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Revision 0.1
27 April 1993

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT

U.S. DEPARTMENT OF ENERGY
FERNALD OFFICE

SITEWIDE
CERCLA QUALITY ASSURANCE PROJECT PLAN

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Prepared by
Fernald Environmental Restoration Management Corporation
27 April 1993

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TERMINOLOGY (cont.)

Hazardous Substance. Any material that poses a threat to human health and/or the environment as defined in 40 CFR 300.5. Typical hazardous substances are toxic, corrosive, ignitable, explosive, or chemically reactive. Any substance designated by EPA to be reported if a designated quantity of the substance is spilled in the waters of the United States or if otherwise emitted to the environment.

Hazardous Waste. Any waste or combination of wastes that pose a substantial present or potential hazard to human health or living organisms because such wastes are nondegradable or persistent in nature, or they can be biologically magnified, or they can be lethal, or because they may otherwise cause or tend to cause detrimental cumulative effects. Also a waste or combination of wastes of a solid, liquid, contained gaseous, or semisolid that may cause or contribute to an increase in mortality or an increase in serious irreversible or incapacitating reversible illness, taking into account the toxicity of such waste, its persistence and degradability in nature, its potential for accumulation or concentration in tissue, and other factors that may otherwise cause or contribute to adverse acute or chronic effects on the health of persons or other organisms. [ed. Hazardous wastes as defined here as those wastes listed by EPA or meeting characteristics specified by EPA in their criteria pursuant to the Resource Conservation Recovery Act (RCRA). Disposal treatment or storage of hazardous wastes can only take place in a site or facility issued a permit by EPA or a state.]

Holding Time. For validation purposes, the time from sample collection to laboratory analysis.

Hydraulic Conductivity. A coefficient of proportionality describing the rate at which water can move through a permeable medium. The density and kinematic viscosity of the water must be considered in determining hydraulic conductivity.

Instrument Detection Limit. This is three times the standard deviation obtained from the analysis of a standard solution (in reagent water) at a concentration of three to five times the IDL on three non-consecutive days with seven consecutive measurements per day. The IDL is extimated sequentially until subsequent estimates do not change.

Laboratory Control Sample. A sample equivalent to internal or external control samples that may be prepared by the same laboratory performing the analyses or by a reference laboratory or agency.

Laboratory Project Manager. Individual employed by a laboratory who is responsible for overseeing the analysis and reporting of all samples from FEMP for a particular program or project. Also responsible for day-to-day liaison with the FEMP project contact.

TERMINOLOGY (cont.)

Leachate. Liquid that has percolated through solid waste and dissolved soluble components. Any liquid including any suspended components in the liquid that has percolated through or drained from waste materials.

Losing Stream. A stream or section of stream that is influent with respect to ground water (i.e., there is a net loss of stream water to the ground-water system). The hydraulic head of the stream surface has a greater potential than the surrounding ground-water environment, so the stream water contributes recharge to the aquifer.

Lower Limit of Detection. Minimum count rate that can be routinely detected (radionuclide analyses).

Matrix Spike. Introduction of a known concentration of a spiking substance into a sample to provide information about the effect of the sample matrix on the digestion and measurement method and on the accuracy of the result.

Method Blank. A blank prepared with the same reagents and put through the same processing as the samples.

Minimum Detectable Activity. Smallest quantity of a radionuclide that can be detected in a sample with a 95 percent confidence level.

Monitoring Well. A well installed in a selected location and screened at a specific depth to allow monitoring of chemical and hydraulic parameters of the ground water and aquifer.

Open-Channel Flow. Flow with a free surface within definable, continuous-channel boundaries. Flow in a stream, river, or unconfined flow in a conduit.

Overland Flow. Water flowing on the land surface without the ordinary constraint of definable, continuous channel boundaries. Most commonly refers to the flow resulting when rainfall rates exceed surface infiltration rates. This is also called rainfall-excess overland flow. May also include flood flows, also termed channel-excess flows. One characteristic of overland flow is that it is ephemeral.

Partially Penetrating Well. A well constructed in such a way that it draws water directly from a fractional part of the total thickness of the aquifer. The fractional part may be located at the top or the bottom of the aquifer or anywhere in between.

TERMINOLOGY (cont.)

Piezometer. A bored, drilled, or driven shaft or a dug hole with a depth greater than the largest surface width; a shaft or pit dug or bored into the earth, generally cylindrical, and often walled with bricks or tubing to prevent earth from caving in with its main purpose being to monitor ground water elevation or pressure; or a nonpumping well used to measure the elevation of the water table or potentiometric surface.

Ponding. Standing water on soils in closed depressions. The water can be removed only through percolation or evapotranspiration.

Practical Quantitation Limit. The lowest quantity of analyte in a sample that can be reliably detected within specified limits of precision and accuracy, during routine laboratory operating conditions. For radiochemical measurements, this is interpreted to mean the minimum quanity of analyte in a sample which results in a total propagated uncertainty (at the 95% confidence level) which is 20% of the measured value.

Precision. A measure of the repeatability of an analysis or measurement. Measurements that are repeatable within small limits are said to be precise.

Process Wastewater. Any water that, during manufacturing or processing, comes into direct contact with or results from the production of or use of any raw material, intermediate product, finished product, byproduct, or waste product.

Program. In the context of this SCQ, a defined set of ongoing activities, such as routine monitoring, that will be continued in basically the same format for an indeterminate length of time (e.g., the CERCLA Program, Environmental Compliance Monitoring Ground-Water Program, and Environmental Monitoring Program). Programs are subject to the same substantive requirements regarding sampling and analysis as projects. Because projects may be subsets of programs, all SCQ requirements for projects also apply to programs conducting similar activities.

Project-Specific Plans. Scoping documents required for any program or project. Project-specific plans for FEMP sampling and analysis activities should include elements defined in Section 6 of the SCQ. Project-specific plans may include but are not limited to, work plans, field sampling plans, health and safety plans, and standard operating procedures.

Project. In the context of this SCQ, a defined set of activities pursued towards a defined final conclusion. Examples of projects at FEMP include the remedial investigation/ feasibility studies for each operable unit, removal site evaluations, and removal actions. A project may be included within a program.

TERMINOLOGY (cont.)

Raffinate. Aqueous solution and impurities (dissolved and suspended solids) resulting from the process of converting uranium ore and other source material to uranyl nitrate.

Reagent Blank. See Method Blank.

Recharge. A process, natural or artificial, by which water is added to the saturated zone of an aquifer.

Recharge Area. An area in which there are downward components of hydraulic head in the aquifer. Infiltration moves downward into deeper parts of an aquifer in a recharge area. **A recharge area is where water reaches the ground water by surface infiltration.**

Record of Decision. A public document that explains which cleanup alternatives will be used at a National-Priorities-List site. The ROD is based on information and technical analysis generated during the remedial investigation/feasibility study and consideration of public comments and community concerns.

Redox Potential. Potential for oxidation and reduction of elements in water. A measure of aqueous electron concentration controlled by reactions involving elements present in more than one oxidation state.

Relative Percent Difference. A measure of precision using results from duplicate analyses.

Remedial Action. Those actions consistent with permanent remedy taken instead of, or in addition to, removal action in the event of a release or threatened release of a hazardous substance into the environment to prevent or minimize the release of hazardous substances so that they do not migrate to cause substantial danger to present or future public health or welfare or the environment.

Remedial Design. A phase of remedial action that follows the remedial investigation/ feasibility study and includes development of engineering drawings and specifications for a site cleanup.

Remedial Investigation/Feasibility Study. Consists of two distinct but related studies usually performed concurrently. The remedial investigation is intended to gather data necessary to determine the types and extent of contamination at a superfund site and assess risk to human health and the environment posed by identified contamination. The feasibility study identifies and screens cleanup alternatives and produces a detailed analysis of the technology and costs of remedial alternatives.

TERMINOLOGY (cont.)

Removal Action. Short term, immediate actions taken to address releases of hazardous substances that require expedited response.

Removal Site Evaluation. A study conducted to determine whether a site poses an imminent or potential hazard to human health and the environment requiring initiation of a removal action.

Rill. See Gully.

Runoff. (1) Precipitation discharged into stream channels from an area. Surface runoff is water that flows off the surface of the land without sinking into the soil. Water that enters the soil before reaching surface streams is called ground-water runoff or seepage flow from ground water. (U.S. Soil Conservation Service) (2) Any rain water, leachate, or other liquid that drains overland from any part of a facility.

Sample Delivery Group. A group of samples, usually fewer than 20, received over a period of up to 14 calendar days. Data from all samples in an SDG are contained in one data package. SDG is synonymous with data package in that the results from the samples in the SDG are (usually) reported in the one package.

Sampling Activity. Total of a number of steps required to be completed to collect a single sample.

Sampling Event. Collection of a sample from a single location for a specific project.

Sampling Round. Collection of samples from one or more locations for a specific project during a specified time period for a similar purpose.

Saturated Zone. The zone in which the voids in the rock or soil are filled with water at a pressure greater than atmospheric. The water table is the top of the saturated zone in an unconfined aquifer.

Seep. An area where water oozes from the earth. A surface expression of the water table. A small spring with little or no discernable flow.

Significant Condition Adverse to Quality. A condition, if left uncorrected, could significantly impact the quality of a measurement or program.

TERMINOLOGY (cont.)

Site. "...shall include all areas within the property boundary of FMPC [now FEMP] and any other areas that received or potentially received released hazardous substances, pollutants, contaminants, or hazardous constituents. The term shall have the same meaning as 'facility' as defined by Section 101(9) of CERCLA, 42 U.S.C. §9601(9)." (Consent Agreement, April 9, 1990)

Slag. Waste solids derived from the molten processing of uranium metal.

Spring. Where water flows without artificial aid from the subsurface to the surface. A surface expression of the water table.

Standard. (noun) In context of equipment calibration, something set up and established by authority as a rule for the measurement of a parameter (e.g. concentration, length, temperature, mass). (adj) A regularly and widely used method (e.g. standard operating procedure), material (e.g. standard gauge), or calculation (e.g. standard deviation).

Stream. Any body of flowing water or other fluid.

Subcontractor. Organization that performs a service for FEMP while contracted to a prime contractor of the Department of Energy and that reports to the prime contractor.

Surface Water. Water that is open to the atmosphere and subject to surface runoff

Surveillance. Spot checks of program implementation to determine conformance to specified requirements. Equivalent to EPA performance audit.

Teflon. A fluorocarbon plastic manufactured by the DuPont Corporation. In this document, teflon refers to any fluorocarbon plastic.

Tracer. A small quantity of a (usually) pure radionuclide, different than those of interest, but expected to behave similarly (i.e., is added to a sample to determine the effect on processing and derive a correction factor if necessary).

Tremie Line Method of Grouting. A method of inserting grout into a borehole to ensure that there are no void spaces. A hose or pipe is inserted into a borehole to within five feet of the bottom of the opening. Grout is pumped through the hose or pipe. As the borehole fills, the tremie line is retracted at approximately the same rate as the hole is filling.

