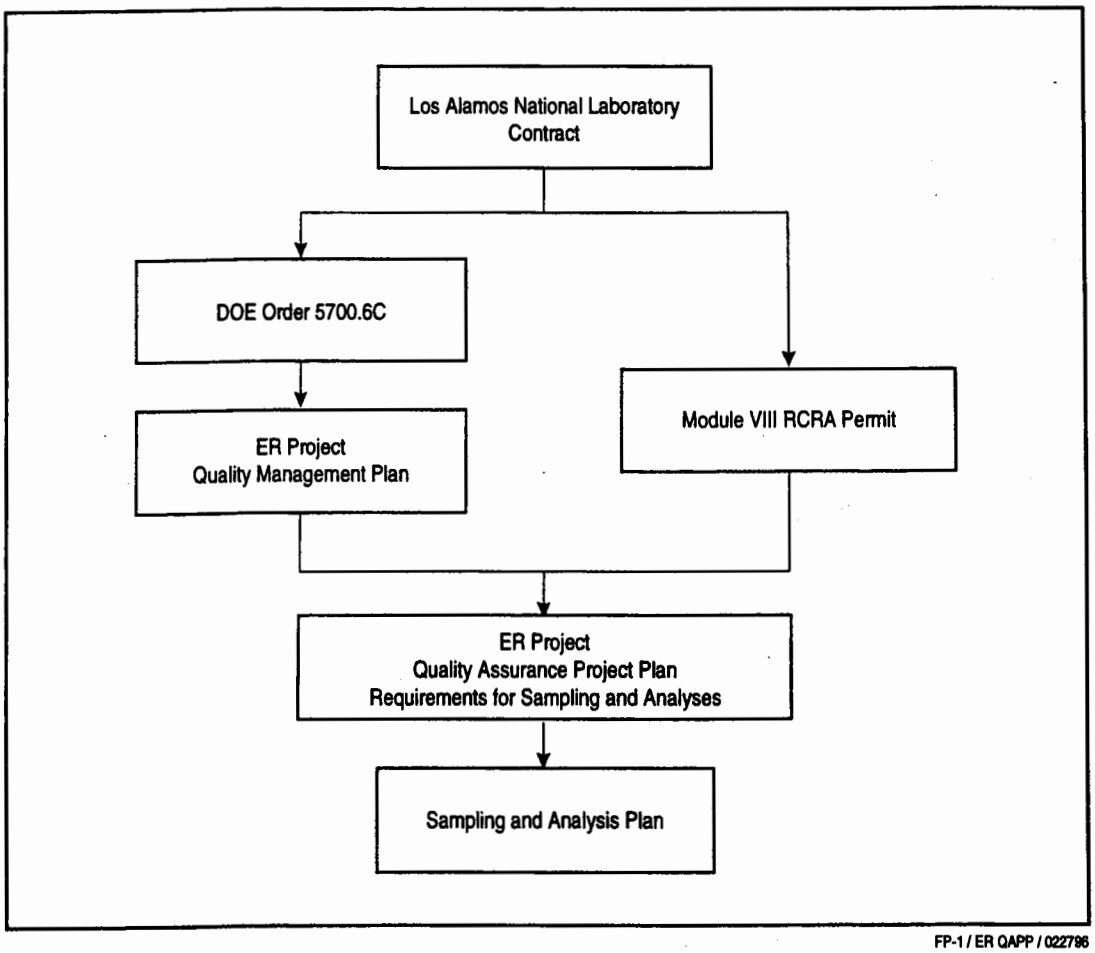
1.0 DOCUMENT PURPOSE AND CONTEXT

This Environmental Restoration (ER) Quality Assurance Project Plan (QAPP) requirements document supersedes the Generic Quality Assurance Project Plan prepared by the Los Alamos National Laboratory (LANL) ER Project in 1991 (LANL 1991, 0412). This document is tiered to the US Environmental Protection Agency's (EPA's) Region VI "Interim Draft Requirements for Quality Assurance Project Plans" (EPA QA/R-5, 1994, 52288).

This document is part of the ER Project Quality Assurance (QA) Program hierarchy of documents (Figure P-1). This document details quality requirements that must be addressed in ER Project sampling and analysis plans (SAPs). Use of this document in conjunction with the ER "Sampling and Analysis Plan Outline and Crosswalk" (Lewis et al., 1996, 52242) will facilitate the development of SAPs. It is intended that compliance with this document will allow for site-specific flexibility in planning and implementing environmental activities and will cause quality and consistency to be designed into ER Project environmental data collection activities.

The requirements presented in this document apply to all ER SAPs whether they are stand alone documents or part of other documents. Those other documents include Resource Conservation and Recovery Act (RCRA) facility investigation (RFI) plans and reports, expedited cleanup (EC) plans, voluntary corrective action (VCA) plans, closure plans, etc.



FP-1 / ER QAPP / 022796

Figure P-1. Document hierarchy.

A1 APPROVALS

1. NAME: J. Jansen

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TITLE: Environmental Restoration Quality Assurance Officer

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3. NAME: B. Garcia

TITLE: New Mexico Environment Department, Hazardous and Radioactive Materials Bureau, Bureau Chief

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TITLE: Field Unit One Project Leader

SIGNATURE: GR Allen DATE 26 Feb 96

2. NAME: T. Glatzmaier

TITLE: Project Consistency Team Manager

SIGNATURE: T. Mann for T.G. DATE 2-27-96

3. NAME: G. Gould

TITLE: Field Unit Two Project Leader

SIGNATURE: Billie M Wheat for TEG DATE 2/26/96

4. NAME: B. Martin

TITLE: Field Unit Three Project Leader

SIGNATURE: B. Martin DATE 2/26/96

5. NAME: D. McInroy

TITLE: Regulatory Compliance Manager

SIGNATURE: DM DATE 2/26/96

6. NAME: A. Pratt

TITLE: Field Unit Four Project Leader

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7. NAME: C. Rofer

TITLE: Field Unit Five Project Leader

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8. NAME: M. Salazar

TITLE: Decommissioning Project Leader

SIGNATURE: _____ DATE 2/26/96

9. NAME: S. Bolivar

TITLE: Field Support Facility Leader

SIGNATURE: _____ DATE 2/26/96

10. NAME: T. Taylor

TITLE: Department of Energy, Los Alamos Area Office, ER Program Manager

SIGNATURE: _____ DATE 3/1/96

TABLE OF CONTENTS

PREFACE i
1.0 DOCUMENT PURPOSE AND CONTEXT i
2.0 DOCUMENT STRUCTURE ii
SECTION A A-7
A1 APPROVALS A-1
A1.1 Concurrence A-3
A2 TABLE OF CONTENTS A-7
A3 DISTRIBUTION LIST A-9
A4 PROJECT/TASK ORGANIZATION A-10
A5 PROBLEM DEFINITION A-10
A6 PROJECT/TASK DESCRIPTION A-12
A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA A-12
A8 PROJECT NARRATIVE A-14
A9 TRAINING/CERTIFICATION A-14
A9.1 Training A-14
A9.2 Certification A-14
A10 DOCUMENTATION AND RECORDS A-14
SECTION B B-1
B1 SAMPLING PROCESS DESIGN B-1
B1.1 Environmental Sampling Plan Design B-1
B1.2 Assessment and QC Sampling Plan B-2
B2 SAMPLING METHODS REQUIREMENTS B-2
B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS B-3
B3.1 Sample Management Office (SMO) B-4
B3.2 Field Packaging and On-Site Measurements B-4
B3.3 Sample Volumes, Containers, Holding Times, And Preservatives B-5
B4 ANALYTICAL METHODS REQUIREMENTS B-5
B5 QUALITY CONTROL REQUIREMENTS B-7
B5.1 Sampling B-8
B6 INSTRUMENT/EQUIPMENT PURCHASING, TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS B-8
B7 INSTRUMENT CALIBRATION AND FREQUENCY B-9
B7.1 Field Equipment B-9
B7.2 Laboratory Equipment B-10
B8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES B-10
B9 DATA ACQUISITION REQUIREMENTS (NONDIRECT MEASUREMENTS; REQUIREMENTS FOR USING ARCHIVED DATA AND NONMEASUREMENT DATA) B-11
B10 DATA MANAGEMENT B-11
SECTION C C-1
C1 ASSESSMENTS AND RESPONSE ACTIONS C-1
C1.1 Internal Assessment C-1
C1.1.1 Field Unit Assessments C-1
C1.1.2 Corrective Action C-1
C1.2 Oversight of Analytical Laboratories C-1
C1.2.1 Laboratory Assessments C-1
C1.2.2 Analytical Laboratories Performance Measurement C-2
C1.2.3 Data Package Assessment C-2
C1.2.4 Monitoring and Tracking Administrative Indicators C-2
C1.2.5 Problem Resolution C-2
C1.3 ER Project Peer Reviews C-3
C1.4 Readiness Reviews C-3
C2 REPORTS TO MANAGEMENT C-3
C2.1 Project Status C-3
C2.2 Quality Assurance Reports C-3

A3 DISTRIBUTION LIST

No.	Name	Title
1.	J. Jansen	Environmental Restoration Project Manager
2.	L. Souza	Quality Assurance Officer
3.	T. Taylor	Department of Energy, Los Alamos Area Office, ER Program Manager
4.	G. Allen	Field Unit One Project Leader
5.	T. Glatzmaier	Project Consistency Manager
6.	G. Gould	Field Unit Two Project Leader
7.	B. Martin	Field Unit Three Project Leader
8.	D. McInroy	Regulatory Compliance Manager
9.	A. Pratt	Field Unit Four Project Leader
10.	C. Rofer	Field Unit Five Project Leader
11.	M. Salazar	Decommissioning Project Leader
12.	S. Bolivar	Field Support Facility Leader
13.	B. Garcia	New Mexico Environment Department, Hazardous and Radioactive Material Bureau, Bureau Chief
14.	R. Kern	New Mexico Environment Department, Hazardous and Radioactive Material Bureau, Program Manager
15.	B. Hoditschek	New Mexico Environment Department, Hazardous and Radioactive Material Bureau, Program Manager
16.	B. Driscoll	Environmental Protection Agency, Region VI

Key planning participants responsible for defining the problems and developing the problem-solving approach should be identified early in the planning process. This core planning team typically includes

- FPLs or their designees;
- field team leaders (FTLs) and selected field personnel;
- Earth Sciences Council (ESC) personnel (geologists, hydrologists, geochemists); and
- Decision Support Council (DSC) personnel (chemists, ecological and human health risk assessment specialists, statisticians).

The core team will contact others, as necessary, to provide historical, technical and regulatory information.

The results of the planning process shall be documented in a SAP. SAPs may be prepared as stand-alone documents or as addenda to existing work plans, or they may be incorporated into corrective action plans or RFI reports. Problem definitions will be documented in the SAP by providing

- a clear statement of the question or questions to be answered by the data to be collected; and
- a clear statement of the decision or decisions for which these answers are required, including anticipated alternative courses of action.

The scope of activities and documentation that address the requirements of this document will be commensurate with the importance of the decisions to be based on the data supporting those decisions. The SAP or the document to which it is attached must provide enough information so that a technically trained reader can understand the activity's historical and regulatory context as well as its objectives. In all cases, the SAP must present either explicitly or by reference the following:

- a physical/historical description of the site and the problem including, as appropriate, a summary of existing information such as
  - engineering drawings and site process histories;
  - a site conceptual model describing known and potential releases and existing or potential exposure scenarios;
  - a list of potential or known contaminants; and
  - a list or summary of existing data.
- identification of practical constraints, such as physical limitations on sample collection, scheduling constraints imposed by the need to coordinate with corrective actions, limitations of available measurement technology, and budgetary constraints; and

- limiting contamination of samples to insignificant levels; and
- maintaining the desired degree of data comparability to allow for statistically valid evaluation or pooling of the data.