TERMINOLOGY (cont.)

Unconfined Aquifer. An aquifer in which there are no confining beds between the zone of saturation and the surface. There will be a water table in an unconfined aquifer. Water-table aquifer is a synonym.

Unsaturated Zone. The zone between the land surface and the water table. It includes the root zone, intermediate zone, and capillary fringe. The pore spaces contain water at less than atmospheric pressure, as well as air and other gases. Saturated bodies, such as perched groundwater, may exist in the unsaturated zone.

Water Table. The surface in an unconfined aquifer or confining bed at which the pore water pressure is atmospheric. It can be measured by installing shallow wells extending a few feet into the zone of saturation and then measuring the water level in those wells.

Well. (1) A hydraulic structure which, when properly designed and constructed, permits the economic withdrawal of water from a water-bearing formation. (2) A bored, drilled or driven shaft, or a dug hole with a depth greater than the largest surface dimension (hole is deeper than it is wide). (3) Any shaft or pit dug or bored into the earth, generally of a cylindrical form and often walled with bricks or tubing to prevent the earth from caving in.

The SCQ provides for document changes in response to evolving program needs as new projects are implemented at FEMP. The SCQ is intended to be a dynamic document, that meets current site needs while retaining the flexibility to respond to advances in analytical methods, field techniques, operating procedures, and changes in the FEMP mission.

Techniques and procedures are appropriately referenced and, as improvements are proposed and accepted, change requests will be drafted and distributed for comment or approval. References to EPA guidance documents, journal articles, textbooks, and FEMP contractor methods and guidelines are an integral part of this document. Referenced documents are available to users and reviewers as public documents or upon request to the DOE Fernald Office. Referenced DOE orders are available in the FEMP library.

1.3 USE OF THE SCQ

The FEMP SCQ is not a standard quality assurance project plan. It differs from the typical CERCLA RI/FS quality assurance project plan because of the complex and diverse nature of the activities and waste sources at the site. The SCQ is a cross between a quality assurance program plan and a quality assurance project plan. The SCQ provides overall site-wide quality assurance planning for sampling and analysis activities planned or ongoing at FEMP. These activities include non-CERCLA environmental monitoring as noted in subsection 1.2.

The SCQ for sampling and analysis has two primary uses: (1) it is a document that establishes the requirements for environmental sampling and analysis, and (2) it is a working-level document with standardized procedures for common field activities that can be incorporated into Project Specific Plans (PSP) (subsection 1.5). Requirements for planning, implementation of plans, and assessment of activities are included so that it may be used like a QA program plan as defined by EPA (1980). The SCQ also fulfills the requirements of a QA project plan as defined by EPA (1983) except the portions that refer to specific samples.

Planning requirements are identified in Sections 2, 3, and 4; Appendices C, E, and F; and, to a lesser degree, Sections 5, 6, and 7. Implementation requirements are set forth in Sections 5, 6, 7, 8, 9, 10, and 13 and Appendices I, J, and K. Assessment requirements are defined in Sections 11, 12, 14, and 15; Appendices D and F; and, to a lesser degree, Section 4 and Appendix E.

Geotechnical analyses and measurements are conducted on soils, sludge, and waste for treatability studies and engineering design purposes and are bound to the requirements of the SCQ. Analyses and measurements for engineering design shall be conducted in accordance with standard methods (see section 5.3.3) at a laboratory facility that has been audited and approved by FEMP. However, engineering data that will not be used for environmental decision making, as determined through the DQO process, are excluded from other administrative requirements of the SCQ.

1.4 ASSOCIATED DOCUMENTS

1.4.1 Laboratory Analytical Methods

Appendix G of the SCQ provides standardized methods or performance criteria for analyzing samples for a wide range of parameters of interest to FEMP.

1.4.2 Project Specific Plans

Project-specific supplements to the SCQ shall be generated for each project initiated after approval of the SCQ requiring sampling and analysis. PSPs shall compliment and enhance the SCQ where appropriate and are not intended to repeat information contained in the SCQ. PSPs shall serve as comprehensive plans (Section 3) that include the following information.

- Historical information relevant to the specific project
- Assessment of existing data
- Identification of data needs and quality requirements through the DQO process described in Appendix C including reference to the appropriate DQO summary forms and specifying the intended use of the data
- Sample collection points and how they were chosen
- Methods for collecting data either by reference to the SCQ or through incorporation of specific procedures including QA/QC requirements and whether grab or composite samples will be collected
- Analytical methods to be used and corresponding analytical support levels (Section 2) including QA/QC requirements and corrective action limits

PSPs may also include the following.

- RI/FS work plan addenda for each operable unit
- Removal action work plans
- RCRA closure plans
- RCRA ground-water quality assessment plans

2.3 PROJECT OBJECTIVES

2.3.1 Specific Objectives

Specific objectives of an environmental sampling and analysis project shall be specified in Project-Specific Plans (PSP). Examples of project objectives are included in Table 2-1 (Appendix A).

2.3.2 Intended Data Usages

The intended use of acquired data is to assess the nature of the site and the degree and extent of potential problems resulting from past activities, evaluate the potential hazard to human health and the environment, evaluate remedial actions, choose and implement preferred remedial actions, and monitor plume migration and the effectiveness of remedial actions. Data partially fulfilling these requirements have been collected in previous and ongoing studies. Use of these data and identification and collection of additional data needs will fulfill the intent of the 1991 amended Consent Agreement and the stated site-remediation objectives of DOE.

2.3.3 Data Quality Objectives

Data Quality Objectives (DQO) are qualitative and quantitative statements that specify the quality of data required to support decision making. Because they are based on end use of the data to be collected, different uses require different levels of data quality. There are five FEMP-defined analytical levels that will be assigned depending on intended use of the data and the Quality Assurance/Quality Control (QA/QC) methods required to achieve the desired level of quality. These levels are analogous to the 1987 EPA-defined DQO levels 1 through 5 (U.S. Environmental Protection Agency, 1987). However, because radionuclides comprise a large proportion of the analyses supporting FEMP programs and projects and because these radionuclide analyses have been used and verified by DOE and DOE contractors for many years, it is appropriate to address these measurements as standard. Therefore, in order to maintain consistency in definition of DQO levels and to avoid confusion between EPA and DOE/EPA programs, DQO levels at FEMP will be referred to as Analytical Support Levels (ASL) A through E.

QA/QC requirements for ASLs are provided in Tables 2-2 and 2-4 (Appendix A). End data users prescribe ASLs for data to develop DQOs as specified in Appendix C. All DQOs will be approved and controlled in a separate document by the FEMP sampling and analysis management coordinator. Analytical methods for use for each ASL are identified in Appendix G. Data validation requirements are specified in Appendix D. Following are definitions of A through E levels of quality. A summary of potential uses for data at each ASL is presented in Table 2-3 (Appendix A) and described in each ASL definition.

ASL A (Qualitative Field Analysis) - Provides the most rapid (real or short time) results. ASL A is often used for preliminary comparison to Applicable or Relevant and Appropriate Requirements (ARAR), initial site characterization to locate areas for subsequent and more accurate analyses, field screening of samples to select those for fixed laboratory analysis, and engineering screening of alternatives (bench scale tests). These types of data include those generated on site through the use of photo- or flame-ionization detectors, pH and conductivity meters, alpha and beta/gamma friskers, or radiological wipe samples. Analogous to EPA DQO level 1.

Example: Field screening for alpha, beta, and gamma radiation conducted with portable field equipment provides real time qualitative analysis for the presence or absence of radioactive isotopes.

Example: Field screening for chemical gases in the well bore of ground-water monitoring wells using photo-ionization detectors provides real time qualitative analysis for presence of volatile organic compounds (e.g., benzene, toluene).

Example: Use of a radiological survey meter to qualitatively estimate the areal extent of radioactive contamination.

ASL B (Qualitative, Semi-Quantitative, and Quantitative Analyses) - Provides more quality control checks than ASL A and results may be qualitative, semi-quantitative, or quantitative. ASL B can be assigned when rapid turnaround results are needed. FEMP-specified analytical protocols in Appendix G shall be used.

Methods may range from more sophisticated screening techniques to fully defined methods similar to ASL C or D for radiological and nonradiological parameters, but with reduced QA/QC frequency and data reporting requirements for more rapid turnaround times. Also included in ASL B are standard methods (e.g., EPA 500-series drinking water methods with QA/QC requirements different than those specified for ASLs C and D) and conventional parameter analysis in support of regulatory requirements such as NPDES permit monitoring. In the event that an ASL B standard method needs to be modified for a specific analyte or group of analytes in support of a higher ASL (i.e., ASL C or D), the appropriate sample preparation method and calibration information will be prescribed and specified in the Project Specific Plan (PSP) as an ASL E. PSPs related to Consent Agreement items are subject to review by U.S. EPA and Ohio EPA; and, proposed modifications of standard methods would thus receive their review and approval.

of observations, as the value to be used to characterize the source strength. This method then results in the ability to completely describe the uncertainty associated with the source term and ultimately the risk.

ASL D data are also used to determine the UCL as discussed above. Both ASL C and D data are used to determine the UCL since the only difference between data collected at these levels is the laboratory documentation accompanying the results. The same QA/QC procedures are implemented and at any time the entire QA/QC documentation package can be requested from the laboratory. Together the level C and D data provide the final step in the quantification of the source term for use in fate and transport modeling and exposure assessments.

2.4 TARGET PARAMETERS

Appendix G identifies analytical methods and performance specifications that are currently expected to be used. PSPs will cite existing methods or performance specifications in the case of radionuclides as identified in Appendix G or specify requirements for new methods needed for ASL E data to analyze for specified target parameters. If the detection limits for a method are not adequate to meet the project needs as identified in a PSP, existing methods will have to be modified or new methods developed to meet those needs. Any method modifications or new methods used would be included in the PSP.

Specific target parameters for each project shall be identified in PSPs. Criteria used to determine target parameters for contaminant source areas and each potential migration pathway shall include a waste inventory of processes contributing to the source; previous source area sampling results; sampling results of potentially upgradient sources; past monitoring data; indicator chemical determination based on mobility, toxicity, and persistence in the environment; and requirements of specific regulatory programs. Total uranium will generally be included as a target parameter for migration pathway sampling based on results of historical sampling.

2.5 SAMPLE NETWORK DESIGN AND RATIONALE

The sample network design and rationale shall be specifically described in PSPs. The description shall include the method and justification for determining sampling locations, number of samples to be collected, frequency of sampling, sampling methods, quality assurance samples, and degree of confidence that DQOs will be met. Whether sampling locations are determined by judgmental, random, or systematic method shall be justified based on DQOs.

A background sampling plan for naturally occurring constituents in soils has been submitted to EPA and Ohio Environmental Protection Agency for review. The purpose of the plan is to determine background ranges for metals, cyanide, and radionuclides in the FEMP area (U.S. Department of Energy, 1991c).