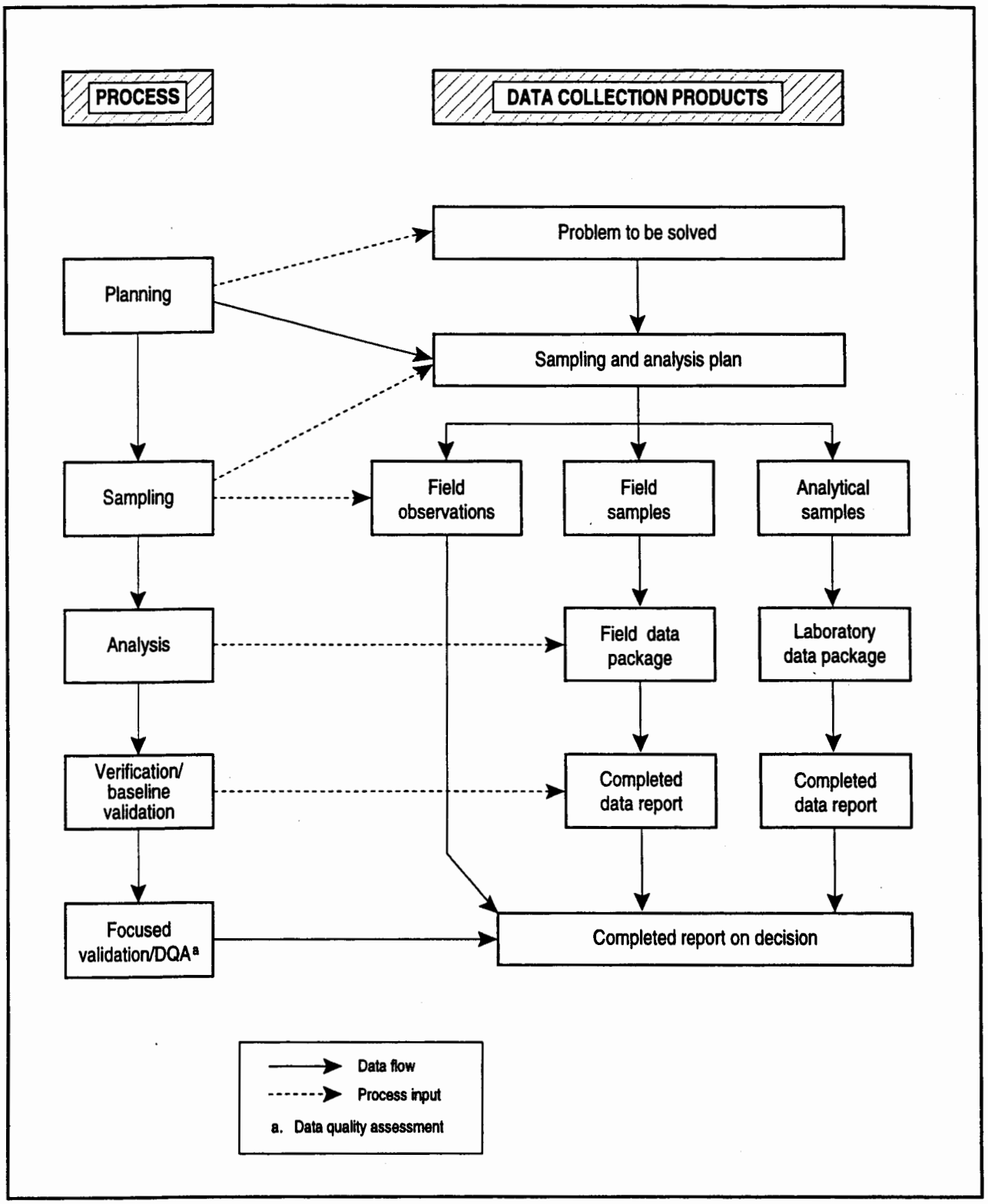
The planning process will result in a list of criteria that are expected to increase the likelihood that data of the right type, quantity, and quality are collected to support the decision(s). In addition to the items listed in Section A5 of this document, such a list should include the following types of descriptors:

- identification of a focused list of environmental variables that must be measured or collected (e.g., analytes, concentrations/ radioactivities, physicochemical parameters, risk exposure model parameters;
- specification of the data reporting units;
- specification of decision or action levels, e.g., screening action levels (SALs) or bases for deriving them (e.g., risk-based criteria);
- geographical boundaries of each PRS or PRS aggregate;
- subpopulations (e.g., geologic strata or risk-based exposure units);
- temporal considerations that affect the time during which data can be collected; and
- sample matrices of interest.

The SAP must document in detail the ways in which the collected data will be summarized and used to make the decisions. Possible uses of measurements include, but are not limited to

- comparison of individual observations with prespecified thresholds, such as background upper tolerance level (UTLs), SALs, or PRGs; and
- calculation of 95% upper confidence bounds for the mean of a measured parameter within a prespecified area or volume, for comparison with thresholds such as PRGs.

The consequences of making an incorrect decision should also be considered. When appropriate, quantitative limits on acceptable decision errors should be specified. The scientific and statistical assumptions that form the basis of the SAP may include contaminant transport models, exposure models, and statistical models to support hypothesis testing or estimation (based on components of variance from sampling and measurement). The planning process will ultimately result in selection of a cost-effective sampling and analysis plan that meets the applicable quality criteria. See Section B1 of this document for more specifics on SAP design and selection.



F A-3 / ER QAPP / 022796

Figure A-1. Project data flow for the Environmental Restoration Project.

B1 SAMPLING PROCESS DESIGN

Alternative sampling and analysis options will be evaluated during planning and the most cost-effective design that is expected to meet the planning specifications will be selected. Cost-effectiveness may be determined through professional judgment or through a cost-benefit analysis. By selecting a particular sampling design, the type and number of samples, and the means of allocating samples, is defined. Specific sampling locations (and/or frequency of sample collection) are selected along with sample acquisition methods, measurement methods, and other procedures that will be used to collect and analyze the samples. The type and number of quality assessment/quality control samples to be collected in the field must also be determined, and the frequency and/or location for these samples documented.

SAP documentation requirements are specified in the following sections. The SAP design must be recorded in the appropriate document (i.e., RFI work plan, RFI report, accelerated cleanup plan, etc.). The SAP outline (LANL1996, 52242), which details additional SAP requirements, is to be followed in developing the SAP.

B1.1 Environmental Sampling Plan Design

All of the information listed below, as appropriate to the design, must be documented in enough detail to make the SAP third-party implementable.

- the number, or frequency of collection, for each type of sample (e.g., composite, grab, integrated) to be collected;
- the sampling network design (e.g., rectangular or triangular grid, stratification) and the assumptions underlying the design;
- the locations of the sampling points (preferably marked on a map);
- when field measurement methods are used, the techniques and/or guidelines to be followed in selecting sampling points, a description of or reference to the measurement technique/method to be used, and a description of how field screening results are to be used;
- if sample point selection will be made during field activities, the method(s) to be used to locate sampling points in the field, including specifics on how locational data are to be collected, stored, and transmitted;
- a description of the portion of each medium that will be collected for analysis;
- specification of nonmeasurement data required as inputs to solving the problem;
- references to all administrative procedures and SOPs used to carry out the work under the SAP;

hydro-punch sampling or for collecting samples for volatile organics in the different media, because each medium could require different volumes and containers);

- size and type of sampling equipment appropriate for collecting the desired samples. This is especially important for analytical methods that require special containers such as air sampling, certain volatile organics analytical methods, and certain on-site measurements;
- decontamination (see LANL-ER-SOP-1.08) that must be performed on nondisposable sampling equipment prior to and between uses. Wash water and other wastes generated during the sampling operation must be managed and disposed of in accordance with LANL-ER-AP-05.3;
- waste minimization (including the minimization of decontamination wastes); and
- constraints on the sampling events that might significantly affect the projected time or costs (e.g., inclement weather or threats to endangered species).

These requirements should be summarized to include references to the procedures that will be used to conduct the sampling. Where existing SOPs or other official guidance provide adequate documentation of any of these required criteria, those documents shall be cited in the SAP. For example, LANL-ER-SOP-1.02 addresses the requirements for sample containers, preservatives, sample volumes, and holding times; routine sampling procedures are documented in the ER Project SOPs, Chapter 6, "Sampling Techniques." Additional guidance is presented in Appendix II for selecting sampling methods and equipment.

If all site-specific requirements are not adequately addressed by reference, then the requirements shall be documented in the SAP by developing and referencing new SOPs or revised SOPs. Otherwise the requirements must be included in the SAP by incorporating the equivalent SOP requirements. For example, implementation requirements and support facilities needed to ensure safety and work of adequate quality should be specified in the SAP. Where site-specific performance requirements are necessary for sampling operations, those requirements should be written into the SAP. For those tasks that might be useful to more than one field unit, developing new SOPs is encouraged in lieu of writing instructions into the SAP.

Ultimate authority and responsibility for field operations lies with the responsible FPL. However, responsibility for corrective actions in the field that address deviations from SAPs and other field-work-related contingencies may rest with the cognizant field team leaders who report to the FPL. When possible, corrective actions should be anticipated and delineated in the SAP.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

All personnel must follow the SOPs addressing sample handling and custody (ER Project SOPs, Chapter 1, "General Instructions"). Those SOPs must be referenced in the SAP. In cases where deviations from an SOP are planned, the deviations must be

on-site measurement methods is included in the Department of Energy (DOE) document "Guidance for Planning On-Site Measurements" (DOE 1995, 52240).

In addition to the above requirements, all ER Project samples must be classified prior to shipment as hazardous or nonhazardous pursuant to International Air Transportation Association, Department of Transportation regulations (see 49 CFR 171-173) and EPA guidance (EPA 1987, 11654). LANL-ER-SOP-1.03 addresses the issues of determining the hazard status, packaging, and shipping of ER Project samples and provides more specific direction on sample packaging and transport.

B3.3 Sample Volumes, Containers, Holding Times, And Preservatives

Requirements for selecting sample volumes, containers, holding times, and preservatives for samples subjected to routine analyses are presented in LANL-ER-SOP-1.02. Routine analyses are addressed in detail in Section B4 of this document. Sample preservation and holding time requirements for nonroutine analytical measurements must be specified directly in the SAP.

B4 ANALYTICAL METHODS REQUIREMENTS

The SAP must include the following information:

- analytical and other measurement methods to be used. This includes sample preparation techniques (e.g., extraction, cleanup, digestion, etc.) and special equipment (e.g., instrument sample preparation equipment critical to the analyses);
- any decontamination procedures needed to prevent compromising the representativeness of the sample and analyses; and
- specific performance criteria for the above bulleted items.

The analytical services contracts, which include SOWs (LANL 1995, 49738) for analytical services, were developed for the ER Project to meet most users' needs in a cost-effective manner. Those SOWs can be especially appropriate for screening assessments and other types of investigations requiring broad-scan methods or very rigorous QC. They include lists of the analytes grouped into standard ER Project analyte suites such as volatile organics and metals (see also Appendix III). It is unnecessary to specify in a SAP any routine analytical requirements that are addressed in the analytical laboratory SOWs (LANL 1995, 49738). The preferred method of specifying which analytical methods will be used is by summarizing them in a table by analytical method number or, when SOW-related analytical services are used, by reference to the analytical laboratory SOWs (LANL 1995, 49738).

Analytical method selection must be based on the requirements of the decision to be made. These decisions are established during the planning process (see Sections A5–A7 of this document). The SAP requirements for analytical methods must reflect the following considerations:

- required analytical information (e.g., analyte list, including whether determinations will be made for total, soluble, extractable, isotopic, volatile species, etc., and how the data will be used);
- sensitivity;

  - spikes (matrix, surrogate, tracers/carriers, etc.)
  - QC samples (laboratory control sample, site-specific performance evaluation materials, etc.); and
- special analytical conditions that require different sample handling, preparation, or analytical procedures.

If standard analytical methods are to be followed, the methods may be cited in the SAP; otherwise the specifications in the SAP must be detailed enough to allow any qualified analyst to repeat the specified work using similar equipment.

Each FPL is ultimately responsible for data quality in his/her respective field unit. Additional information defining the options that need to be considered when selecting nonroutine analytical methods is available in the guidance for analytical method selection in Appendix IV. These options are specific to the type of analytical method needed. The selected options must be specified in the SAP.

B5 QUALITY CONTROL REQUIREMENTS

This section provides the quality control requirements for sampling, analyses, and other measurements (e.g., land surveys and biological assessments that must be performed routinely). The approach at LANL is to tailor QC activities to site-specific needs through planning and eliminating unnecessary QC checks.

SAPs should be designed to assess the major components of total study error to enable the final evaluation of whether environmental data are of sufficient quality to support the related decisions. The QC requirements must be designed to provide measurement error information that can be used to initiate corrective actions that limit the total measurement error. Consequently, SAPs must

- describe the QC samples and procedures associated with sampling and measurement,
- list specific QC checks that are required for each type of sampling and measurement data to be collected. The list must include
  - the frequencies of the control checks, and
  - the required acceptance criteria for each QC check.

The SAP must also provide

- as necessary, procedures for calculating QC statistics; a reference to this document's glossary (see Precision and Bias) might suffice,
- an explanation delineating how contingencies such as missing data, nondetects and out-of-range data will be addressed (see also Section D3 of this document), and
- anticipated corrective actions associated with failure of sampling or measurement systems to meet acceptance criteria.

delineated in SOPs, the SAP must define the requirements or cite manufacturers' maintenance and calibration schedules. When maintenance and calibration requirements that exceed those recommended by the manufacturer are deemed appropriate, and such requirements are not delineated in an SOP, they must be stated explicitly in the SAP.

Service contracts may provide a vehicle for routine preventive maintenance and emergency repair service. In such cases, actions taken by an instrument service representative shall be documented in the records for that instrument.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

Equipment designated for use in ER Project work plans shall be specified to meet site-specific planning specifications. Measuring and testing equipment used in the field or an analytical laboratory must be controlled by formal calibration procedures, which are required for proper operation of equipment and instruments. If available and applicable, instrument manufacturer directions for calibration may be cited instead of repeating them in ER Project documents. All calibration standards shall be traceable to nationally recognized standards such as those from the National Institute of Standards and Technology, unless such traceability is inappropriate or not possible. If traceability is inappropriate or not possible, the manner in which the suitability of calibration standards is determined must be stated in the SAP.