Thirty off-site locations northwest and west of FEMP have been identified as primary background sampling sites. These locations are not likely to have been affected by contaminants migrating from FEMP because of the surface and ground water hydrology and prevailing wind

directions. The areas were historically used for agricultural purposes prior to construction of FEMP. Each location will be evaluated based on property owner interviews, proximity to potential pollutant sources, and historical data. If a location is found to be unacceptable, an alternate location will be evaluated. Samples will be collected at various depths from four borings at each location, and background levels of the parameters will be determined from their distribution in these samples.

2.6 PROJECT SCHEDULES

A schedule for completion or for conducting routine, ongoing projects shall be included in each PSP. It shall consist of the anticipated start date, duration of each project phase including field work, laboratory analysis, data validation, data assessment and interpretation, and submittal of interim and final reports. For PSPs related to Consent Agreement items thirty calendar days shall be allowed for each phase of regulatory review, and thirty days shall be allowed for comment resolution and resubmittal of documentation by FEMP.

Schedules for major deliverable items for each OU and for the site as a whole are included in Figures 2-12 through 2-17 (Appendix A). These schedules are for reference only, and the 1991 amended Consent Agreement or addenda should be consulted for official schedules.

2.7 REFERENCES

Federal Register, 43, p.47707. Federal Compliance with Applicable Pollution Control Standards. Executive Order 12088, October 13, 1978.

U.S. Department of Energy. 1988. ***Remedial Investigation and Feasibility Study Work Plan.*** Rev 3, Vol 1. Prepared by Advanced Sciences, Inc., Oak Ridge Operations, March 1988.

U.S. Department of Energy. 1990a. ***Remedial Investigation and Feasibility Study Ground Water Report.*** prepared by Advanced Sciences, Inc., Oak Ridge Operations, December 1990, draft.

U. S. Department of Energy. 1990b. ***Initial Screening of Alternatives for Operable Unit 3.*** Task 12 Report, FMPC-0312-5. prepared by Advanced Sciences, Inc., and International Technology Corp., Oak Ridge Operations, November 1990. draft.

U.S. Department of Energy. 1990c. ***Remedial Investigation Report for Operable Unit 4.*** Task 6 Report, FMPC-0406-5. Oak Ridge Operations Office, October 1990. final draft.

U.S. Department of Energy. 1990d. ***Initial Screening of Alternatives for Operable Unit 5.*** Task 12 Report, FMPC-0512-6. Oak Ridge Operations Office, December 1990. final.

U.S. Department of Energy. 1990e. ***Initial Screening of Alternatives for Operable Unit 4.*** Task 12 Report, FMPC-0512-6. Oak Ridge Operations Office, October 1990. final.

- Location, number, and description of sample collection locations including background stations shall be described
- Media to be sampled shall be identified
- Frequency of sampling shall be defined
- Quality Assurance/Quality Control (QA/QC) samples to be collected and protocols to be followed shall be specified
- The methods for collecting samples (Section 6) and whether sample is a composite or grab sample shall be specified
- Detailed method descriptions must be included if they differ from those in the SCQ or are not included in the SCQ
- Volume of samples to be collected and reference shall be specified
- Sampling schedule shall be included
- Define the organizational structure of the sampling teams as well as the roles and responsibilities of the team members
- Determine and identify equipment and materials necessary to perform required sampling activities and field analyses
- Identify appropriate field collection sampling reports pertinent to the particular sampling activity
- Forms to be used and requirements for tracking field activities will be clearly defined
- Specify sample preservation, packaging, storage, and shipping requirements in accordance with Appendix K by reference
- Specify the sample labels and chain-of-custody documentation (Section 7) to be used by reference. Provide any project specific variations in detail
- Specify decontamination procedures for sampling activities in accordance with decontamination requirements in Appendix K by specific reference. Provide any project-specific variations in detail

3.3.2.5. Analytical Methods. The description of the analytical methods used shall incorporate the target parameters, required detection limits, and the ASL. Maximum use of reference to the SCQ is encouraged and descriptions of supplemental information, site specific details, and new information shall be addressed in the PSP.

- Specify analytes of interest, reason, and performance requirements (including calibration conditions, when they are not specified within a method; refer to Section 9.6).
- Specify methods and ASL (Section 9)
- Methods must be included as ASL E if they differ from those identified in Appendix G or performance specifications in the case of radionuclides
- Types of field analyses and reasons
- Type and kind of laboratory analyses (Section 9)
- Additional quality control checks
- Define data validation requirements for ASLs B and E data
- Data validation and data reporting requirements must be specified if they differ from the SCQ
- Specify calibration requirements for field equipment, which shall be in accordance with the National Institute of Standards and Technology or the American Society for Testing and Materials if available. Otherwise specify manufacturers instructions and calibration procedures or provide specific variations in the PSP in accordance with Section 8
- Specify appropriate documentation of calibration performance
- Field measurements including replicate measurements

3.3.2.6. Project Requirements for Surveillance and Audits. Project specific surveillance and audit requirements shall be addressed in the PSP.

3.4 ANALYTICAL LABORATORY RESPONSIBILITIES

Analytical laboratories providing services for FEMP are responsible for compliance with the requirements of their specific contract, Appendix E, and Appendix G. Laboratory performance will be evaluated on an ongoing basis through use of audits (Section 12) and performance evaluation samples.

- Field spike control samples are used to determine precision and accuracy of analytical laboratory performance. They are prepared in a laboratory environment and transported to the sampling site for numbering and shipment to the laboratory with the remaining field samples. If required, field spike control samples are included once every sixty days or at least once per project, more frequently if appropriate, or when accuracy of a particular laboratory is in question. Intended use of field spike control sample analytical data shall be stated in the PSP, and quantitative requirements for accuracy by chosen analytical method shall be justified. Field spike control samples may be specified for ASLs B through E.

- Materials blanks are samples of material used in construction, decontamination, or other activity (e.g., drilling fluids, annular sealants, cleaning solutions) that are retained for quality control purposes in case unexpected contaminants are detected in related media. A material blank shall be collected in a controlled environment from each solution or mixture of materials (e.g., cleaning solutions and drilling fluids) that have the potential to introduce contamination not otherwise present in the media being sampled. These samples shall be clearly marked as retained samples and placed in an archive for future analysis if an anomalous contamination is identified upon review of sample analysis. Material blanks may be analyzed at any ASL.

4.1.2 Type and Frequency of Analytical Quality Control Samples

The following types of QC samples shall be analyzed as applicable for analytical methods identified in Appendix G. Types of QC samples required for specific analytical methods are based on ASLs. They are discussed in Section 9 and Appendix G. Internal QC checks are specified in Section 10. Analytical QC samples appropriate for ASL E and user-defined ASL B analyses shall be described in PSPs.

Frequency of QC sample collection and analysis may be increased but shall not be less stringent than that specified in Tables 2-2 and 2-4 (Appendix A) or Appendix G unless so specified in a PSP.

- Laboratory Control Samples (LCS), such as reference standards, may be certified reference material or a control matrix spike with analytes representative of target analytes. LCS results shall be compared to established control limits for accuracy and bias to determine useability of data.

- A method blank (e.g., reagent blanks, preparation blank) is a volume of the analyzed matrix to which reagents used in sample processing are added in the same volumes or proportions required by the method. Method blanks are submitted to the full analytical procedure and used to assess background contamination levels in the laboratory. Guidelines shall be established for acceptance or rejection of analytical data based on the level of contamination in the blank.

- A matrix spike is an aliquot of a sample spiked with a known amount of target analytes for the purpose of monitoring laboratory accuracy. Matrix spikes shall be analyzed when commercially available, certifiable standards exist appropriate to the method used if quantity of sample permits. (Examples of methods not requiring matrix spikes include pH and flash point.) For determination of trace metals by atomic absorption and inductively coupled plasma methods, post-digestion (analytical spikes) shall be analyzed for every sample injection to assess matrix interference.

- Matrix duplicate/replicate or matrix spike duplicates are used to assess the matrix effect on method precision. A matrix duplicate/replicate is an intra-laboratory split and spiked sample used in organic analyses.

- Surrogate spikes are used to assess matrix interferences in individual organic samples. A surrogate is an organic compound not normally found in the environment that is similar to target analytes in chemical composition and behavior relative to the method. A surrogate is added to each analytical and QC sample (organics only) prior to analysis. Surrogate spikes can also be used for radionuclide samples.

- Blind and double blind QC samples are used for long term assessment of accuracy and precision of the analysis or operator. Blind samples are submitted so the analyst knows it is a QC sample but does not know the analyte concentration. Double blind samples are submitted so the analyst is not aware it is a QC sample and does not know the analyte concentration. Types of blind and double blind QC samples include LCSs, spikes, and duplicates/replicates. Some types of these QC samples are included in requirements for certain methods at frequencies specified in Appendix K or the PSP. If additional types or frequencies of these QC samples are required they will be specified in the PSP.

- Intercomparison study samples are supplied by an external source to a series of laboratories. Results are evaluated against the expected value and against results from other participating laboratories. If available, a FEMP laboratory shall participate in at least one study for the analytes it is contractually permitted to analyze.

4.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSIS

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analyses is to meet QC acceptance criteria of analytical protocols. The accuracy and precision objective for each major measurement parameter for FEMP are pertinent to laboratory methods. Specific information on accuracy, precision, and sensitivity is presented in Section 14.

Standard operating procedures shall be written for laboratory analyses and shall include required accuracy, precision, and sensitivity specifications for the analyses. PSPs shall include project required precision, accuracy, representativeness, completeness and comparability guidelines. Procedures for field equipment to measure pH, conductivity, redox potential (Eh), temperature, dissolved oxygen, and alkalinity are provided in Appendix K. Accuracy and precision requirements for field screening analyses are also provided in Section 6.

4.2.1 Analytical Precision

To assess precision of an analytical method, instrument, or laboratory analysis, a routine program of duplicate or replicate analysis shall be established. Results of these analyses are used to calculate relative percent difference (defined as 100 times the absolute difference of each data set, divided by the average of the data set) for duplicate, matrix spike duplicates, or replicates. (See Section 14 for further explanation and the equation for evaluating relative percent difference). The data set relative percent difference may be used to generate precision control charts for organic and inorganic laboratories.

Range analysis may be used to evaluate the precision or reproducibility of radiological data derived from methods for which performance data are not currently available. Statistical range analysis is used to calculate the expected mean range and control limits for a replicate or duplicate result and assess whether the result is "in control." A range analysis result that lies within three standard deviations of the mean is considered in control. Range analysis results greater than three standard deviations from the mean are considered to be "out of control." Results that are out of control may be re-analyzed as required by the method, or results may be flagged or qualified for use during data validation.

4.2.2 Laboratory Accuracy

To assess accuracy of a chemical method or a chemical laboratory analysis, analytical results of method blanks, matrix spikes/matrix spike duplicates, field blanks, and container blanks shall be assessed along with a periodic program of sample spiking. The results of sample spiking are used to calculate percent recovery, which is the quality control indicator for accuracy. Percent recovery is defined as 100 times the observed spike sample result or concentration minus observed sample result or concentration divided by amount of spike added to the sample. Percent recovery of matrix spikes is used to generate accuracy control charts. Percent recovery is calculated from the equation in Section 14.

Range analysis may be used to evaluate the accuracy of radiological data. Statistical range analysis is used to calculate the expected mean range and control limits for a replicate or duplicate result and assess whether the result is "in control." A range analysis result that lies within three standard deviations of the mean is considered in control. Range analysis results greater than three standard deviations from the mean are considered to be "out of control."

Results that are out of control may be re-analyzed as required by the method, or results may be flagged or qualified for use during data validation.

4.2.3 Sensitivity of Analysis

The QA objective with respect to sensitivity is the achievement of specified method detection limits and quantitation limits. These limits depend on instrument sensitivity and matrix effects associated with the analysis. Therefore, it is important to monitor and take into account sensitivity to ensure data quality.

Analytical methods are identified in Appendix G. Instrument sensitivity is monitored by the analysis of method blanks, calibration check samples, and laboratory control samples.

4.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

4.3.1 Completeness

Completeness can be defined by the percentage of total useable points from the set of total data points collected, analyzed, and available. A formula for estimating completeness is presented in Section 14. Data points may not be useable if sample holding times were exceeded, quality control criteria were not met, and it is not possible to re-analyze the sample. Also, data points may not be useable if sample bottles were damaged during shipment to the laboratory. Completeness is expected to be at least 90 percent for FEMP projects.

If sufficient valid data points are not obtained to meet project objectives, the valid data obtained shall be used and additional sampling and analysis may be considered to meet project objectives.

Example: Fifty soil samples are collected and analyzed. After data validation, forty four data points are determined to be valid. Completeness is estimated as (44/50) x 100 = 88 percent. Completeness was not achieved.

4.3.2 Representativeness

Representativeness is a qualitative parameter based on professional judgement that reflects the design of the sampling program, standard operating procedures, the proper selection of sampling locations, and collection of a sufficient number of samples. Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at sampling points, or an environmental condition.

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Requirements for performing and documenting surface geophysical surveys are presented in Appendix J. PSPs shall specify the method and instruments to be used, grid spacing, speed at which survey is to be conducted, information desired, and frequency of duplicating lines for quality control purposes. A minimum of five percent of the total linear distance of the survey shall be duplicated. Provisions for verifying interpretations through use of borings or excavations shall be included.

Project-specific log forms shall be maintained with information recorded as specified in Appendix J.

Operators shall be trained in use of equipment, and training shall be documented in project files as specified in Section 4. Instruments shall be operated in accordance with manufacturer instructions. If these instructions are not used, a complete description of variations along with justification shall be provided in the PSP, or the situation shall be presented as a variance as specified in Section 15.

5.3.3 Geotechnical Testing

All geotechnical testing must be conducted to the requirements of this document. Data Quality Objectives must be prepared and used as the basis for the development of the Project Specific Plan. All testing methods must be identified in the Project Specific Plan.

5.4 FIELD RADIOLOGICAL CONTAMINATION SURVEYS

Radiological contamination surveys at FEMP are conducted to determine personnel protection requirements, monitor for or detect releases of radioactive materials, and screen samples for laboratory analyses for gross characterization of areas or materials for the presence of radiological contaminants. These include site-wide field surveys conducted during the remedial investigation/feasibility study.

Surveys are conducted in accordance with DOE Orders 5400.5 and 5480.11 in support of activities such as decontamination and decommissioning of facilities and equipment, construction, and release detection. Radiological contamination surveys in support of Comprehensive Environmental Response, Compensation, and Liability Act activities include health and safety monitoring in the field and screening of samples to determine need for laboratory analysis, laboratory licensing requirements, and shipping and packaging requirements. Such surveys are conducted in the field to characterize an area, a facility, or equipment for contamination.

Requirements for health and safety contamination surveys are included in FEMP Health and Safety Department procedures. Requirements for screening of samples are included in Section 6 and Appendix K. Requirements for radiological surveys follow.

Contamination survey techniques at FEMP shall be based on standard nuclear industry techniques combined with process knowledge of potential contaminants at the site. Field radiological contamination surveys may include loose alpha and beta/gamma surveys and fixed alpha and beta/gamma surveys.

Loose contamination is defined as radiological contamination, including soils and sediments, that can be readily removed from a surface by collecting a smear sample. Surveys are performed for area characterization, determining level of personnel protection required, ensuring that vehicles and packages meet Department of Transportation requirements (Section 6), and identifying free releases.

Fixed contamination is defined as radioactive contamination that has become part of the structure being surveyed at conditions prevailing at the time of the survey. Fixed contamination cannot be measured with smear samples; it must be measured directly from the material of interest.

Total contamination of a material or structure is defined as the sum of loose and fixed contamination. Direct survey techniques are used to measure the amount of total activity on various surfaces.

Scoping requirements for radiological contamination surveys shall be documented in PSPs and shall include the following.

- Regulatory driver or other reason for conducting survey
- Types of radiation expected
- Types of measurement equipment plus calibration and operating requirements
- Types of samples to be collected (e.g., smears, surface soil, sediment)

The following applies to instruments used for radiological field screening.

- Instruments used shall be calibrated at least annually and after any adjustments or repairs and in accordance with manufacturer's specifications. Response shall be checked daily using a source of known activity.
- Field survey procedures shall specify the type of instrument to be used, specifications for geometry of detector and source used, maximum speed allowable for the specified instrument, and maximum allowable background for given lower limits of detection.
- The lower limit of detection for instruments used shall be determined so that a 95-percent confidence level is achieved.
- The type of material surveyed shall determine the survey technique used.
- Survey methodology and techniques shall be specified in PSPs.

6.2.2.2 **General Ground-Water Sampling Requirements.** The primary technical consideration in ground-water sampling is to obtain a representative sample of the ground-water body at the well location. Additionally, ground-water sampling at FEMP must meet certain requirements in order for subsequent data to be used by the CERCLA program. Procedures for collecting ground-water samples are provided in Appendix K. Additional requirements specific to a project may be included in PSPs.

6.2.2.3 **Parameter-Specific Sampling Procedures.** Ground-water samples are collected from monitoring wells and piezometers for volatile organic compounds, acid and base-neutral extractable compounds, inorganic parameters, and radionuclide parameters in accordance with procedures provided in Appendix K.

6.2.2.4 **Sampling Ground-Water from Private and Other Production Wells.** Private water wells near FEMP have been sampled as part of FEMP programs, including the REMP and RI/FS. DOE has authorized sampling of private wells by FEMP personnel when requested, and they may be sampled during a routine project or at request of the property owner. Data collected from private wells may be qualified for certain uses. Procedures for collecting water samples from private or other production wells are included in PSPs. Other procedures are provided in Appendix K.

6.2.3 Surface Water Sampling

Surface-water sampling is currently being conducted at FEMP. Samples from Paddys Run and the Great Miami River are collected routinely in accordance with National Pollutant Discharge Elimination System (NPDES) requirements as part of routine monitoring. Samples have also been collected in support of RI/FS.

Procedures and practices are described in Appendix K for collection of water samples from streams, ponds, lakes, rivers, springs, and seeps. Two different techniques are used for collecting surface water samples: grab sampling and composite sampling, which are discussed in Appendix K.

6.2.4 Waste-Water Sampling

Waste-water sampling is regulated by the Ohio Environmental Protection Agency (OEPA) under the Clean Water Act. As such, data are collected in accordance with permit-specific requirements. Samples are also collected for DOE environmental monitoring purposes and to fulfill requirements of the 1986 Federal Facility Compliance Agreement.

6.2.4.1 **Purpose of Data Collection Activity.** NPDES is a statutory requirement under Title IV, Section 402, of the Clean Water Act. Regulatory authority is provided under 40 Code of Federal Regulations (CFR) 122. This system requires that point source discharges into the nation's waterways have a permit that stipulates allowed limits for certain pollutants entering a particular body of water. The Feed Materials Production Center (now FEMP) was issued an NPDES permit renewal (number 1I000004*BD) on 12 February 1990 that expires on 9 February 1995. The permit covers two outfalls to receiving streams and five internal monitoring points located throughout FEMP including remediation process-related waste water, storm water, and sanitary-waste water. The permit is based on both technology-based and water-quality-based limitations depending on water-quality goals of OEPA and the best available technology for treating waste waters specific to an industry. Permitted discharges are as follows.

- 1I000004001: manhole 175; outfall effluent to the Great Miami River
- 1I000004002: spillway outfall from the storm-water retention basin to Paddys Run
- 1I000004601: sewage-treatment-plant effluent part stream after disinfection
- 1I000004602: general sump effluent part stream to manhole 175
- 1I000004604: storm-sewer, lift-station effluent part stream to manhole 175
- 1I000004605: effluent part stream from biodenitrification effluent-treatment system to manhole 175
- 1I000004606: storm-water retention basin pump station effluent part stream to manhole 175

NPDES includes a self-monitoring program to ensure compliance with permit limits. The program consists of sampling waste water, analyzing it for regulated parameters, and reporting results in a monthly discharge monitoring report, which is the end use of the data for FEMP. However, OEPA collects these data plus data from other facilities discharging into waters of the state and uses it to track and regulate water quality in Ohio.

In addition to NPDES requirements, FEMP routinely monitors waste-water discharges on a per-work-shift basis. These data become part of the waste-water treatment plant records. Uranium data are reported monthly to EPA as required under the Federal Facility Compliance Agreement attachment to the 1991 amended consent agreement.

FEMP has an ongoing program of sampling, analyzing, and reporting as required by its NPDES permit, the Federal Facility Compliance Agreement, and DOE. A sampling schedule is developed for the year by the Environmental Compliance and Quality Assurance Department to ensure that, over the course of time, the reported data provide an accurate picture of the volume and nature of waste-water flow in the permitted discharges.

Sampling and analysis requirements are regulated pursuant to 40 CFR 136. The FEMP permit defines the applicable regulation as that version of 40 CFR in effect on 1 July 1989, the effective date of the permit.

The Utilities Section is responsible for sample collection and for operation and maintenance of monitoring equipment such as continuous pH monitors and flow meters. The section is also responsible for operation of automatic samplers and for ensuring that proper techniques are used for grab sample collection when an automatic sampler is not or cannot be used.

The FEMP sampling and analysis management coordinator is responsible for disseminating samples to appropriate laboratories in compliance with specified sample custody (Section 7) and preservation requirements. The laboratories are responsible for analysis of samples including proper use and calibration of analytical equipment and implementation and verification of documented QA/QC requirements.

6.2.4.2 **Field Procedures.** The NPDES permit requires that effluent be monitored continuously for pH at every permitted sampling location except the general sump and for flow when a discharge occurs at each sampling location. Meters are in place to fulfill both permit requirements. Procedures for collecting flow meter information for each NPDES outfall that requires total daily flow reports are provided in Appendix K.