B7.1 Field Equipment

Field equipment must be properly calibrated and charged, as appropriate, and must be in good general working condition before the beginning of each day of use. ER Project SOPs and SAPs specify the required checks and calibration for each type of field equipment. These requirements include the frequencies of checks and calibrations necessary to ensure that operability is acceptable. Field equipment that does not meet calibration requirements shall be taken out of service until acceptable performance can be verified. Nonoperational field equipment shall also be removed from service and may be returned to the supplier for replacement. Maintenance records must be maintained for each field instrument according to a unique number affixed to the instrument used to facilitate tracking of instrument records. The unique serial number for each instrument shall be used on all related documentation concerning that instrument. These records should be reviewed before equipment use to ensure that maintenance and calibration are current.

All instruments used for environmental investigations must be properly protected against inclement weather as needed.

Logbooks specific to individual equipment items shall be used to record the

- equipment identifier;
- inspection, maintenance, and calibration action(s) performed;
- trigger(s) for the maintenance, calibration, or inspection action(s);
- identity of each person performing the work;

B9 DATA ACQUISITION REQUIREMENTS (NONDIRECT MEASUREMENTS; REQUIREMENTS FOR USING ARCHIVED DATA AND NONMEASUREMENT DATA)

If archived data or nonmeasurement data (e.g., interviews, maps, spreadsheets, computer data bases, calculations) are to be used in decision-making, the acceptability of the data shall be assessed on a case-by-case basis. The data acceptability may be confirmed by comparing data from more than one source or by corroborating the data through additional data collection. Information received through interviews shall be documented with written concurrence by the interviewee. For numerical data, prior validations shall be reviewed to assess the technical validity of the data as well as their suitability for use in making decision(s).

The responsible FPL shall determine the level of effort to be used in the data review. The effort will be commensurate with the amount of information available and the importance of the data relative to decision(s).

It could be important to prepare a pedigree for data of interest that describes the procedures used to collect the data and the qualifications of personnel who collected the data. The FPL or designee shall determine the need for, and method of, documenting a data pedigree.

B10 DATA MANAGEMENT

Chapter 5 of the IWP (LANL 1995, 52009) presents LANL's approach to data and records management. Following this approach, electronic data are stored in FIMAD and all other records are stored in the RPF. Data and records management requirements not specified in Chapter 5 of the IWP, applicable SOPs, or applicable SOWs must be specified in the SAP. See also Section A10 of this document.

Figure A-1 illustrates the flow of data generated for the ER Project. Results obtained from field instruments, field measurements, and field laboratories are verified and validated in the field to permit decisions to be made rapidly. The criteria and process for these reviews are discussed in Section D of this document. The results of radiological screening conducted in the field or in a mobile radiological van should be documented and sent along with the samples to the SMO.

Manually recorded data are recorded in accordance with LANL-ER-SOPs-1.04 and 3.12. They are reviewed by the field team as required by LANL-ER-SOPs 1.01, 1.04, and 3.12. Data that are transferred electronically are not subject to this review. However, the portion of the data that will be manually entered into the database (e.g., some nonroutine and field analytical methods, field notes, and other data recorded on forms in the field and then entered into FIMAD) must be reviewed for data entry errors. Field records, even if rendered illegible, must be kept as permanent records and may not be discarded.

Data generated as a result of analytical services by internal or contract laboratories must be submitted to the SMO, which is responsible for routine data verification and baseline validation as defined in Section D1 of this document. Nonroutine data verification/validation is the responsibility of the field unit team. Upon completion of the data verification/baseline validation process (see Section D1 of this document), the data must be transferred to accessible FIMAD files. Data entries include any qualifying flags assigned during baseline validation.

C1 ASSESSMENTS AND RESPONSE ACTIONS

The following sections provide a summary of the assessment activities required by the ER Project. The FPLs/DPLs and quality assurance officer (QAO) are responsible for tracking the results of assessments and response actions to ensure that deficiencies are corrected in a timely manner. The QAO is responsible for identifying the personnel who participate in planned assessments, surveillances, etc., and ensuring that they are qualified to implement those evaluations.

Assessments planning includes delineation of responsibilities and reporting authorities. The manner in which evaluation results will be reported, and to whom they will be reported, are determined during planning for the evaluation. Schedules for preliminary and follow-up interviews, meetings, etc., are decided in advance of the evaluation and designed to adversely affect work schedules as little as possible.

C1.1 Internal Assessment

The process by which the ER Project assesses systems (programmatic assessments) and performance is described in LANL-ER-QP-01.5Q. System assessments provide an effectiveness evaluation of systems established to ensure the quality of project activities. Performance assessments provide feedback on the effectiveness of activities in meeting ER Project objectives.

C1.1.1 Field Unit Assessments

The ER Project uses self-assessments and formal, independent field assessments to assess compliance with the SOPs identified in work plans, RFI reports, site characterization analyses (SCAs), ECs, closure plans, SAPs, etc., and associated QA documents (including this document). The FPLs/DPLs are responsible for determining the number and types of assessments to be conducted and for arranging for their implementation. The number, frequency, and purpose of each assessment must be specified in the SAP. At a minimum, assessments should review the processes used in the field to record information about each sample taken, control the chain of custody, determine the locations of sampling points, implement the specified sample collection methods, and implement the specified procedures for sample handling.

C1.1.2 Corrective Action

Deficiencies identified during assessments are documented in accordance with LANL-ER-QP-1.04Q. Corrective action requests are issued to the FPL/DPL to identify, document, and implement the necessary corrective actions.

C1.2 Oversight of Analytical Laboratories

C1.2.1 Laboratory Assessments

The performance of LANL's analytical chemistry and contract laboratories, including mobile analytical laboratories are assessed prior to acceptance for use and annually. These assessments are typically performed under the Albuquerque DOE FSMP Program, by LANL representatives, or through audits by other organizations (DOE, EPA, and other DOE management and operations contractors).

C1.3 ER Project Peer Reviews

ER Project plans and reports are peer-reviewed in accordance with LANL-ER-AP-01.3. This procedure provides for selecting appropriate personnel to conduct reviews and for formal comment resolution.

C1.4 Readiness Reviews

Before performing selected field activities, a readiness review is conducted in accordance with LANL-ER-AP-5.1. Implementing this procedure ensures that field work complies with applicable directives, guidance, SOPs, administrative requirements, and applicable regulations.

C2 REPORTS TO MANAGEMENT

C2.1 Project Status

Periodic reports are generated to describe ER Project status and to satisfy the requirements of Hazardous and Solid Waste Amendments (HSWA) Module VIII of the RCRA permit. The FPL or DPL is responsible for identifying the types of reports and frequencies in their respective SAPs or project plans. More detailed descriptions of RFI, corrective measures study/corrective measures implementation (CMS/CMI), voluntary corrective action (VCA) and EC plans and reports are provided in Chapter 3 of the IWP (LANL 1995, 52009).

C2.2 Quality Assurance Reports

The results of QA assessment activities identified in Sections C1.1 and C1.2 of this document are assembled, summarized, and distributed to the ER Project management team on a quarterly basis. These reports describe significant quality problems, recommend solutions, and identify personnel responsible for resolving the problems.

D1 DATA REVIEW: VERIFICATION AND BASELINE VALIDATION

All data generated by ER Project data collection activities will undergo a data review process that accomplishes two goals. First, "data verification" assures that needed data are available for further evaluation, assures that contract, or other, specifications have been met (or noted where not met), and provides the information needed for prompt and appropriate payment for analytical services. Second, "baseline validation" attaches qualifiers to data that do not meet specifications and provides information on potential deficiencies of that data. Reason codes for the qualifiers are also assigned to data to help users understand why a qualifier was added and the potential impacts of the data deficiency. The product of this first process is a report in FIMAD that can be used, as is, for data quality assessment (DQA) (see Section D3 of this document) and, as necessary, to focus further validation efforts. See Figure D-1 for a portrayal of the data verification/baseline validation process and Figure D-2 for a flow diagram that shows where the process fits into the entire data collection process.

For routine analytical services (RAS), the verification and baseline validation processes are carried out simultaneously. Those processes make use of a checklist for data completeness and compliance that is based on the routine analytical contracts, and that use standard validation qualifiers based on the commonly accepted contract laboratory program (CLP), "CLP Functional Guidelines" for review of analytical data. During this process, missing items are obtained from the laboratory that generated the data and any required corrections to erroneous data are made. These error corrections include both problems with compliance and problems with data entry into FIMAD.

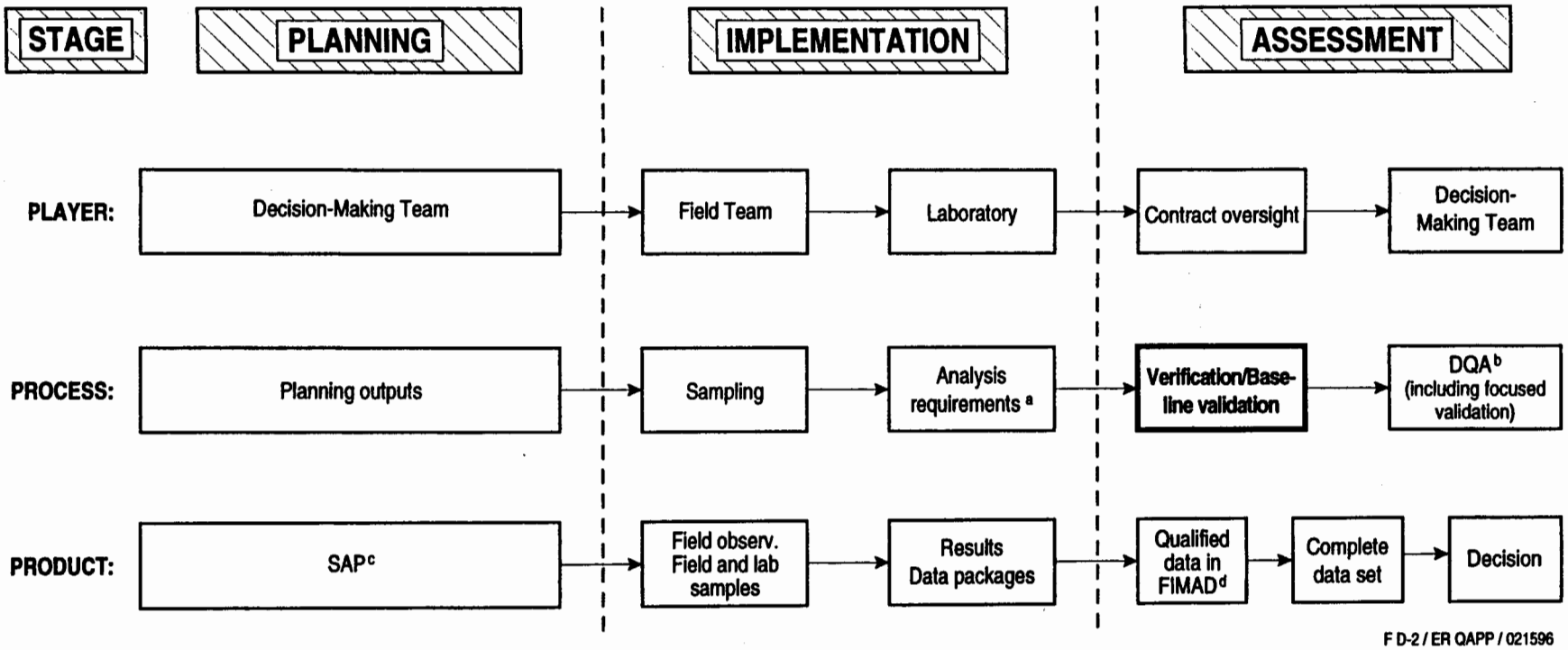
For routine analytical services, the SAP must state that the "LANL ER Checklist and Criteria for Verification and Baseline Validation" (LANL 1995, 52241), including data qualifiers and reason codes, will be used for verification and baseline validation. Forms and checklists may be provided for clarification, based on the analytical services used, e.g., organics, inorganics, high explosive (HE), radiochemistry, or commonly used mobile laboratory SOWs.

If known, the SAP should identify anticipated needs for focused validation (see Section D2 of this document). For example, when petroleum hydrocarbons are anticipated to be an interference in semivolatile analyses, the SAP should specify that the chromatograms will be reviewed to assess the effect or potential effect of interferences on the reported data.

For nonroutine analytical services (NRAS), which include off-site analytical services, field analyses, and field measurements, verification criteria must be stated in the SAP or SOP. These verification, or acceptance, criteria are most efficiently used when they are provided as a checklist or data review SOP. The qualifiers that have been stipulated for the routine analytical services should be used to provide consistent data qualifiers within FIMAD. The SOP must also provide reason codes that are appropriate for the specific analyses. In the case of NRAS, the verification and baseline validation criteria should be combined. This will create a single set of requirements that must be met. Data failing these requirements will be qualified and reason codes will be attached.