An NPDES sampling plan has been developed and is on file with OEPA. The plan identifies samples to be collected weekly under NPDES and contains information relative to location, type of container, number and volume of samples, type of analysis, preservation method, and lab destination. The basic requirements for NPDES sampling are specified in Appendix K.

FEMP participates in a quality assurance program under the authority of Section 308 (a) of the Clean Water Act. Periodically, samples of the same type of normally tested constituents are sent to FEMP for analysis. Analysis is performed and findings reported to EPA or their designated contractor in accordance with instructions provided with the samples. Results are compared to the true values to determine accuracy of FEMP laboratory analyses.

6.2.4.3 **Additional Sources of Information.** Sampling procedures are governed by 40 CFR 136. FEMP standard operating procedures are implemented for waste-water sampling and analysis and are available upon request from the DOE Fernald Office. References of importance are as follows.

- *Manual of Sampling, Analytical, and Reporting Procedures for Wastewaters.* (Ohio Environmental Protection Agency, 1976).

- *Standard Methods for the Examination of Water and Wastewater.* (American Public Health Association, 1989).

- *Annual Book of Standards.* Part 23, "Water; Atmospheric Analysis" (American Society for Testing and Materials, 1991).

Analysis procedures used in FEMP laboratories for testing waste water are identified in Appendix G.

6.2.5 Compliance with DOE Order 5400.5

The FEMP is required to monitor all liquid effluent to comply with DOE Order 5400.5 (U.S. Department of Energy 1990). Currently operating systems are described in paragraph 6.2.4 and Appendix K.

6.3 SOLID MATRIX ENVIRONMENTAL SAMPLES

6.3.1 Surface Soil Sampling

Surface soil samples are from soils that can be collected with manually operated, hand-held tools and that usually occur within three feet of the land surface. As part of routine monitoring, samples are collected by FEMP prior to excavation in order to characterize the soil for presence of hazardous or radioactive constituents. Surface soil samples have also been collected as part of the RI/FS. Procedures for collecting samples are provided in Appendix K.

6.3.2 Sediment Sampling

Sediments are materials that have been transported from their place of origin by fluid action and redeposited. Stream sediments are of the most interest at FEMP. Sediment sampling in Paddys Run and the Great Miami River is conducted for routine characterization. Sediments have also been analyzed as part of the RI/FS. Specific sampling stations are documented in PSPs. Procedures for collecting sediment samples are provided in Appendix K.

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Section 7

SAMPLE CUSTODY

Sample custody procedures and documentation at FEMP are conducted in accordance with guidelines in the EPA Region V Model Superfund Quality Assurance Project Plan (U.S. Environmental Protection Agency, 1991), which are derived from EPA sample custody protocols described in ***NEIC Policies and Procedures***, EPA-330/9-78-001-R (revised May 1986). Custody requirements are addressed in three parts: (1) sample custody and handling in the field, (2) custody during laboratory receipt and analysis, and (3) evidence files. Sample custody flowchart is shown in Figure 7-1 of Appendix A.

A sample or evidence file is considered in the custody of a person if any one of the following are true.

- The person has physical possession of the sample or file.
- The sample or file is in view of the person, after being in possession.
- The sample or file is placed in a secure location by the custody holder.
- The sample or file is in a designated secure area.

Environmental samples at Analytical Support Levels (ASL) B (sub-level 1), C, and D require complete custody documentation. ASLs B (sub-level 2) and E samples shipped to off-site facilities or that have custody transferred on site also require complete custody documentation. ASLs B (sub-level 2) and E analyses performed at FEMP without custody transfers require completion of field and laboratory documentation as appropriate.

Compliance with sample packaging and shipment requirements in Section 6 and the custody requirements in this section will provide adequate documentation of sample custody from the time of sample collection to final disposition.

The ERMC accepts full responsibility for ensuring that all off-site laboratories' chain of custody (COC) procedures will be contained in written Quality Assurance Plans or SOPs, and that these COC procedures are fully consistent with the field COC procedures defined within.

7.1 FIELD PROCEDURES

The FEMP project manager is responsible for implementation of sample custody procedures.

The Designated FEMP Quality Assurance Organization is responsible for verifying that sample custody procedures are implemented and followed.

The field team leader or designee is responsible for the care and custody of the samples collected until they are transferred to a transporter or an analytical or processing facility. The actual sample collector must sign the chain of custody, and any transfer of the sample within the sampling team will be documented on the chain of custody. Any transfer of sample custody from the original samplers in the field must be documented by double transfer signature on the SAR/CR. The timeframe between sample collection and the arrival at at the sample processing facility shall be minimized to ensure that all holding times can be acheived by the lab. All samples requiring refrigeration will immediately be placed in coolers that already have ice or other cooling agents added. The field procedures shall be conducted as follows:

1. The Sitewide Analysis Request/Custody Record (SAR/CR) (Form 7-1, Appendix B) shall be generated either prior to or at the point of sample generation for transferring custody on site. If samples are shipped to an off site laboratory by a commercial carrier, an Offsite Custody Transfer Record (OCTR) form shall be used to transfer custody. Samples to be shipped off site shall be packaged in accordance with all applicable DOT regulations.

2. Print out duplicate sample labels containing sampling information for each individual sample as specified in section 7.1.3. Sample labels may be printed from a computer or handwritten using black waterproof ink. One label shall be permanently affixed to the sample bottle, while the second label shall be temporarily affixed to the same sample bottle.

3. Collect only the number of samples needed to represent the media being sample. As much as possible, determine the quantity and types of samples and sample locations prior to the actual field work. The number of persons having sample custody shall be minimized.

4. Record the information concerning the sample collection in a field log as specified in section 7.1.2. Record the date and time of collection on the SAR/CR once a sample has been collected. All samplers involved in the sample collection shall sign the SAR/CR.

5. Seal the sample immediately upon sample collection using custody tape around the lid of the jar/bottle in such a manner that when the jar is opened, the tape would be destroyed. The sampler will initial and date the custody tape prior to sealing the sample jar. Figure 7-2 (Appendix A) is an example of sample custody tape.

6. If the samples require refrigeration, the samples are placed immediately in a cooler which is to be kept under the rules of custody.

7. Deliver the samples directly to an analytical or processing facility, a transporter, or lock the samples in a secure area for the night when the sample collection has been completed for the day. For field personnel shipping samples directly to an offsite laboratory, see section 7.1.5. If the samples are not transferred immediately, then the SAR/CR shall contain the name of the storage area (room number) and state how custody was maintained (locked room or sealed cooler).

8. If analysis is completed in the field, the rules of custody shall apply (e.g., the sample always in possession of sampler or under lock and key).

9. The FEMP project manager or designee shall review activities to determine whether proper custody procedures were followed during field work and to decide if additional samples are required.

7.1.1 Sample Tracking and Control Documentation

Sample custody shall be documented from time of collection through disposal and final disposition of the sample shall be documented. The following sample custody records shall be maintained.

- Bound field log book with sequentially numbered pages or sequentially printed and numbered daily field activity log forms
- Sample identification and labeling
- Three-part SAR/CR

The first two items shall be completed for all samples regardless of ASL. The SAR/CR is required for samples shipped off site or for samples analyzed on site by a party other than the sample collector (i.e., a custody transfer occurs).

7.1.2 Daily Logs

Data collection activities shall be recorded in a bound field log or on daily field log forms (Form 5-1, Appendix B). Entries shall describe activities sufficiently for the sampling team to re-construct a particular situation without reliance on memory.

Field logs shall be bound field survey books or notebooks with sequentially numbered pages, preferably with water-resistant paper (standard engineering field book). Logs shall be

assigned to field personnel. They shall be stored in a secure area when not in use. Each log shall be identified by a project-specific control number.

Use of daily log forms was approved by EPA for the Remedial Investigation/Feasibility Study program (U.S. Department of Energy, 1988). Similar forms are used by other programs at FEMP. Each form shall be sequentially printed and numbered and logged into the data management system. Requirements for daily log entries at FEMP are provided in Section 5.

7.1.3 Sample Identification and Labeling

Samples shall be marked for identification from the time of collection and packaging through final disposition through the use of sample labels. Duplicate labels shall be printed or handwritten in black waterproof ink and attached to the sample jar/bottle. The sample label shall include the following information:

- Sample ID
- Date sampled
- Time sampled
- Material name
- Sample type
- Preservatives
- Container type
- Gross weight
- Collectors initials
- Tare weight
- Comments (CMT)
- Chain of Custody Number (COC #)

The duplicate sample label shall be attached to the original sample label by a perforation. The backing shall also be perforated at the point of the duplicate label. When the original

label is attached to the sample bottle, the backing shall be left attached to the duplicate label which will stay attached to the original label.

Form 7-2 (Appendix B) is an example of sample labels. Label A is automatically generated by the Laboratory Information Management System (LIMS) for known samples to be taken. Label B is for samples which does not have labels already preprinted for them. An established sample numbering system will be automatically generated for each sample (Label A) using a 10 digit number that would be assigned to each sample in consecutive order. The generated number will appear on the sample label as the SAMPLE ID and in a barcode format for the known sample ID. An example of this numbering system would be 1000000101: the one hundredth and first sample container logged into the LIMS system.

7.1.4 Request for Analysis

Analysis requests shall be prepared to specify the testing or analyses program required for collected samples using Form 7-1 (Appendix B). Analysis requests shall be confirmed prior to sample collection and coordinated by the FEMP sampling and analysis management coordinator. The analysis request shall be hand-carried or telefaxed to a FEMP-approved analytical laboratory (Table 3-1, Appendix A) to ensure laboratory capacity prior to sample collection. The laboratory project manager or representative shall sign the copy and transmit it by telefax to the FEMP project contact, committing laboratory resources to proper, on-time completion of requested analyses. Failure of the laboratory project manager to respond within one working day shall be interpreted as a lack of capacity, and other arrangements shall be made for sample analysis. Other properly documented communications with subcontractor laboratory personnel may substitute for this procedure if defined in a project-specific plan.

If the laboratory initially contacted cannot perform the analysis, an alternate FEMP-audited and approved subcontractor laboratory shall be chosen by the FEMP project contact. The analysis request process shall be repeated. This process eliminates capacity problems and excessive sample turn-around times. Record the following information from the analysis request process for the project file.

- Project name and number
- Number of samples
- Date samples shipped
- Required report date and turnaround times for testing or analysis
- Contact (with telephone number) for receipt of analytical report and invoices
- Sample identification numbers

- Sample media
- Sample volume collected and preservatives used
- Types of analyses required

Information on the SAR/CR shall be consistent with that on the sample labels. When a discrepancy exists, the laboratory project manager or representative shall notify the FEMP project contact immediately. The written discrepancy resolution shall be transmitted from the FEMP project contact to the laboratory within one working day of notification by the laboratory.

7.1.5 Shipment of Samples to Off Site Laboratory

Samples collected at FEMP within the scope of this SCQ shall be accompanied by the OCTR (Form 7-1, Appendix B). Instructions for its completion are included with the form.

The SAR/CR shall follow the samples from sample collection to sample disposal. If the samples are delivered to a processing facility for shipment to an off site laboratory, an OCTR shall accompany the sample shipment in place of the SAR/CR. The timeframe between arrival of samples and delivery to the analytical laboratories shall be minimized to ensure that all holding times can be acheived by the laboratory.

The shipment of samples to off site laboratories shall be done as follows:

1. The processing laboratory shall verify that the sample seals are intact and check sample identification on sampling containers against that listed on the SAR/CR. When discrepancies exist, record that on the SAR/CR and sign and date the notation. Notify FEMP project contact immediately and store the sample(s) until a resolution is received from FEMP project contact.

2. The processing laboratory shall originate and sign the OCTR at time of sample shipment and file a copy of the OCTR with the original SAR/CR. The duplicate labels are **not** removed from the sample bottles until the samples are received in the laboratory.

3. Maintain sample preservation (refrigeration) from receipt of samples until sample shipment. It is the responsibility of the processing laboratory to ship samples in a manner as to maintain sample preservation requirements during shipment.

4. Package the samples properly for off site shipment as specified in Section 6 and dispatched to the laboratory for analysis. A signed OCTR shall be enclosed in a watertight container (e.g., a zipper lock plastic bag) and shall accompany each shipment. The bill of lading (waybill) number shall be noted on the OCTR (when applicable) before sealing in the container.

5. Secure shipping containers with custody tape and FEMP custody seals (Figure 7-2 and Figure 7-3 in Appendix A) and/or locked if appropriate, so that access to the container can be gained only by breaking a seal. The custody seal number shall be documented on the OCTR. If the shipping container is secured with custody tape, the packager shall initial and date the custody tape prior to placement on the shipping container.

6. If samples are sent by common carrier, a bill of lading (waybill) shall be used. Receipts for bills of lading shall be retained as part of permanent custody documentation.

7. Commercial carriers are not required to sign the custody form as long as forms are sealed inside the sample container and the custody seals remain intact.

7.2 ANALYTICAL LABORATORY

7.2.1 Laboratory Sample Receipt

The laboratory personnel are responsible for the care and custody of samples from the time they are received until the sample is exhausted or returned to the FEMP project contact. Within eight business hours of sample receipt by a laboratory, the designated sample custodian, laboratory project manager or representative shall examine the samples as follows:

NOTE

If samples arrive with either an incorrect SAR/CR, or OCTR for off site laboratories, or no SAR/CR or OCTR, custody is broken and analysis results can only be used for information purposes only. Notify FEMP project contact by telephone or telefax and store samples until a resolution is received. Documentation of the discrepancy and its resolution by the FEMP project manager shall be contained in a **laboratory non-conformance form** (Form 15-3, Appendix B). This form shall become a permanent part of the project file.

NOTE

The laboratory project manager or representative shall notify the FEMP project contact of discrepancies noted during sample receipt by telephone immediately and within twenty-four hours in writing (by telefax if necessary). The laboratory project manager may use a **laboratory non-conformance form** with the SAR/CR or OCTR attached. The FEMP contact shall advise the laboratory of disposition to be made of samples within twenty-four hours of notification by telephone or telefax followed in writing.

1. Examine the shipping container custody seals for breakage and tampering, if

applicable. Record condition of custody seals on the SAR/CR or OCTR.

2. Measure temperature of shipping containers holding samples that require refrigeration with a calibrated, standard laboratory thermometer and record temperature on the SAR/CR or OCTR. If the temperature is outside the range of 2 to 6 degrees Centigrade, document this information on a laboratory non-conformance form and notify the FEMP project contact. Store samples until directions for disposition are received.

3. Examine custody seals on samples for breakage and tampering. Record condition of custody seals on the SAR/CR or OCTR. Check sample identification on sample container against that listed on the SAR/CR or OCTR.

4. When applicable, verify the bill of lading (waybill) number against that on the OCTR. If the waybill number is not written on the OCTR, verify with the FEMP project contact that the number on the waybill is identical to that recorded in the project files.

5. Sign and date the OCTR and attach waybill to it (when applicable). Remove the temporary duplicate sample label from the sample bottle and affix them permanently to the back of the top copy of the SAR/CR or OCTR. This is to verify the identification of the samples that were sent for analysis. Off site laboratories return the signed top copy of the OCTR to the FEMP project contact. On site laboratories, distribute the bottom copy directly to the samplers (green) at time of delivery and the middle copy directly to the FEMP project contact (yellow).

6. Assign a unique laboratory tracking number to each sample and affix a label with the number onto each sample container if the FEMP sample number is not used for internal laboratory tracking purposes. Numbers shall be assigned sequentially as samples are coded in. Log sample receipt information, including holding times, test assignments, and anticipated reporting date into laboratory information management system. If sample holding time has been exceeded or cannot be met, notify FEMP project contact and complete a laboratory non-conformance form. Enter samples in laboratory tracking system with the following information.

   - Project identification number
   - Sample numbers
   - Types of samples
   - Date received in the laboratory

7. Store samples as required in laboratory facility. Custody rules shall be followed throughout the life of the sample in the laboratory.

8. Each laboratory must follow its established system for assuring that sample custody is

documented for all movements of both the sample and its extracts/digestates. Each laboratory shall have an approved, controlled SOP that gives stepwise intralaboratory custody procedures complete with copies of documentation to be used. This SOP shall be approved by the FEMP project contact before use. Any changes to the SOP shall also be approved by the FEMP project contact before installing. Transfers that shall be documented include:

- from sample receiving to sample preparation,
- return of original sample to sample receiving,
- from sample extraction to digestion,
- from digestion to analysis,
- from analysis to storage of both original sample and extract,
- from sample storage to disposal.

9. All documentation of sample custody within the laboratory shall become a permanent part of the laboratory project files.

10. The bottom copy the OCTR shall be signed and dated and accompany the samples when samples are shipped back to the FEMP by the offsite laboratory after approval by the FEMP project contact. Upon receipt at the FEMP the contents of the shipment shall be check against the accompanying OCTR. If any discrepancies exist they shall be noted on the OCTR and the FEMP project contact contacted immediately.

11. The original (white) copy of the SAR/CR or OCTR is to be held in the laboratory project files until either the samples are disposed of or returned to the FEMP customer. At that time the original copy of the SAR/CR or OCTR is to be placed in the FEMP project files with the duplicate sample labels attached to the back. A copy is to be kept in the laboratory project files.

7.2.2 Assignment of Processing Priorities

The laboratory manager is responsible for assigning priorities to samples to ensure that holding times will not be exceeded during the time needed to process the samples through the laboratory work stream.

7.2.3 Sample Holding and Disposal

It is essential to track the final disposition of each sample because of potential liabilities incurred through improper disposal of samples. Therefore, the SAR/CR for the sample shall be completed with the final disposition of the sample. Analysis will confirm if the sample contains non-hazardous or hazardous waste or non-radioactive or radioactive material as defined by the Department of Transportation and the Comprehensive Environmental Response, Compensation, and Liability Act. Non-hazardous and non-radioactive samples shall be disposed of in accordance with standard laboratory practices or returned to FEMP as

specified by the FEMP project contact.

The disposition of hazardous and radioactive samples shall be determined on a laboratory specific basis. The majority of these samples will be returned to FEMP prior to determination of final disposition.

When environmental samples are held for re-analysis, proper environmental control and holding times shall be observed. When re-analysis is not anticipated, but samples must be held for a specific time, environmental conditions for storage will not be observed.

When hazardous waste samples are held for re-analysis, they shall be stored according to their hazard classification under the Resource Conservation and Recovery Act, defined environmental conditions, and holding times.

When radiological samples are held for re-analysis or for a specific time, they will be stored in accordance with DOE regulations, individual laboratory licensing requirements, and environmental conditions.

When mixed waste samples are held for re-analysis or for a specific time, they shall be stored in accordance with DOE regulations, their hazard classification under the Resource Conservation and Recovery Act, and environmental conditions.

Special arrangements may be necessary for samples maintained longer than six months.

Returned hazardous waste and radiologically contaminated samples shall be transported to FEMP in accordance with 49 CFR 171 through 177 (Section 6). Record disposition on the SAR/CR and file results.

FEMP shall maintain a sample disposal log defining methods for disposal of FEMP-generated samples. Contract laboratories shall provide information identifying sample disposal methods to FEMP. Following are examples of sample disposition.

- Consumed in analysis
- Returned to FEMP
- Stored
- Non-hazardous/non-radioactive-contaminated samples disposed of in accordance with standard laboratory disposal practices
- Hazardous waste/radiological-contaminated samples disposed of in accordance with standard laboratory disposal practices

Disposal methods of samples analyzed at FEMP shall be documented on the SAR/CR.

7.3 EVIDENCE FILES

Evidence files for analytical data are maintained at FEMP and contain relevant records, reports, correspondence, logs, field logs, original laboratory data packages, pictures, subcontractor reports, SAR/CRs, and data review reports. All information supporting FEMP CERCLA decisions shall be included in the final evidence file as support for the Administrative Record in accordance with the 1991 amended Consent Agreement.

Evidence files shall be in the custody of the FEMP project manager responsible for generating the data. They are kept in a locked, secure storage area. The file custodian is the FEMP Administrative Record Coordinator, who controls the central file for environmental sampling and analysis at FEMP in addition to managing the Administrative Record. The final evidence file shall be maintained for at least ten years after remedial activities at FEMP are complete. If DOE decides to discard the files after this time, the 1991 amended Consent Agreement specifies that the files be offered to EPA.

Data generated by subcontractors for FEMP are the property of DOE and shall be maintained under contract at the facility where it was generated. No files shall be discarded without written consent of the FEMP project manager. If a storage, security, or other problem is discovered at the facility, files shall be transferred to FEMP.

7.4 REFERENCES

U.S. Department of Energy. 1988. ***Remedial Investigation and Feasibility Study, Feed Materials Production Center, Fernald Ohio, Work Plan.*** Revision 3. prepared by Advanced Sciences, Inc. for U.S. Department of Energy, Oak Ridge Operations. March 31, 1988.

U.S. Environmental Protection Agency. 1991. ***Region V Model Superfund Quality Assurance Project Plan.*** Chicago, Illinois. May 1991.

Westinghouse Environmental Management Co. of Ohio. 1992. ***Processing the Site-Wide Analysis Request/Custody Record for Sample Control.*** Procedure SSOP-0018. February 14, 1992.

Westinghouse Environmental Management Co. of Ohio. 1991. ***Packaging, On-Site Movement, and Off-Site Shipment of Material.*** Procedure FMPC-0314. December 31, 1991.