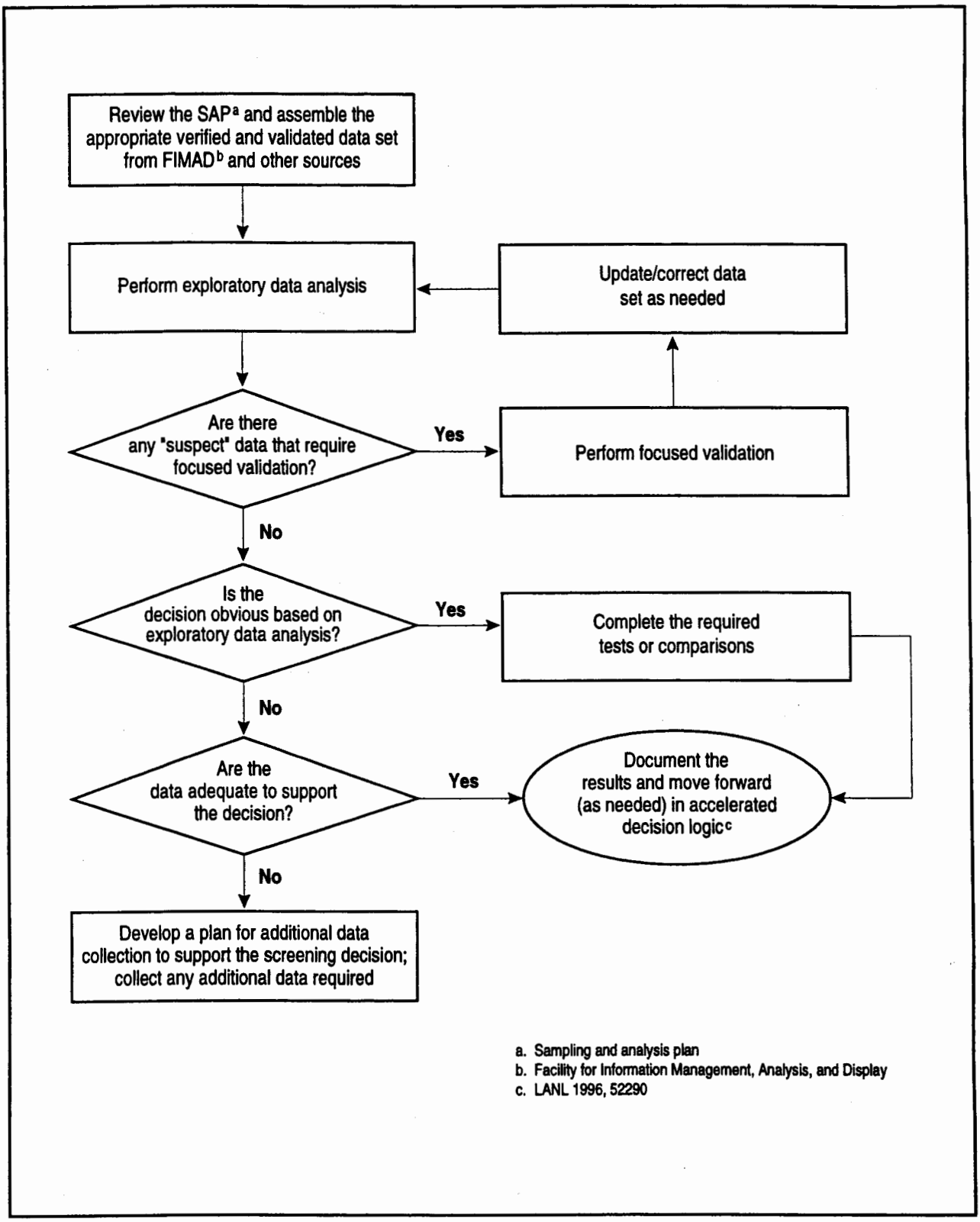
For NRAS, the SAP must provide the following:

- the problem-specific verification and baseline validation criteria (the analytical data generator must be made aware of these criteria). Note that if the nonroutine service closely resembles



a. Analysis requirements are specified in a formal statement of work or contract.
b. Data quality assessment
c. Sampling and analysis plan
d. Facility for Information Management, Analysis, and Display

Figure D-2. Data flow diagram for the Environmental Restoration Project.



F D-4 / ER QAPP / 022096

Figure D-3. The data quality assessment process.

Following exploratory data analyses and any required focused validation, the DQA process will determine the validity of

- removing questionable results from the data set,
- correcting incorrect data, or
- leaving the data set unaltered.

Any changes made to the data set must be fully documented.

The remainder of the DQA process is intended to reconcile the data with the requirements specified in the SAP, and to assess the adequacy of the data to support the SAP objectives. The DQA process addresses the questions "Did we get what we asked for?" and "Did we ask for what we need?" How this is done depends in part on how quantitatively the original requirements were formulated.

To assess the adequacy of the sampling design to support a decision (e.g. "Did we ask for what we need?"), the data analyst must work with other members of the DQA team to determine if the number and types of samples, as specified in the SAP and as actually collected, were appropriate. This includes

- determining if the number and location of samples required by the SAP were taken;
- determining if the appropriate media were sampled;
- judging the adequacy of the sample number and locations, given the updated understanding of the problem; and
- determining if the understanding of the problem changed since the SAP was prepared because of observations made by the field team.

While problems on one or more of the above do not automatically rule out using the data as planned, they can suggest that supplemental data must be collected before proceeding.

In some cases, the correct decision will be obvious by inspection of the data set; for example, when reported values are far above or are uniformly below SALs. Provided that the sampling design was adequate to support this obvious decision, the evaluation of data adequacy for that decision may terminate after the initial exploratory analysis and the site moves forward in the accelerated decision logic (LANL, 1996, 52290).

If the decision is not obvious, either because the data do not all point in the same direction, or because of some minor problem with the design, or if the SAP specifies that the decision will be based on the results of certain statistical tests or calculations (e.g., on upper confidence bounds for certain population parameters), further examination of the analytical data is required. Qualitative evaluation of the analytical and field data will determine if

- analytical measurements for all variables specified in the SAP were generated;
- the appropriate suite(s) of analytes were requested, given the updated understanding of the problem;

Results of DQA will be documented in adequate detail for the decision-maker and peer reviewers to evaluate the effect of these results on decision-making. If a decision can be made based on the data, the documentation will include both the decision outcome and also the level of confidence that can be ascribed to the decision. The data analyst and other members of the DQA team will develop recommendations in cases where the data are not deemed sufficient to support a decision, which may be included in the documentation or presented to the decision-makers in a less formal manner. If further investigations appear to be required, the data analyst will summarize information contained in the existing data as it applies to the design of subsequent SAPs for this site. As appropriate, the DQA team may recommend that limitations be placed on current or future uses of the data.

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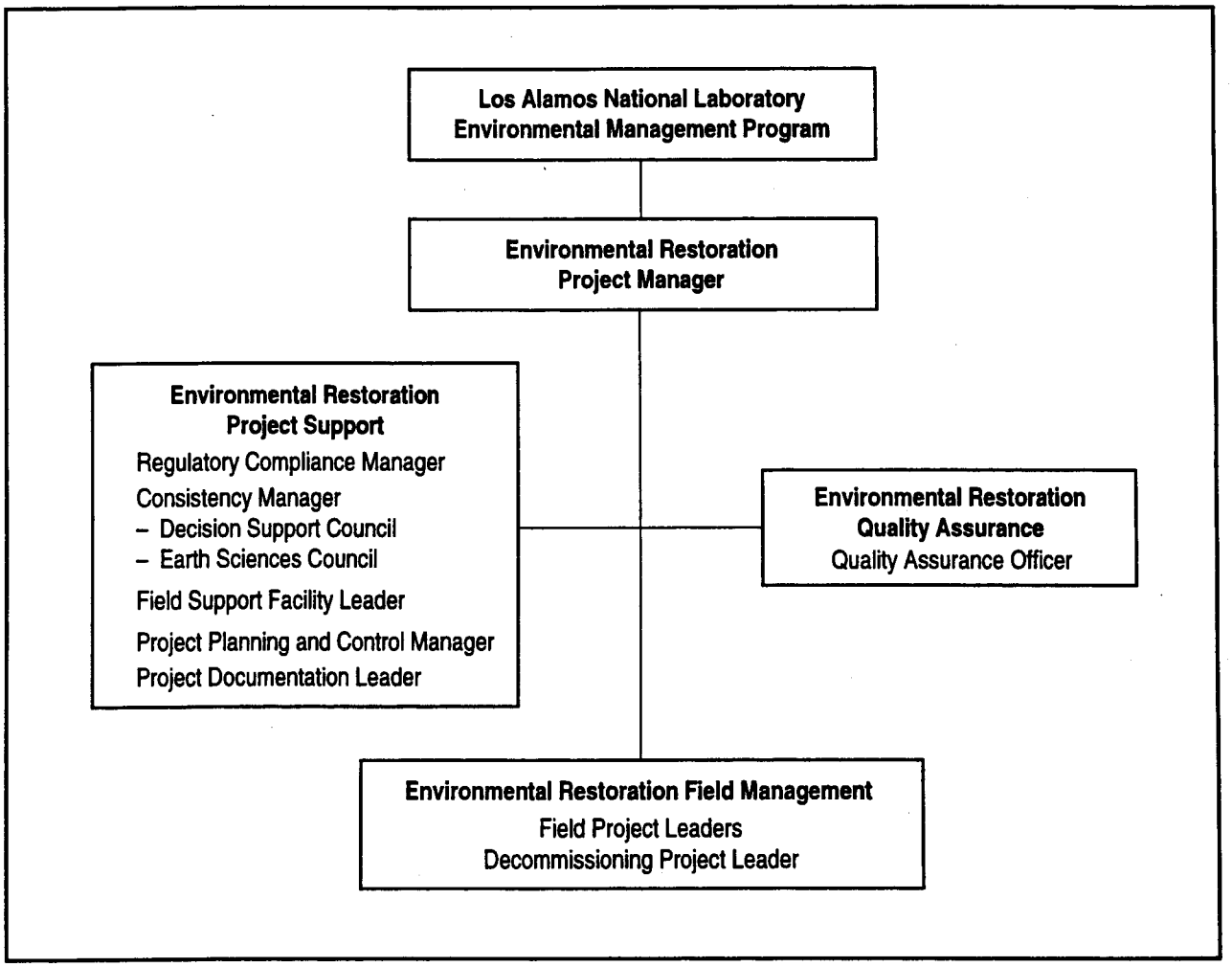
ACRONYMS AND ABBREVIATIONS

AAS	atomic absorption spectroscopy
ADS	activity data sheets
AEC	Army Environmental Center
ASTM	American Society for Testing and Materials
CLP	Contract Laboratory Program
CMS/CMI	corrective measures study/corrective measures implementation
COC	chemical of concern
COPC	chemical of potential concern
CRDL	contract-required detection limit
DOE	US Department of Energy
DPL	Decommissioning Project Leader
DQA	data quality assessment
DQO	data quality objective
DSC	Decision Support Council
EC	expedited cleanup
EDL	estimated detection limit
EPA	US Environmental Protection Agency
EQL	estimated quantitation limit
ER	Environmental Restoration
FIMAD	Facility for Information Management, Analysis, and Display
FPL	Field Project Leader
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
HAZWOPER	hazardous waste operator and emergency response
HE	high explosive
HSWA	Hazardous and Solid Waste Amendments
ICP(ICPAES)	inductively coupled plasma
IWP	Installation Work Plan
LANL	Los Alamos National Laboratory
NFA	no further action
NRAS	nonroutine analytical services
OM	on-site measurements
PE	performance evaluation
PPC	Project Planning and Control
PRS	potential release site
QA	quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QMP	Quality Management Plan
RAS	routine analytical services
RCRA	Resource Conservation and Recovery Act
RFI	RCRA facility investigation
RPF	Records-Processing Facility
SAL	screening action level
SAP	sampling and analysis plan
SCA	site characterization analyses
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TCLP	Toxicity characteristic leaching procedure
TIC	tentatively identified compound
USATHAMA	US Army Toxic and Hazardous Materials Agency
UTL	upper tolerance level
VCA	voluntary corrective action
VOC	volatile organic contaminant/compound
VOST	volatile organic sampling train

1.0 OVERVIEW

The ER Project is part of the LANL Environmental Management Program. The ER Project is led by a project management team consisting of the ER Project Manager, a Regulatory Compliance Manager, a Consistency Manager, five FPLs, a DPL, a Field Support Facility Leader, a Project Planning and Control (PPC) Manager, Project Documentation Leader, and a QAO (Figure I-1). Project-wide responsibilities, lines of communication, and the organizational structure are divided into functional areas as described below.



F A-1 / ER QAPP / 022096

Figure I-1. Project organizational structure for the Environmental Restoration Project.

- identifying training opportunities and requirements for ER Project and contractor personnel; and
- implementing this document and other applicable documents, policies, orders, etc., as set forth in the LANL ER QMP.

Each of these project leaders have teams (contractor and LANL employees) that assist them in developing appropriate work plans, implementing the work plans, reviewing data, making decisions, and reporting on the results of their activities.

1.3 ER Project Support

The ER Project Compliance Manager, Consistency Manager, Field Support Facility Leader, PPC Manager, Project Documentation Leader, and their teams make up the ER Project support functional area. These managers and leaders report directly to the ER Project Manager. They are responsible for providing technical and administrative support to the ER Project leaders and each other, and for implementing the LANL ER QMP and this document.

1.3.1 Regulatory Compliance Manager

This manager is the primary interface with the State of New Mexico, the EPA and regulatory agencies, as appropriate. The Compliance Manager

- provides technical support to the FPLs and the DPL on regulatory issues;
- is responsible for initiating permit modifications based on results of ER Project activities;
- provides technical support to the Field Support Facility, FPLs, and the DPL concerning waste management issues;
- is responsible for managing site closure activities for the ER Project; and
- is responsible for implementing this document and other applicable documents, policies, orders, etc., as set forth in the LANL ER QMP.

1.3.2 Consistency Manager

The Consistency Manager

- has primary responsibility for promoting consistency throughout ER Project activities;
- is responsible for the development and revisions of ER Project procedures, policies, etc.;
- is assisted in the above task by the DSC, ESC, and the resources available to the ER Project through LANL, DOE, and contractor personnel; and

1.3.5 ER Project Documentation Leader

The ER Project Documentation Leader (PDL) supports the ER Project by managing the RPF and functions. This task includes

- processing hardcopy records of ER Project activities to the LANL central records facility,
- making records available to the public as part of the ER Project administrative record,
- distributing documents (including controlled documents), and
- preparing documents and retrieving documents in support of ER Project activities.

1.4 ER Project Quality Assurance Officer

The ER Project QAO reports to the ER Project Manager and provides support to the ER Project management team. The QAO is responsible for

- identifying ER Project QA requirements;
- advising ER Project management on QA matters;
- developing, reviewing, and approving the ER Project QMP and other applicable quality assurance/control/assessment documents; and
- assessing the effectiveness of the ER Project's implementation of applicable governing documents and regulations such as the HSWA Permit, the LANL ER QMP, and the LANL ER QAPP, by
  - performing assessment and oversight of ER Project activities,
  - implementing the analytical laboratory oversight functions (including laboratory qualification and performance monitoring),
  - reporting quality problems to the appropriate level manager and requesting the implementation of corrective actions, and
  - tracking corrective actions to completion.

1.0 INTRODUCTION

The objective of environmental sampling is to obtain samples of material that represent a particular population about which information is needed. The population could be a geographical area, the collection of waste material in one or more containers, a stream of fluid, etc. Decisions concerning the possibility of taking NFA or of having to remediate a site will be based on the data derived from analysis of the collected samples. If the samples do not reflect the true contaminant distribution of the site, environmental problems could go unaddressed or a great deal of effort might be expended in unnecessary site remediation.

Contaminants of potential concern (COPCs) must be identified, and sampling locations and depths must be identified, prior to selecting the devices or methods for collecting the samples. The sample collection methods and tools are then selected to satisfy the investigation's quality objectives.

2.0 FACTORS AFFECTING SAMPLING TOOL SELECTION AND SAMPLE HANDLING

Most of the environmental samples collected at LANL are soils. However, liquids are sampled occasionally, and when it is necessary to establish the presence or distribution of permeable layers, or to establish stratigraphic control, continuous coring may be necessary. Those items that must be identified before sampling methodology can be selected are addressed below.

1. *The Intended Use of the Data, i.e., Objectives of the SAP.* These might include

    - providing input such as contaminant location, variability, and site contaminant concentration profiles for future SAP design;
    - determining whether contaminants are present above predetermined action levels such as SALs;
    - providing information for selecting remediation alternatives;
    - determining the volume and location of media that must be removed or treated to achieve cleanup levels; and
    - verifying attainment of cleanup levels.

    Involving the correct personnel to develop SAPs is essential to success of the investigation. This selection will usually mean that at least one statistician will be involved at the outset of planning. Where soils are to be collected and analyzed, subject matter experts representing the disciplines of soil science, geology, geochemistry, hydrology, risk assessment, and analytical chemistry should also be involved, as necessary. The responsible FPL should be involved; the public and the regulators should be included as necessary.

    When evaluating the problem to be resolved and identifying associated contaminants, not only should the primary LANL process at the site be considered but also those processes that are related. For example, HE casting and milling operations would leave a potential legacy of HE contaminants. However, the milling machines must have been lubricated periodically, and spilled lubricants might have

polychlorinated biphenyl (PCBs), TPH, metals, and radionuclides are generally not subject to loss through volatilization, and therefore are not subject to the gentle handling constraints associated with the volatiles.

Enough sample must be collected to provide sufficient material for completing all of the required analyses. This amount can be determined easily by consulting the chosen analytical methods, reviewing past experiences, consulting the appropriate LANL ER SOP, or consulting a member of the SMO or DSC Chemistry Team. Coordination with the SMO can be especially important if a particular sample type is being collected for the first time or a particular suite of analytes is to be determined for the first time. It is usually best to collect more sample than necessary for the required analyses in the event that an analysis of the sample must be repeated.

If a sample requires special handling, it is advisable to record on the chain-of-custody form accompanying the sample, and on the analytical order, all special handling requirements. For example, if stones, vegetable matter, other debris, etc., should be excluded from the analyses, a note to that effect should accompany the sample.

7. *The Preservatives and Containers Used to Store the Samples (Dictated by the Analytes to be Determined and the Analytical Protocol).* Many analytes tend to adsorb to the inside walls of their containers. This causes an apparent loss of analyte because the adsorbed analytes may not be transferred during sample preparation with the rest of the sample. Even worse, the loss of analyte may not be apparent at all. Conversely, contaminants can leach into the sample from the containers, especially if liquid is present in the sample. Thus, the choice of container can be critical to obtaining accurate analytical results. Chemical preservatives can retard or prevent the plating of contaminants onto container walls. The choice of container closure (i.e., lid, cap, etc.) is also important, as the glues used to fasten liners into the closures can release contaminants into the sample. When standard analytical protocols are used, containers and preservatives are generally dictated by the analytical protocols. In cases where sample preservation conditions are not specified for a particular analysis, an experienced chemist should be consulted for advice. LANL-ER-SOP-01.02 also provides guidance for the preservation and containerization of samples.

8. *Sample Holding Times, Storage, and Shipping.* Because loss of analytes from sample degradation is a common problem, it is important not to store a sample for too long a period before it is analyzed. The acceptable storage period (i.e., holding time) is a function of the analytes of interest, the sample matrix, and the storage conditions. Most degradation rates are greatest soon after sample collection and decrease over time. However, biodegradation rates can increase with time as microorganisms increase in number.

   If a sacrifice in sensitivity is acceptable for volatile organic contaminants/compounds (VOCs), the methanol extraction (NMED circa 1994, ER ID number 52243) may be used to extract the analytes from the sample matrix on-site. The advantage of this on-site extraction technique is that the extract submitted for analysis is more stable than the original sample with regard to analyte loss. Water samples to be submitted for VOC analyses must be preserved with acid (e.g., sodium bisulfate or hydrochloric acid) upon collection.

   Light-sensitive analytes must be stored in dark-colored containers or in the dark to prevent photodegradation. Volatile organic compounds are easily lost through

1. *Soil and Sediment Sampling.* Table II-1 presents those sampling tools most useful for collecting environmental soil and sediment samples. For ephemeral streams these methods are suitable for sampling stream beds in the absence of water; when the stream bed is under water, refer to Table II-2 or II-3.

   Augers are especially suited to collecting composite samples because the augering action homogenizes the soil, whereas they are not useful for collecting samples for volatile analyses because the augering action causes loss of volatiles. Augers are not recommended for collecting cohesionless soil samples as the sample may not be retained when the auger is removed from the ground. The open tube sampler is recommended for collecting samples that are to be characterized lithologically. Scoops are useful only for collecting surface grab samples, which may then be composited either at the sampling site or in a laboratory. They are not recommended for collecting volatiles because the act of scooping and pouring the samples into a container can cause loss of volatiles. Thin-walled tube samplers can be used with or without sample liners to collect core samples. When used with a stainless steel or brass liner, the liner can be easily sealed with end caps after sample collection and submitted for volatiles analyses. If a clear plastic liner is used, lithologic descriptions of the core can be obtained. The thin walled tube sampler, because it is pushed into the ground hydraulically rather than being tamped or hammered into the ground, does not compact the soil. It is thus well suited to the determination of geotechnical parameters such as porosity, hydraulic conductivity, grain size distributions, and Atterberg limits. For soil sampling at depths greater than 5 ft., mechanical drivers such as auger drill rigs are typically used to push the sampling tool into the ground.

   Table II-2 presents sampling tools useful for sampling drainage sediment from flowing rivers, streams, and surface water drainage. If the flowing water source is dry at the time of sampling, the sampler should refer to Table I-1 for soil sampling methods.

   Sediment sampling may be used to determine if contaminants are migrating downstream of the potential contaminant source. Samples should be taken from those areas such as ponds and low-lying ponding areas in which contaminants can accumulate during periods of flow. If background samples are needed, they should generally be taken from upstream of the potential contaminant source.

   Use of dippers and scoops should be confined to shallow waters of low flow rates. The dipper may be more effective than the scoop at retrieving fine grained sediments, but due to the lack of a good cutting edge its use is generally limited to soft sediment.

   The methods most appropriate for sampling sediments in standing water include the scoop, dipper, and box and dredge samplers. All of these except the dredge sampler are most useful in shallow water. The box and dredge samplers used with a wire line can be used in deep water. Table II-3 is a tabulation of likely applications of these sampling tools.

2. *Water Sampling.* Table II-4 presents sampling methods useful for collecting samples from streams, rivers, and drainage flows. The bottle submersion approach is the simplest, requiring that a bottle attached to an extendible arm be submerged below the water surface until it is full. The subsurface filling of the bottle prevents the loss of volatiles. If samples are to be composited after collection, volatiles analyses should not be performed on the composited sample because of the great

TABLE II-3

SOIL SAMPLING TOOLS USEFUL FOR SAMPLING IN STANDING WATER

	Analyses				
Sampling Tool	Volatiles	Non-volatiles[1]	Sample Type[2]	Approximate Sampling Depth	Applicable ER SOP
Dredge Sampler (Ponar Grab)	Yes[3]	All except geotechnical	Grab; AC; Lithology	0 to 0.5 ft.	06.14
Gravity Corer	Yes	Yes	Grab; AC; VC; Lithology	0 to 3 ft.	06.14
Hand Corer	Yes	Yes	Grab; AC; VC; Lithology	0 to 3 ft.	06.14
Scoop/Trowel	Yes[3]	Yes[3]	Grab; AC	0 to 0.5 ft.	06.14

1 Includes geotechnical parameters, herbicides, metals, PCBs, pesticides, radionuclides, semivolatile organics, and total petroleum hydrocarbons.
2 Grab = grab sample; AC = areal composite sample; VC = vertical composite sample; Lithology = lithology description
3 Can be used to collect samples for these analyses, but is not recommended for this use.

potential for loss of analytes during compositing. All but the dipper method are useful for collecting composite samples. The dipper method should only be used for collecting samples that will not have volatile organic analyses performed on them.

Sampling of standing surface water (see Table II-5) should be conducted based on the SAP requirements which should include a consideration of suspected contaminant concentrations and natures of the COPCs. For example, if dense organics that are immiscible with water are to be sampled, those contaminants are most likely to be found at the bottom of the body of water. However, water-immiscible COPCs that are less dense than water are most likely to be found at the surface.

TABLE II-4

TOOLS USEFUL FOR SAMPLING FLOWING SURFACE WATER

	Analyses				
Sampling Tool	Volatiles	Non-volatiles[1]	Sample Type[2]	Approximate Sampling Depth	Applicable ER SOP
Bottle Submersion	Yes	Yes	Grab, AC, I	0 to 0.5 ft.	06.13
Dipper	No	Yes	Grab, AC, I	0 to 0.5 ft.	06.13
Extendible Bottle Sampler	Yes[3]	Yes	Grab, AC, I, VC	0.5 to 5.0 ft.	06.13
Extendible Tube Sampler	Yes[3]	Yes	Grab, AC; I; VC	0.5 to 5.0 ft.	06.13
Single Stage Sampler	No	Yes	Grab	Surface	06.29
Peristaltic Pump	Yes	Yes	Grab, AC; I; V	0.5 to 5.0 ft.	06.13

1 Includes herbicides, metals, PCBs, pesticides, radionuclides, semivolatile organics, and total petroleum hydrocarbons.
2 Grab = grab sample; AC = areal composite sample; I = integrated sample; VC = vertical composite sample
3 Can be used to collect samples for these analyses, but is not recommended for this use.