Section 8

CALIBRATION PROCEDURES AND FREQUENCY

Measuring and test equipment used in the field and the laboratory shall be controlled by formally prescribed calibration requirements. Equipment shall be of the type, range, accuracy, and precision necessary to provide data compatible with the Analytical Support Level (ASL) (Section 2) specified in applicable Data Quality Objectives (DQO) (Appendix C) or Project-Specific Plans (PSPs). Calibration of measuring and test equipment shall be performed using documented and approved procedures. When available, accepted procedures published by the American Society for Testing and Materials, the EPA, the National Institute of **Standards and** Technology, or manufacturer equipment manuals shall be used. Variance from these procedures shall be justified and documented in PSPs.

8.1 RESPONSIBILITIES

Responsibility for calibration requirements and documentation is as follows.

8.1.1 Analytical Laboratory Equipment and Instrumentation

Responsibility for ensuring that calibration requirements are met rests with the laboratory manager, whether on-site or a subcontractor.

Individual laboratory analysts responsible for performing analytical procedures shall maintain required calibration logs.

8.1.2 Field Equipment and Instrumentation

The assigned FEMP project manager or designee shall be responsible for ensuring that field equipment and instrumentation calibration requirements are met as specified in Appendix G, Appendix I or the applicable PSP.

Field users of calibrated instruments are responsible for inspecting calibration status before using the equipment and documenting the inspection in the calibration log.

8.2 CALIBRATION PROCEDURES

Calibration procedures for measurement and test equipment used in the field shall be specified in Appendix I or the applicable PSP. Equipment used in analytical laboratories shall be specified in the applicable PSP or the method in Appendix G. After identifying the appropriate procedure for calibrating the subject instrument, the source of the

procedure shall be recorded and implementation shall be documented in the instrument-specific calibration log.

When available, accepted procedures published by American Society for Testing and Materials, EPA, or the equipment manufacturer shall be used.

8.2.1 Procedure Requirements

The following requirements shall be included in procedures for measurement and test equipment calibration in PSPs.

- A list of field measurement and test equipment to be used on the project by manufacturer, type, and identifier
- Source of the calibration procedure or the procedure itself if not otherwise available
- Provision for recording unique identification numbers for equipment requiring calibration on sampling or field logs. (The number assigned may be the manufacturer serial number, a calibration system identification number, or other equipment-unique identifier.)
- Reference standards with known relationships to nationally recognized standards (e.g., National Institute of Technology) or accepted values of natural physical constants (If national standards do not exist, reference and document the basis for calibration.)
- Standards required for the specified ASL
- Maintenance and inspection requirements prior to use of equipment
- Prescribed intervals for calibrating measurement and test equipment
- Calibration log and minimum required information

8.2.2 Calibration Frequency

Frequency of calibration shall be determined based on the following elements.

- Type of equipment
- Inherent stability
- Manufacturer recommendations

- Values given in national standards
- Intended use
- Instrument response to spot checks with standards
- Instrument response time
- Experience

8.2.3 Calibration Documentation Requirements

Documentation shall be maintained for each piece of calibrated measurement and test equipment to indicate that established calibration procedures have been followed. Calibration records for field equipment shall be retained in project files. Records for laboratory equipment shall be maintained by the laboratory. At a minimum, the following information shall be recorded and available for project use.

- Equipment identification number
- Type and manufacturer of equipment
- Calibration frequency and acceptable tolerances
- Calibration dates, results, and any problems encountered during calibration
- Identification of calibration procedures employed
- Identification of personnel and organization performing calibration
- Dates of maintenance and inspections
- Certification or statement of calibration provided by manufacturer or external agency, if applicable
- Statement of calibration acceptance or failure
- Disposition of equipment that fails calibration

8.2.4 Equipment Failure

Equipment that cannot be calibrated or becomes inoperable during use shall be tagged and removed from service until it can be repaired and recalibrated to the acceptance criteria

specified in the applicable procedure. Equipment that cannot be repaired shall be permanently removed from the program and replaced.

8.3 FIELD MEASUREMENT AND TEST EQUIPMENT FOR ASLs A AND B

Calibration checks shall be performed on all field instruments before use each day. If the instrument does not meet the criteria specified in Appendix G, Appendix I or the PSP, use of the instrument shall be discontinued until the appropriate corrective action has been taken.

The responsible FEMP project manager or designee shall maintain a list of field measurement and test equipment used for the collection of project data. The list shall include the following information.

- Identification number
- Description of equipment
- Manufacturer of equipment
- Required calibration frequency
- Number and title of applicable calibration procedure
- Source of procedure

The FEMP project manager or designee shall validate the list for adequacy and review the calibration procedures periodically to ensure adequacy for the specified ASL (Section 2). Procedures for calibration of commonly used field equipment are provided in Appendix I.

8.4 ANALYTICAL LABORATORY INSTRUMENTATION AND EQUIPMENT FOR ASLs B, C, D, AND E

Method-specific calibration requirements are specified in Table G-2. These include concentration of standards, frequency for initial and continuing calibration requirements, and quality control acceptance criteria.

If initial calibrations do not meet acceptance criteria, analyses shall not be performed, corrective action shall be taken, and the calibration standards shall be re-analyzed. If continuing calibration check samples do not meet acceptance criteria, corrective action shall be taken and the instrument shall be recalibrated. Samples analyzed since the last calibration that met specified criteria shall be re-analyzed.

If deviations from procedures are necessary, the FEMP project contact shall be notified immediately, and documentation of the deviation and the reason for it shall be presented in the final analytical report.

Calibration information shall be documented in the applicable calibration log.

8.4.1 Laboratory Equipment Calibration Schedules

Equipment shall be calibrated at least annually or at the time of a repair that affects the function of the equipment. Equipment requiring calibration schedules includes, but are not limited to, the following.

- Ovens and refrigerators
- Automatic/manual pipettors
- Thermometers
- Laboratory balances

8.4.2 Laboratory Instruments

Schedules shall require calibration at least as frequently as the Appendix G method specifies or as specified in the laboratory contract. Instruments requiring calibration schedules include, but are not limited to, the following.

- Liquid scintillation counting systems
- Alpha spectrometer systems
- Alpha/beta counting systems
- Germanium spectroscopy systems
- Alpha scintillation counting instruments
- Gas Chromatograph/Mass Spectrometer (GC/MS)

- UltraViolet/Visible Spectrum (UV/VIS) spectrophotometer
- Thermal ionization mass spectrometer
- Gas Chromatography/Electron Capture Detector (GC/ECD)
- Gas Chromatography/Flame Ionization Detector (GC/FID)
- High Performance Liquid Chromatography with UV
- Inductively Coupled Plasma (ICP) spectroscopy
- ICP/Mass spectrometer
- Flame Technique Atomic Absorption Spectroscopy (FTAAS)
- Graphite Furnace Atomic Absorption (GFAA) spectroscopy
- Cold Vapor Atomic Absorption (CVAA) methods for mercury analysis
- Infrared (IR) spectrometer
- Manual/semi-automated spectrophotometer

Section 9 ANALYTICAL PROCEDURES

9.1 CHEMICAL ANALYSES 1

9.2 RADIOCHEMICAL ANALYSES 1

9.3 HISTORICAL DOE METHODS 2

9.4 USE OF CHEMICAL AND RADIOCHEMICAL PROCEDURES 2

9.4.1 Chemical Procedures 2

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9.5 COMPLIANCE AND PERFORMANCE REQUIREMENT 4

9.6 CALIBRATION STANDARDS FOR CHEMICAL ANALYSES 4

9.7 NATURAL WATERS ANALYSIS 5

9.8 ASBESTOS ANALYSIS 5

SECTION 9

ANALYTICAL METHODS

Analytical methods and associated quality control elements are identified in Appendix G. The objectives of the analytical procedures and quality control elements are to ensure quality at each ASL, to promote comparability of past, current and future data, to ensure completeness and validatability, to ensure compliance with performance criteria and specifications, to promote cost effectiveness and to promote throughput and turnaround time.

The methods included in Appendix G are those either commonly used for FEMP analyses, or those projected to be used in the near future. As new analytical requirements are identified, additional methods will be added.

9.1 Chemical Analyses

Organic, inorganic and various wet chemical analyses are performed at the FEMP for a wide variety of programs encompassing the entire range of ASLs. Such analyses are performed for CWA, SDWA, RCRA and CERCLA programs at the FEMP. Additionally, such analyses are performed for treatability studies, for monitoring various operating plant processes, for environmental monitoring, for QC programs and for routine investigations.

The inorganic, organic and wet chemical methods listed in Appendix G are EPA methods or other standard methods commonly used at CERCLA and RCRA sites and readily performed by the commercial analytical laboratory community. EPA methods include 200 and 500 Series methods (40CFR141), 600 Series methods (40CFR136), SW846 methods (40CFR261) and CLP-SOW methods (EPA-540/G-89-004). Other standard methods include those listed in Standard Methods for the Analysis of Wastewater (latest edition) and those listed in American Society for Testing and Materials (ASTM) publications (latest revision).

9.2 Radiochemical Analyses

Unlike organic and inorganic chemical analytical methods, few standard methods are available for the radiochemical analysis of environmental samples. Additionally, standard established quality assurance/quality control requirements and acceptance criteria are not available for environmental radiochemical methods. As a result, different EPA, DOE and commercial environmental laboratories may have different detection and sample preparation techniques for specific radiochemical analyses. Nonetheless, multi-lab validation studies and inter-laboratory comparison studies have demonstrated that accurate, comparable radiochemical data are obtainable even though different procedures are used.

In view of the above discussion, FERMCO has adopted the approach of utilizing performance based methods for radiochemical analyses. In such methods frequencies and acceptance criteria for quality control, performance parameters are specified. Table G-4 in Appendix G present performance specifications as a function of radionuclide and matrix for the analyses of interest at the FEMP. The specifications represent a consensus from six commercial laboratories as well as from radiochemists at the Fernald site. Ability to meet these performance criteria will ensure comparability of radiochemical data.

9.3 Historical DOE Methods

Because of the presence of radionuclides at the FEMP and other DOE sites, methods have been developed at those sites for the radiochemical and chemical analysis of certain elements (uranium and thorium, for example). Although these methods have a long history of use, they have not been promulgated nor have they been compiled as a "standard" method anywhere due to limited applicability.

Table G-2 in Appendix G lists six such inorganic methods. They include X-ray fluorescence determination of total uranium and total thorium in soils and other inorganic matrices, colorimetric determinations of low concentrations of total uranium and total thorium in waters, soils and wastes; and determinations of high concentrations of total uranium and total thorium in waters, soils and wastes. These methods, because of their historic routine use at the FEMP for a variety of programs, are more appropriately treated as ASL B than as ASL E. These methods are referenced in Table G-2.

9.4 Use of Chemical and Radiochemical Procedures

To ensure consistency and comparability of analytical data, the requirements governing the use of analytical procedures in Appendix G are delineated below and shall be followed.