TABLE II-6

TOOLS USEFUL FOR SAMPLING GROUNDWATER AND SOIL WATER

	Analyses				
Sampling Tool	**Volatiles**	**Non-volatiles[1]**	**Sample Type[2]**	**Approximate Sampling Depth**	**Applicable ER SOP**
Bailer	Yes[3]	Yes	Grab, I[4]	0 to 30 ft.[5]	06.03
Bladder Pump	Yes	Yes	Grab, I, VC[4]	0 to >30 ft.	06.03
Vacuum Lysimeter[6]	Yes	Yes	Grab, I	0 to 6 ft.	06.05
Pressure-Vacuum[6] Lysimeter	Yes	Yes	Grab, I	0 to 50 ft.	06.05
High Pressure-Vacuum Lysimeter[6]	Yes	Yes	Grab, I	0 to >50 ft.	06.05
Piston Pump	Yes[3]	Yes	Grab, I, VC[4]	0 to >30 ft.	06.03
Submersible Pump	Yes[3]	Yes	Grab, I, VC[4]	0 to >30 ft.	06.03
Syringe Sampler	Yes	Yes	Grab	0 to >30 ft.	06.03

1 Includes herbicides, metals, PCBs, pesticides, radionuclides, semivolatile organics, and total petroleum hydrocarbons.
2 Grab = grab sample; AC = areal composite sample; I = integrated sample; VC = vertical composite sample
3 Can be used to collect samples for these analyses, but is not recommended for this use.
4 Acceptable, but not the preferred application.
5 Can be used at depths greater than 30 ft., though the preferred depth is less than 30 ft.
6 Not recommended for clay soils.

The hand auger is recommended for sampling soils from drums when toxicity characteristic leaching procedures (TCLP) or radionuclide testing will be performed. Compositing is also recommended except when collecting samples for volatile organic analytes. The auger is especially effective for collecting composite samples because the augering action tends to homogenize the samples.

4. *Air Sampling.* Air canisters such as SUMMA canisters can be used to collect relatively large volumes of gases for subsequent analyses. Air and exhaust gases can be trapped on a sorbent and later released from the sorbent material for analysis. The volatile organic sampling train (VOST) can be used to collect volatile organic contaminants with boiling points less than 100 °C. See Table II-8.

1.0 INTRODUCTION

Tabulated in this appendix are the analyses and related information provided through the LANL analytical laboratory contracts Statement of Work (LANL 1995, 49738). Section 2 lists, for RAS, estimated detection limits (EDLs) for inorganic analytes, and estimated quantitation limits (EQLs) for organic, HE and radiochemical analytes. Section 3 lists data for NRAS.

EDLs are based on the Contract Laboratory Program "Contract Required Detection Limits (CRDLs)," which are not necessarily achievable in real world samples. EQLs listed for soil/sediment are based on wet sample weight but, normally, data are reported on a dry weight basis, thus causing EQLs higher than those cited for dry weight.

In parts of this appendix, references are made to analytical methods that are approved for quantifying specific analytes or analyte suites, by citing the associated method numbers. Method numbers in this appendix that are preceded by "SW" indicate methods belonging to the SW-846 analytical methods compendium (EPA 1986, 31732).

2.0 ROUTINE ANALYTICAL SERVICES

2.1 Inorganics (Metals and Inorganic Compounds)

2.1.1 Sample Preparation

Following are the sample preparation procedures that are appropriate for use in determining metals. Methods from the most recent version of SW-846 should be used, although CLP sample preparation procedures (from Statement of Work ILM03.0 or more recent) may be used, if appropriate for the matrix.

- SW-3005 Acid digestion of waters for total recoverable or dissolved metals for analysis by flame AAS or ICP;
- SW-3010 Acid digestion of aqueous samples and extracts for total metals for analysis by flame AAS or ICP;
- SW-3020 Acid digestion of aqueous samples and extracts for total metals for analysis by furnace AAS, with the exception of As and Se, which are to be prepared according to methods 7060 and 7740;
- SW-3040 Dissolution procedure for oils, greases or waxes (microwave digestion of these samples is preferred);
- SW-3050 Acid digestion of sediments, sludges, and soils.
- SW-1311 TCLP (note that changes made in the Federal Register, Volume 57, No. 227, p. 55114, must be incorporated); and
- SW-3015 and SW-3051 Microwave digestion procedures.

TCLP metals (identified as footnote 3 in Table III-1) may be requested as a separate determination. Laboratories should consider EDLs for TCLP metals using the TCLP to be the regulatory limits. Method SW-1311 (7/92) is the method to be used for TCLP.

Cyanide may be determined using methods SW9010, SW9010A, SW9012, or EPA 335.2. The EDLs for cyanide are 10 µg/L (water) and 0.05 mg/kg (soils). The soil EDL for CLP ILM03.0 method 335.2 is based upon a 5-gram sample taken to a final volume of 250 mL.

The contractor may vary weights and final volumes for metals and cyanide analyses; however, any allowable variance must still meet the EDL.

2.2 Volatiles

Table III-2 identifies the volatile target analytes and associated EQLs. The US EPA methods that are options for use are method SW8260 (11/90 or more recent) or the CLP method for volatiles (OLM02.0 or more recent, using capillary column). These methods are based on purge and trap sample extraction/concentration followed by gas chromatography/mass spectrometry analysis.

Tentatively identified compounds (TICs) may be requested. If requested, they should be identified and quantitated per the CLP method for volatiles, OLM02.0 (or more recent).

TABLE III-2

VOLATILE TARGET ANALYTES AND EQLS

Target Analyte	Water, mg/L	Soil/Solids, mg/kg
Chloromethane	10	10
Vinyl Chloride	10	10
Bromomethane	10	10
Chloroethane	10	10
Acetone	20	20
Dichlorodifluoromethane	10	10
Iodomethane	5	5
Trichlorotrifluoroethane	5	5
Trichlorofluoromethane	5	5
Methylene Chloride	5	5
1,1-Dichloroethene	5	5
Carbon Disulfide	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene (total)	10	10
Bromochloromethane	5	5
Chloroform	5	5
1,2-Dichloroethane	5	5
1,1-Dichloropropene	5	5
2-Butanone	20	20
2,2-Dichloropropane	5	5

2.3 Semivolatiles

Table III-3 identifies the semivolatile target analytes and associated EQLs. The US EPA methods that are options for use are method SW-8270 (11/90 or more recent) or the CLP method for semivolatiles (OLM02.0 or more recent). These methods are based on solvent extraction, concentration, and GC/MS detection and quantitation.

TICs may be requested. If requested, they should be identified and quantitated per the CLP method for semivolatiles, OLM02.0 (or more recent).

2.4 Pesticides and Aroclors

Table III-4a identifies the pesticide and aroclor target analytes and associated EQLs. The US EPA methods that are options for use are methods SW-8081, *dual column option*, (11/92 or more recent) or the CLP method for pesticides/aroclors (OLM01.8 or more recent). These methods are based on solvent extraction, concentration, and GC/EC detection and quantitation.

Since the EQLs are sensitive to the nature of the sample matrix, Table III-4b presents factors by which the EQLs in Table III-4a are to be multiplied, depending on the matrix.

2.5 High Explosives

Table III-5a presents the HE target analytes for method SW8330 and associated EQLs. For water samples these analytes may be determined using either of the following methods:

- SW8330, or
- US Army Toxic & Hazardous Materials Agency (USATHAMA), 1990, "Improved Salting-Out Solvent Extraction Method for Determination of Low Levels of Nitroaromatics and Nitramines in Groundwater" coupled with the USATHAMA 6/30/88 "Determination of Explosives in Water by High Pressure Liquid Chromatography" (method no. UW14).

For soil samples, the analytes in Table III-5a may be determined using either of the following methods:

- SW8330, or
- USATHAMA, August 1989, "Reversed-Phase Method for the Determination of Explosive Residues in Soil."

Table III-5b lists additional HE analytes that may be determined using USATHAMA analytical methods.

TABLE III-3 (Continued)

SEMIVOLATILE TARGET ANALYTES AND ASSOCIATED EQLS

Target Analyte	Water, mg/L	Soil/Solid, mg/kg[1]
Fluoranthene	10	330
Fluorene	10	330
Hexachlorobenzene	10	330
Hexachlorobutadiene	10	330
Hexachlorocyclopentadiene	10	330
Hexachloroethane	10	330
Indeno(1,2,3-cd)pyrene	10	330
Isophorone	10	330
2-Methylnaphthalene	10	330
2-Methylphenol	10	330
4-Methylphenol	10	330
Naphthalene	10	330
2-Nitroaniline	50	1600
3-Nitroaniline	50	1600
4-Nitroaniline	20	660
Nitrobenzene	10	330
2-Nitrophenol	10	330
4-Nitrophenol	50	1600
N-Nitrosodimethylamine	10	330
N-Nitrosodiphenylamine	10	330
N-Nitroso-di-n-propylamine	10	330
2,2'-oxybis(1-Chloropropane)	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Phenol	10	330
Pyrene	10	330
1,2,4-Trichlorobenzene	10	330
2,4,5-Trichlorophenol	50	1600
2,4,6-Trichlorophenol	10	330

1 EQLs for soil are based on no Gel Permeation Chromatography (GPC) clean-up being performed. The laboratories' GPC equipment will determine what the EQL is, based on the volume of extract the GPC equipment uses. However, if possible, the laboratories should concentrate the GPC extract to a volume that makes the EQL for a sample that underwent GPC clean-up no more than twice the listed EQL.

TABLE III-5a

NITROAROMATIC AND NITRAMINE HE TARGET ANALYTES AND EQLS

		EQL[1]		
		Waters, µg/L		Soils, mg/Kg
Target Analyte	Abbreviation	Low Level	High Level	
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	(HMX)	ND	13.0	2.2
Hexahydro-1,3,5-trinitro-1,3,5-triazine	(RDX)	0.84	14.0	1.0
1,3,5-Trinitrobenzene	(1,3,5-TNB)	0.26	7.3	0.25
1,3-Dinitrobenzene	(1,3-DNB)	0.11	4.0	0.25
Methyl-2,4,6-trinitrophenylnitramine	(Tetryl)	ND	20.0	0.65
Nitrobenzene	(NB)	ND	6.4	0.26
2,4,6-Trinitrotoluene	(2,4,6-TNT)	0.11	6.9	0.25
4-Amino-2,6-dinitrotoluene	(4-Am-DNT)	0.06	ND	ND
2-Amino -4,6-dinitrotoluene	(2-Am-DNT)	0.035	ND	0.26
2,4-Dinitrotoluene	(2,4-DNT)	0.02	5.7	0.25
2,6-Dinitrotoluene	(2,6-DNT)	0.31	9.4	0.26
2-Nitrotoluene	(2-NT)	ND	12.0	0.25
3-Nitrotoluene	(3-NT)	ND	7.9	0.25
4-Nitrotoluene	(4-NT)	ND	8.5	0.25

1 These EQLs apply only to the SW-846 and USATHAMA methods listed above. If "ND" appears in a cell, the EQL has not been determined. In those cases, assume an EQL of 1.0 mg/L for the low level waters and an EQL of 10 mg/L for the high waters where values are missing.

TABLE III-5b

ADDITIONAL HE TARGET ANALYTES AND EQLS

Analyte	Water, µg/L[1]	Soil, mg/Kg[1]	Analytical Method
Nitroglycerine	25	0.50	USATHAMA Aug., 1989, Reversed Phase HPLC Method for the Determination of NG and PETN in Water (or Soil, as applicable)
Pentaerythritol Tetranitrate	25	0.50	USATHAMA Aug., 1989, Reversed Phase HPLC Method for the Determination of NG and PETN in Water (or Soil, as applicable)
Nitroguanidine	5.0	0.51	USATHAMA Aug., 1989, Reversed Phase HPLC Method for the Determination of Nitroguanidine in Water (or Soil, as applicable)
Tetrazene	6.11	1.3	USATHAMA Reversed Phase HPLC Method for the Determination of Tetrazene in Water (or Soil, as applicable)
Nitrocellulose	70.0	ND	USATHAMA Reversed Phase HPLC Method for the Determination of Tetrazene in Water

1 These are assumed EQLs based on the USATHAMA lower limit of the linear concentration range.

TABLE III-6b

GAMMA SPECTROSCOPY SUITE DEFINITION

Nuclide symbol	Nuclide name	Nuclide symbol	Nuclide Name
Ac-228	actinium-228	Pa-231	protactinium-231
Am-241	americium-241	Pa-233	protactinium-233
Ann Rad	annihilation radiation	Pa-234m	protactinium-234m
Ba-140	barium-140	Pb-210	lead-210
Bi-211	bismuth-211	Pb-211	lead-211
Bi-212	bismuth-212	Pb-212	lead-212
Bi-214	bismuth-214	Pb-214	lead-214
Cd-109	cadmium-109	Ra-223	radium-223
Ce-139	cerium-139	Ra-224	radium-224
Ce-144	cerium-144	Ra-226	radium-226
Co-57	cobalt-57	Ru-106	ruthenium-106
Co-60	cobalt-60	Rn-219	radon-219
Cs-134	cesium-134	Se-75	selenium-75
Cs-137	cesium-137	Sn-113	tin-113
Eu-152	europium-152	Sr-85	strontium-85
Hg-203	mercury-203	Th-227	thorium-227
I-129	iodine-129	Th-234	thorium-234
K-40	potassium-40	Tl-208	thallium-208
La-140	lanthanum-140	U-235	uranium-235
Mn-54	manganese-54	Y-88	yttrium-88
Na-22	sodium-22	Zn-65	zinc-65
Np-237	neptunium-237		

3.0 NONROUTINE ANALYTICAL SERVICES

Table III-7 presents individual analytes and analyte suites that comprise the NRAS included by the laboratory contracts SOW (LANL 1995, 49738). In some cases, references are made to the analytical methods that are approved or recommended for quantifying the listed parameter. Where analyte suites are listed but the analytes within the suite are not defined, the field unit is responsible for identifying which analytes are to be quantified, and for selecting the analytical method appropriate for quantifying the selected analytes at the desired concentration levels in the applicable sample matrices.

TABLE III-7

NONROUTINE ANALYTES, ANALYTE SUITES AND RELATED INFORMATION (Continued)

Chlorophenoxy Herbicides
Organophosphorus Pesticides
Dioxins/Furans
Metals
TCLP (extractions and associated analyses)
Zero Headspace Extraction
Tumbler Extraction
Volatiles
Semivolatiles
Herbicides
Pesticides
Metals
Reactive CN/Sulfide
Density
Flash Point
Ignitability
Free Liquids
RADIOCHEMISTRY
Uranium by KPA
Carbon-14 (C-14)
Technetium-99 (Tc-99)
Air Analyses
Method TO-1
Method TO-2
Method TO-5 PAHs
Method TO-13 PAHs
Method TO-14
Volatile Organic Sampling Train (VOST)
Fixed Gases
Benzene, Ethylbenzene, Toluene, Xylene (BTEX)
Method EPA 504.1
Halogenated Volatiles, Method SW8010
Aromatic Volatiles, Method SW8020
Organophosphorus Pesticides, Method SW8140
Volatile Organics by GC/MS, Method SW8240
Total Petroleum Hydrocarbon, TPH-G
GFAA Metals Al, Se, Pb, Tl, Cd, Sb, Ag
UNUSUAL MATRICES
Tissue/Vegetation:
Organochlorine Pesticides and PCBs, Method SW8080
Metals by ICPAES, Method SW6010 (Each Metal for Kemron)
Mercury, Method SW7470
Non-soil solids:
Volatiles
Semivolatiles

TABLE III-7

NONROUTINE ANALYTES, ANALYTE SUITES AND RELATED INFORMATION (Continued)

Oil and Grease-Gravimetric
Petroleum Hydrocarbons
pH
Phenolics, Total
Phosphate-Ortho
Phosphate
Phosphate-Total
Phosphorus
SOC (soluble organic carbon)
Solids - Percent Ash
Solids-Percent Moisture
Solids-Percent Solids
Solids-Settleable
Solids-Total
Solids-Total Dissolved (TDS)
Solids-Total Suspended (TSS)
Solids-Total Volatile (TVS)
Specific Conductivity
Specific Gravity
Sulfate
Sulfide
Sulfite
Surfactants, Methylene Blue Active Substances (MBAS)
Temperature
TOX (total organic halides)
Turbidity
GEOTECHNICAL
1-Dimensional Consolidation
Atterburg Limits
Bulk Density
Cation Exchange
CU Triazial (3pt.) (Shelby Tube) -Triazial Shear
Dimensional Swell
Grain Size - Hydrometer
Grain Size - Sieve Analysis
Grain Size, Method ASTM 0422
Hydraulic Conductivity
Hydraulic Extrusion/visual classification
Modified Proctor (4 inch diameter. mold)
Modified Proctor (6 inch diameter mold)
Moisture Ash & Organic Matter
Moisture Content
Particle Size (%passing N 200 sieve)
Particle Size (combined)
Paste pH (rock)

1.0 INTRODUCTION

Selection of analytical methods is complicated by the diversity of methods available and the tendency of individuals making method selections to use those with which they are familiar. Method selection may be further complicated by the belief that certain standard analytical protocols such as SW-846 are applicable beyond their intended purposes. The scientist sometimes feels forced to use standard methods because they are widely accepted as being robust, accurate, precise, etc., even though a more accurate, more precise, more robust, cheaper, or less time consuming method is available or can be readily developed. It is also frequently easier and more cost-effective for commercial laboratories to standardize their operations by selecting a few robust methods that are applicable to most routine samples.

Overshadowing all other considerations, the use of certain analytical methods might be governed by Federal, state, or other regulations, or ER Project representatives may enter into agreements with regulators to use specific analytical methods. For example, the RCRA mandates the use of solid waste methods, SW-846, in the following circumstances:

- determination of hazardous waste characteristics (SW-846 method 1311) followed by appropriate analytical method,
- determination of free liquid (SW-846 method 9095),
- analyses associated with submission of delisting petitions,
- analyses associated with a hazardous waste incinerator trial burn, or
- determination of air emissions from process equipment.

While the first two determinations listed above may occasionally be relevant for the LANL ER Project, the others are not likely to apply at all. It is imperative to notify the SMO when one of these five circumstances dictates strict use of an SW-846 method so that the laboratory can be informed.

2.0 CONSIDERATIONS IN SELECTING ANALYTICAL METHODS AND QC

Analytical method performance criteria derive from the site-specific planning requirements. Communicating with regulators in the early stages of planning is good practice and is an integral part of a thorough planning process, as it espouses full participation of *all* stakeholders. Since it is not always possible to have regulators actually present during scoping meetings, it is important to gain acceptance from them of the approach taken to identify the important performance criteria. The approach used could lead to selecting other-than-traditional (e.g., SW-846/CLP) methods. Negotiation of method selection with regulators is possible and is encouraged. The DSC Chemistry Team will be helpful in these negotiations.

Large-volume RAS contracts have been developed for the ER Project, including the laboratory-required QC procedures and criteria. In addition, these contracts include the ready capacity to allow for many NRAS. The list of RAS and allowed methods, with detection or quantitation limits identified for each analyte, is presented in the analytical laboratory Statement of Work (SOW for RAS [LANL 1995, 49738]) and Appendix III of this document. The services under NRAS are listed in that same appendix. Note that

Precision. Method selection should take into account the allowable precision error associated with the analytical measurement as determined through the DQO planning process. Ideally, the precision error is known prior to analytical method selection. However, method selection can be made without knowing the precision error and precision error may be determined later. In those cases, the project may be at risk of having selected inadequate methods.

Confidence in estimating certain statistical parameters, such as the arithmetic mean, can be increased by averaging many measurements. This may be especially useful when using on-site measurements (OM) or abbreviated routine measurements that cost less and require less time to implement than fixed laboratory methods. A good example is the use of OM, optimized, or focused methods for a known contaminant problem.

Stability and Robustness. Instrument stability is a function of precision and drift, which influences the measurement system stability. Stable instruments require relatively fewer recalibrations whereas unstable methods may require frequent recalibrations and may require averaging the results of several repeat analyses on the same sample to increase the confidence in the results. It is important to understand the stability of a measurement system on both short (hourly or daily) and long (weekly or monthly) time scales. Frequently, the stability of a system is not stated explicitly in a particular analytical protocol but can be inferred from the required calibration frequency. If a measurement system is robust, it will be stable and will yield results that are comparable, even when used by different operators, on different instruments, in different laboratories, and on samples of varying matrix compositions.

Bias. The impact of bias on data quality should be evaluated in the SAP planning process. In some cases, use of the data without a consideration of the bias may be acceptable if the bias consideration does not change the decision (e.g., high bias on sample results less than the action limit or low bias on sample results greater than the action limit). This decision needs to be made during the evaluation of the data during DQA. When bias needs to be addressed, the data reviewers must be consulted to determine the direction and magnitude of the suspected bias as well as the significance of the bias. Without sufficient information to assure that the bias is real and significant, corrections for bias cannot be justified. Selection of methods with no bias (or a well characterized bias) relative to the methods used previously, generally facilitates planning, but it is not necessary as long as the degree of bias and its significance can be determined before making the required decision.

Sample Preparation. Prior to chemical analysis, a sample is usually treated chemically or physically to yield a derivative of the sample. It is the derivative of the sample (extract, digestate, electroplate, pulverized sample, etc.) that is actually analyzed. The sample preparation method must be compatable with the sample matrix and is usually specified as part of the analytical procedure, either explicitly or as a reference to another procedure. The sample preparation procedure may be followed with a sample cleanup procedure designed to remove the majority of the interferences, either without affecting the analytes of interest or by affecting them in a quantifiable manner. If interferences are known to exist at a particular site, methods should be selected that allow for mitigation of the interferences and this information should be conveyed to the analytical laboratory. Advice on method selection for mitigating interferences can be obtained from members of the DSC Chemistry Team. When special interferences and methods for mitigating them are known in advance, the analytical laboratory should be alerted to the situation. The conveyance of special instructions to analytical laboratories should be coordinated through the SMO.

When regulatory holding times are exceeded, and the context for the sampling is not regulatory, there may be more room for flexibility in dealing with the potential problem of changes in the sample. However, for water samples this should never be the case as *all water samples for volatiles analyses must be acidified* (preferably as described below).

If adherence to holding time requirements is critical, it may be appropriate to analyze the samples in the field or to take direct field measurements at the sampling point. An example would be the determination of VOCs using GC or GC/MS analytical methods based on fixed laboratory methods. Holding times can sometimes be extended by limited sample preparation in the field. For example, water samples to be analyzed for VOCs can be, and *should be*, acidified to prevent microbial degradation (250 mg of sodium bisulfate per 40 ml sample has proved to be very effective). Methanol extraction of soils in the field (NMED 1994, 52243) is another way to extend holding times for volatile organics, although this will raise the detection limits. Immediate freezing of samples (using dry ice in the field) is a possibility for HE, and is recommended for soil samples to be analyzed for HE components.

Turnaround Time. The selected sample preparation and analysis methods must allow for the sample results to be generated in a timely manner. The required turnaround time could play a major role in the method selection and is often a primary factor in selecting field analyses over fixed laboratory analyses. Some of the reasons for requiring measurements on-site are

- a decision must be made in, for example, less than 24 hours to continue work efficiently, such as during a remediation;
- to direct work in real time, such as during an Expedited Site Characterization (a DOE-HQ initiative) and when there is a potential for a change in direction from the original design, and new knowledge gained from on-site measurements will allow for a speedy change;
- when there are constraints such as sample degradation (addressed under "holding times") or temporal constraints that require instant analysis; and
- for gaining important health and safety information to protect the workers and the public.[4]

When time is a factor, it is important to assure that data quality needs can be met, even if abbreviated methods are used, because of the time constraints. An example of when it is important to make sure that "fast" analyses will not compromise data use is when counting times for radioisotopes will be inadequate to meet sensitivity needs if the time is constrained.

Cost. While cost is not a technical issue, it will factor into the selection of analytical methods. As budgets are reduced, the ability to generate sufficient data with adequate quality becomes more difficult. If the EPA- and DOE- mandated DQO process is used,

3 Standard Practice for Estimation of Holding Time for Water Samples Containing Organic Constituents," ASTMD4515-85.

4 "Planning Guidance for Using Onsite Measurements," Fisk, J, Bath, R, and Klevano, C, draft, December 1995 - for DOE-EM26.

3.1 Selection of SW-846, CLP, and AEC (formerly USATHAMA) Methods

When using CLP or Army Environmental Center (AEC) methods (CLP and SW-846 are comparable in performance, the differences being transparent to the data user), the appropriate SW-846 method should be identified, then the CLP or USATHAMA method equivalent to the selected SW-846 method substituted. The first step is to identify those methods that appear to serve the intended purpose (Table IV-1 can be helpful here). Table IV-1 shows the relationship among the analytical methods allowed by the analytical support subcontracts for routine services. In most cases, an SW-846 method is allowed and use of SW-846 methods will support most needs. However, especially with regard to radionuclides (techniques allowed are cited) and selected high explosives, laboratory-specific methods are allowed or specific USATHAMA methods are required. The user must then verify the applicability of the method by referring to the particular protocol and

- verify that the method is applicable to supporting the decision to be made based on results generated from the method either alone or in conjunction with other data;
- verify that the sensitivity, comparability to other analytical methods, detection limits, selectivity, stability bias, and precision of the protocol meet the needs of the SAP;
- verify that the protocol is not subject to interferences that are anticipated to be present in the sample at concentrations that will render the analyses invalid; and
- balance factors such as turnaround times, holding times, and analytical costs.

Selected AEC methods are available for high explosives analyses for which no equivalent CLP or SW-864 methods exist. Refer to Table IV-1 when selecting these high explosives routine analytical serves. Because radiochemistry methods are not as standardized as other chemistry methods, the allowed techniques are cited, but the method numbers (nonexistent) are not.

4.0 NONROUTINE ANALYTICAL SERVICES

A large number of NRAS can be provided through the contract mentioned above. Appendix III provides the list of NRAS available through the RAS contracts. Many of the NRAS services involve use of standard or commonly accepted methods (though not routinely provided through these contracts). In addition, many of the NRAS methods are simple modifications of the RAS methods, in which case the deliverables are alike, the QC procedures can be cited, and criteria can be modified to meet needs (e.g., a lower detection limit may need to be demonstrated). However, for specialized or emerging methods some prescriptive narrative in the SAP is needed to make sure that project goals are met. The following information is required as a set of deliverables from the contractors when NRAS is requested;

- target analytes/measurement parameters and associated analytical results and quantitation or measurement limits,
- citation of sample preparation and analysis method used (when a "standard" method is used) or a description of the technology used when a standard method cannot be cited,

TABLE IV-2

TARGET ANALYTE EQL[1] BY MATRIX; pCi/g OR pCi/L UNLESS INDICATED

Analyte	Soil	Water	Technique[2]
Gross alpha/beta	10.0	3.0	gas-proportional
Gross alpha/beta	10.0	NA	liquid scintillation
Strontium-90[3]	2.0	5.0	gas-proportional
Americium-241	0.1	0.1	alpha spectroscopy
Plutonium-238, -239	0.1	0.1	alpha spectroscopy
Thorium-228, -230, -232	0.1	0.1	alpha spectroscopy
Thorium-230, -232	0.1	0.1	ICP-MS-FIA (commonly requested nonroutine analysis)
Uranium-234, -235, -238	0.1	0.1	alpha spectroscopy
Uranium-234, -235, -238	0.1	0.1	ICP-MS-FIA (commonly requested nonroutine analysis)
Tritium	300 pCi/L	300	liquid scintillation
multiple isotopes (Table III.F.4)	Am-241: 1 Cs-137: 1	Am-241: 20 Cs-137: 20	gamma spectroscopy
Gross gamma	2.0	100	NaI(Tl) or HPGE detection
Total uranium	0.5 mg/g	1 mg/L	KPA[4] (commonly requested nonroutine analysis)
Total uranium	0.5 mg/g	1 mg/L	ICP-MS (commonly requested nonroutine analysis)
Radium-226	1.0	1.0	assorted
Radium-228	0.5	0.5	assorted
Thorium-234	1.0	20	assorted
Lead-210	2.0	5.0	assorted

1 EQL
2 The Los Alamos National Laboratory methods for these analytes are contained in LA-10300-M, "Health and Environmental Chemistry: Analytical Techniques, Data Management, and Quality Assurance."
3 It may be presumed that strontium-89 is not present.
4 Kinetic phosphorescence analysis, also referred to as pulsed-laser phosphorimetry (ASTM D 5174-91) or kinetic laser phosphorescence.

- calibration data,
- raw analytical data (instrument outputs),
- manual calculations used for generating results (unless specified in cited method), and
- all QC documentation.

4.1 Selection of NRAS Methods

Whether choosing the NRAS method from the list available through the analytical contracts or citing alternate (including new, emerging, and innovative) technologies, there are performance criteria to be considered, just as in selecting "standard" methods.

In some cases, a particular performance criterion or additional information item may not be readily quantifiable. For example, selectivity is inherently difficult to quantify, even for standard methods such as the CLP and SW-846 methods. In such cases, the applicability of the method should still be demonstrated through the analysis of reference materials or spiked samples, etc., before it is used for analysis of environmental samples under the SAP. If the method is used prior to this verification, the field unit is at risk and will be required to demonstrate applicability at a later date. At times the best professional judgment of a competent analyst, such as a DSC Chemistry Team member, may be involved in the assessment of the items above. This is especially true in the case of difficult-to-measure parameters such as data comparability. In all cases, the evaluation of the nonroutine analytical method, including the bases for conclusion regarding the method's applicability to the intended data use, should be documented. This documentation may be included in the SAP either directly or by reference.

When making the decision to use a method that is new, emerging, innovative, or not demonstrated for the site-specific matrix, it is critical that method be tested first as to its applicability. This testing is best done by using site-specific performance evaluation (PE) materials that have been well characterized for the analytes of interest. During the ongoing period of sampling and analyses, performance information should be gathered, documented, and evaluated so that another potential user of the method may benefit from the precedent.

5.0 ON-SITE MEASUREMENTS

This special case of using on-site analytical methods needs special attention. Complete guidance for planning for the use of OM is provided in (DOE 1995, 52240). There are many benefits to be derived from using OM, such as

- abbreviated methods that are focused for the analytes of interest, and that may provide better data and be less costly for those parameters than the standard survey-type analyses;
- faster analyses—this is especially important when a decision must be made on-site and data must be collected in "real-time";
- the ability to make changes in the SAP based on new knowledge that may be gained from the OM data; and
- the opportunity for decreasing total error by maximizing the number of samples using the abbreviated methods.

When making a decision to use OM there are several critical elements that must be considered and criteria that should be met to justify the use. There are times that the same benefits can be derived from using fixed laboratories (either close-support such as the LANL laboratories or contractor laboratories) to perform the abbreviated, or even "screening" methods with a rapid turnaround time, if time is a factor. Unless there is a large number of analyses to be performed, it is not often cost-effective to set up in the field (other than for hand-held or "back of the pick-up" methodology). The critical elements are outlined in Chapter 2 of the cited draft OM guidance (DOE 1995 52240).

GLOSSARY

Abbreviated method A shortened form of a method. Usually refers to analytical methods that have been modified to require less rigorous sample preparation, analysis conditions or quality control.

Aliquot A portion of a sample or sample derivative taken for analysis.

Analysis A process used to measure one or more attributes of a sample in a clearly defined, controlled, systematic manner. Often requires treating a sample chemically or physically before the measurement step to render the sample or a derivative thereof (e.g., a digestate or extract) ready for measuring the selected attribute.

Analyte The particular chemical or radiochemical species to be identified and/or quantified in a sample of interest.

Assessment The evaluation process used to measure the performance or effectiveness of a system and its elements. In this document, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, management system review, peer review, inspection, and surveillance.

Audit (quality) A systematic and independent examination to determine whether quality activities and related results comply with planned arrangements, whether these arrangements are implemented effectively, and whether they are suitable to achieve objectives.

Baseline data validation Data validation directed toward determining whether the data in question satisfy clearly defined quality control checks. This validation is used to assign a consistent set of qualifiers to data that draw attention to potential data deficiencies.

Bias (1) The degree to which the value obtained for a measured parameter deviates from the value accepted as the true, or reference, value. (2) A systematic deviation from the true value that remains constant over replicated measurements within the statistical precision of the measurement process. Synonymous with *deterministic error, fixed error,* and *systematic error.* Sometimes referred to as *accuracy*, though the mathematical equation for computing accuracy differs from that for bias. Typically expressed as a percentage deviation, bias is computed as follows:

$$Bias = \frac{1}{T}(\bar{x} - T) \times 100\%,$$

where

$\bar{x}$ is the average of several determinations and T is the true value.

The true value may be the value established for a spiked sample or a certified standard reference material such as a performance evaluation sample.

Blank sample A sample expected to have negligible or unmeasurable amounts of the analytes of interest. Results of blank sample analyses indicate whether or not field samples might have been contaminated during one or more steps of the sample collection, transport, storage, preparation and analysis process.

"Completeness" means all required information is present—both hard copy and electronic. "Correctness" means the reported results are based on properly documented and correctly applied algorithms. "Consistency" means that values are the same when they are reported in different reports or are transcribed from one report to another. "Compliance" means that the data pass numerical QC tests based on parameters or limits specified in a contract or in an auxiliary document. The primary purposes of verification are to determine appropriate payment to those providing services and to point out areas of noncompliance with QC specifications that may affect data use and that can be made a focus of further data validation or data quality assessment activities.

Double blind sample A sample whose analyte concentration and sample identity are unknown to the analyst. Double blind samples are usually submitted to an analytical laboratory without the laboratory's knowledge so that the ER Project can evaluate the laboratory's performance.

Duplicate analysis An analysis (includes sample preparation and analysis) performed on one of a pair of identically prepared subsamples of the same sample. Not to be confused with a duplicate measurement.

Duplicate measurement One of a pair of measurements performed on a prepared sample (e.g., digestate or extract) under identical conditions.

Environmental sample See *field sample*.

Error The inevitable uncertainty associated with scientific measurements or decisions. Measurement error comprises three types of errors: (1) systematic error (or bias), which is always of the same algebraic sign, (2) random error which varies in algebraic sign and is unpredictable, and (3) blunders which are unpredictable human errors such as transcription errors. Decision error comprises (1) false positive error which is quantified as the probability of rejecting a null hypothesis when the hypothesis is actually true and (2) false negative error which is quantified as the probability of not rejecting a null hypothesis when the hypothesis is false.

Estimated quantitation limit The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine analytical laboratory operating conditions. The estimated quantitation limit is generally 5 to 10 times the method detection limit. However, a nominal value may be chosen for the estimated quantitation limit within these guidelines to simplify data reporting. For many analytes, the estimated quantitation limit is selected as the lowest non-zero standard in the calibration curve. Sample estimated quantitation limits are highly matrix-dependent, and the specified estimated quantitation limits might not always be achievable.

Equipment blank (equipment rinsate blank) A blank sample that is used to rinse the sample collection equipment and is then transferred to a sampling container. The equipment blank is collected after equipment decontamination is completed but prior to collection of another field sample.

Error The difference between an observed or computed value and the value accepted as the true value.

Matrix spike duplicate An intralaboratory split sample spiked with a known amount of target analyte (s). Spiking occurs prior to sample preparation and analysis.

May Denotes permission but not a requirement.

Method A body of procedures and techniques for systematically performing an activity.

Method blank An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing and which is prepared and analyzed in the same manner as samples.

Method detection limit (MDL) The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of samples of a given matrix type containing the analyte after subjecting the sample to the usual preparation and analyses.

Mixed waste Hazardous waste material as defined by 40 CFR part 261 (RCRA), mixed with radioactive contaminants.

Must Denotes a requirement that has to be met.

Nonroutine analysis Those analytical requests not defined as routine analyses. The LANL ER statement of work for analytical services provides more details concerning the nature of nonroutine analyses.

Out of control A condition for which the quality of outputs of a process are suspect based on a statistical interpretation of QC sample data.

Performance criteria Measurable criteria used to assess all or part of a process.

Performance evaluation A type of audit in which the quantitative data generated in a measurement system are obtained independently and compared with routinely obtained data to evaluate the proficiency of an analyst or laboratory.

Population (statistical) The total aggregate of observations that conceptually might occur as the result of performing a particular operation in a particular way. For example, the soil comprising a PRS or PRS aggregate.

Population unit The smallest subunit of the population that is of interest for a particular study.

Population variability The degree to which a particular characteristic of the population varies.

Precision A concept used to describe dispersion of measurements with respect to a measure of location or central tendency. Precision may be represented by the standard deviation of a set of measurements. The standard deviation is computed as follows (assuming each measured value, x_i, is statistically independent of the others and the measured values are normally distributed about an average value:

Quality Management Plan (QMP) A formal document that describes the quality system in terms of the organizational structure, functional responsibilities of management and staff, lines of authority, and required interfaces for those planning, implementing, and assessing all activities.

Quality system A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Radioactive tracer A radioactive material added to, or induced in, a sample for the purpose of monitoring chemical or physical losses of the target analytes. The tracer is assumed to behave in the same manner as that of the target analytes.

Radioactive waste Waste material containing radionuclides, or contaminated by radionuclides.

Random Being or relating to a member of a set (1) whose members have equal probability of occurring or (2) from which each member has equal probability of being selected. Frequently applied to selection of sampling points. Should not be confused with haphazard.

Relative precision (See also *Precision*) The precision measured relative to a particular value. Relative precision expressed as the relative standard deviation (RSD) may be calculated as follows:

$$RSD = \frac{s_x}{\bar{x}} \times 100\%,$$

where

s_x is the standard deviation, and

$\bar{x}$ is the arithmetic mean of all the measurements used to compute the standard deviation.

Remediation The process of reducing the concentration of a contaminant)or contaminants) in air, water, or soil media to a level that poses an acceptable risk to human health.

Replicate measurement A re-analysis (remeasurement) of a prepared sample.

Replicate sample One of multiple samples taken from and expected to be representative of the same population and carried through all steps of the sampling and analysis procedures in an identical manner. One type of replicate sample is a duplicate sample.

Representativeness A measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Rinsate Blank See *Equipment blank.*

closely as possible those matrices representative of environmental samples from a particular location. They may be naturally occurring materials or manufactured materials that have been characterized exhaustively, at least with respect to selected analytes and with respect to interferences associated with quantifying those analytes by selected analysis methods.

Split sample A sample that has been subdivided into two or more portions expected to be of the same composition. Used to characterize within-sample heterogeneity, sample handling, and measurement variability.

Standard operating procedure (SOP) A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks.

Surrogate compound An organic compound used in the analyses of organic analytes that is similar to the target analytes in chemical composition and behavior in the analytical process but is not normally found in the field samples.

Third Party Implementable Enough information is provided at a level of detail that enables any qualified party to execute the plan as intended.

Total measurement error The sum of all errors that occur from sampling through reporting of results; the difference between the reported result and the true value of the population that was to have been sampled.

Total Quality Management (TQM) The process of applying quality management to all activities of the organization, including technical and administrative operations. See *Quality Management* and *Quality System*.

Trip blank A sample of analyte-free media taken to the sampling site and returned to the analytical laboratory unopened along with samples taken in the field. It is stored with the samples until the samples have been analyzed. Used to monitor cross contamination of samples during handling and storage both in the field and in the analytical laboratory.

Variance (statistical) The square of the standard deviation (See *Precision*). A concept used to describe the dispersion of measurements with respect to a measure of location or central tendency.