9.4.1 Chemical Procedures

- All organic, inorganic and wet chemical methods to be used under the jurisdiction of the SCQ shall be listed in the Method Selection Table (Appendix G, Table G-1)

- Organic, inorganic and wet chemical methods to be included in this Table must be standard methods as discussed in 9.1 or historic, routine FEMP methods as discussed in section 9.3.

- The Method Selection Table relates standard analytical methods to analyte group, ASL level, and matrix; and, where appropriate, standard sample preparation methods are also included.

- Analytical methods not listed in the Method Selection Table must either be added at a

later date via a Document Change Request or treated as ASL E methods.

- Methods to be used for ASL C and D analyses shall be delineated in the most recent CLP-SOW. Methods to be used for ASL B analyses will be standard methods or FEMP historic methods as discussed above.

- Utilization of the Method Selection Table will ensure uniformity of analytical method application across the FEMP and at subcontractor laboratories.

- Each analytical method listed in the Method Selection Table (Table G-1) for an ASL B must be accompanied by a Performance Criteria Specification Table in Appendix G (Table G-2).

- Where concentrations, frequencies and acceptance criteria of QC elements are delineated in the referenced methods, they will be adopted without modification on a Performance Criteria Specification table.

- Where concentrations, frequencies and acceptance criteria of QC elements are <u>not</u> delineated in a referenced method, but <u>are</u> required as per SCQ, Section 10 (Internal Quality Control Checks and Frequency), such performance criteria will be specified on a Performance Criteria Specification table.

- CLP-SOW methods do not have a Performance Criteria Specification Table in Appendix G. Such analytical methods are sufficiently rigorous that all necessary performance criteria are spelled out in those methods.

- When CLP-SOWs are not available for an ASL C or D analysis, the most appropriate standard method or FEMP historical method will be upgraded as an ASL E. Performance method specification criteria will be delineated in project specific plans.

9.4.2 Radiochemical Methods

- All radiochemical analyses to be performed under the auspices of the SCQ shall be represented by Radiochemical Performance Criteria Tables in Appendix G.

- The Radiochemical Performance Criteria Tables present performance criteria for QC elements relative to radionuclide species and matrix types. The performance criteria for ASLs C and D are identical. However, data reporting requirements differ for ASLs C and D as denoted in Section 11.3 (Data Reporting). ASL B QC levels differ analytically from ASLs C and D by having shorter counting times in order to reduce analysis times. Performance parameters affected by shorter count times have different acceptance criteria as delineated in the ASL B Radiochemical Performance Criteria Tables.

- Analyses not listed in the Radiochemical Performance Criteria Tables must either be

added at a later date via a Document Change Request or treated as ASL E methods.

9.5 Compliance with Performance Requirement

In order to insure that subcontractor analytical laboratories can perform the analyses in Appendix G, can meet chemical and radiochemical performance specifications, and can report the data in validatable formats, the laboratories will be rigorously evaluated prior to contract award and during contract performance. The guidelines for evaluating laboratory capability to provide analytical services for the FEMP are delineated in numerous sections of this document. Some of the more important sections are delineated below.

- 3.1.5.2 Analytical Laboratory Subcontractors' Requirements
- 3.4 Analytical Laboratory Responsibilities
- 12.4 Laboratory Qualification and System Audits
- 12.4.1 Laboratory Capacity
- 12.4.2 Hazardous Materials Handling Ability, License, Permits
- 12.4.3 Quality Requirements
- 12.4.3.1 Administrative (Items)
- 12.4.3.2 Technical (Items)
- 12.4.4 Performance Evaluation
- 12.4.5 Continuing Satisfactory Performance
- 12.4.6 Quality Assurance Plan
- Appendix E Analytical Laboratory Performance Requirements

Radiochemistry, historical DOE methods and ASL E methods shall be validated prior to use. This method validation data must include information to show that the method can meet all identified data quality objectives and performance criteria. The method and method validation data must be approved by the FEMP Site Sample Management Office prior to analysis of any FEMP samples.

9.6 Calibration Standards for Chemical Analyses

In some standard analytical procedures (SW846, for example) for chemical analyses, exact concentrations and/or range of calibration standards are not given. The following guidelines shall apply to procedures in the Method Selection Table for which ranges/concentrations of calibration standards are not delineated.

- The calibration range should span the expected concentration range of analytes in a sample. If analyte concentrations fall outside the calibration range, the measuring instrument shall be recalibrated or the sample diluted (if applicable).

- The lowest point of the calibration range shall be at or near the Practical Quantitation Limit (PQL) for organic analysis or the Instrument Detection Limit (IDL) for non-organic anaysis. An additional concentration point shall be at the midpoint of the range.

- The highest concentration of calibration standard shall not exceed the linear dynamic range of the detector.

- The concentrations of all standards used in any calibration should be recorded in a laboratory notebook or documented by other hard copy records. Similarly, any sample dilutions must be recorded in a laboratory notebook or documented by other hard copy records.

- Laboratories will not be routinely required to report working range, calibration concentrations/ranges or sample dilutions unless specified as part of the data validation package or in a contract laboratory Statement of Work (SOW). All records must be maintained for audit purposes.

9.7 Natural Waters Analysis

The following field methods for determining properties of natural waters at ASL A are provided in Appendix K.

- pH
- Temperature
- Specific conductance
- Alkalinity
- Redox potential (Eh)
- Dissolved oxygen content

9.8 Asbestos Analysis

Bulk materials and filters will be analyzed for asbestos to identify presence and to monitor airborne concentrations. Analyses shall be performed as specified in 40CFR763.

Section 10 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

10.1 QUALITY CONTROL CHECKS AND PROCEDURES 1

10.2 INORGANIC QUALITY CONTROL 1

10.3 ORGANIC QUALITY CONTROL 2

10.4 RADIOLOGICAL SAMPLE ANALYSIS QUALITY CONTROL 2

10.5 INORGANIC (NON-METALS) QUALITY CONTROL 3

10.6 FIELD QUALITY CONTROL 3

Section 10

INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Internal Quality Control (QC) checks are performed to verify the quality of measurements of field and laboratory investigations and associated tasks. Required frequencies for internal QC checks are specified in Tables 2-2 and 2-4 (Appendix A).

10.1 QUALITY CONTROL CHECKS AND PROCEDURES

QC operations performed to satisfy requirements for Analytical Support Levels (ASL) are defined in specific methods or performance specifications identified in Appendix G.

10.2 INORGANIC QUALITY CONTROL

Types and required frequencies for field and laboratory QC samples for inorganic analyses performed for ASLs B, C, and D are summarized in Tables 2-2 and 2-4 (Appendix A). Definitions of the different types of QC samples are provided in Section 4. QC samples for inorganic analyses may include some or all of the following.

- Preparation (method) blank
- Inductively Coupled Plasma (ICP) interference check
- Inductively Coupled Plasma serial dilution
- Matrix spike analysis
- Laboratory replicate sample analysis
- Graphite Furnace analytical (instrument) spike
- Use of Method of Standard Additions (MSA)
- Laboratory Check Samples

QC acceptance criteria for each of the QC sample types and required corrective actions are specified in the applicable tables in Appendix G. Data reporting requirements are specified

in Section 11. Data validation requirements are described in Section 11 and detailed in Appendix D.

10.3 ORGANIC QUALITY CONTROL

Types and required frequencies for field and laboratory QC samples for organic analyses performed for ASLs B, C, and D are summarized in Tables 2-2 and 2-4 (Appendix A). Definitions of the different types of QC samples are provided in Section 4. QC samples for organic analyses may include some or all of the following.

- Preparation (method) blank
- Surrogate spike analysis
- Laboratory replicate sample analysis
- Matrix spike/matrix spike duplicate analysis
- Retention-time window establishment and retention-time shift evaluation
- Method linear range determination
- Endrin/DDT breakdown product evaluation
- Laboratory check samples

QC acceptance criteria for each of the QC sample types and required corrective actions are specified in Table G-2 (Appendix G). Data reporting requirements are specified in Section 11. Data validation requirements are described in Section 11 and detailed in Appendix D.

10.4 RADIOMETRIC SAMPLE ANALYSIS QUALITY CONTROL

Types and required frequencies for field and laboratory QC samples for radiological analyses performed for ASLs B, C, and D are summarized in Tables 2-2 and 2-4 (Appendix A). Definitions of the different types of QC samples are provided in Section 4. QC samples for radiometric analyses may include some or all of the following.

- Preparation (method) blank
- Matrix spike/matrix spike duplicate analysis

- Tracer analysis
- Laboratory check samples (check-source samples)
- Laboratory replicate sample analysis

QC acceptance criteria for each of the QC sample types and required corrective actions are specified in the applicable Appendix G method table. Data reporting requirements are specified in Section 11. Data validation requirements are described in Section 11 and detailed in Appendix D.

Laboratory check-source results for radiometric analyses must fall within the method-required range. Check-source results will also be examined for high or low bias, or for regular fluctuations within the specified range. If data are biased high or low, or exhibit fluctuations according to a regular trend, the cause of the bias or trend shall be identified and corrected.

10.5 INORGANIC (NON-METALS) QUALITY CONTROL

Types and required frequencies for field and laboratory QC samples for conventional analyses performed for ASL B are summarized in Tables 2-2 and 2-4 (Appendix A) and are specified, as applicable, in each Appendix G specified method. Definitions of the different types of QC samples are provided in Section 4.

QC acceptance criteria for each of the QC sample types and required corrective actions are specified in the applicable Appendix G specified method. Data reporting requirements are specified in Section 11. Data validation requirements are described in Section 11 and detailed in Appendix D.

10.6 FIELD QUALITY CONTROL

The assigned field FEMP project manager is responsible for field activities and QC. Quality Assurance/Quality Control sample requirements for field activities and measurements are specified in Section 5 and Appendix J (field procedures). QC acceptance criteria for each of the QC sample types and required corrective actions are specified in the applicable method in Appendix J. Data reporting requirements are specified in Section 11. Data validation requirements for field activities are described in Section 11 and detailed in Appendix D.

Section 11

DATA REDUCTION, VALIDATION, AND REPORTING

The following procedures shall be used by FEMP personnel, the FEMP laboratory, and subcontractor laboratories for data reduction, validation, and reporting as applicable for each Analytical Support Level (ASL) (Section 2). The Data Validation Plan is described in Appendix D.

11.1 DATA REDUCTION

Data reduction is the process of converting raw data to a useable format beginning with data processing and continuing through review and reporting of results as shown in Figure 11-1 (Appendix A). Data reduction can either be performed by the analyst who obtained the data or by another analyst. Data review begins with the laboratory manager, field supervisor, or designee who verifies that data reduction has been correctly performed. In general, data shall be reduced in one of the following ways.

- Manual computation of results directly on the data sheet or on attached calculation pages
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer

11.1.1 Responsibilities

Data reduction shall be performed by the laboratories analyzing samples or field personnel responsible for obtaining field measurements. The individual analyst shall verify appropriate forms for completeness and correctness of data acquisition and reduction. The certificate of analysis provided with sample results shall ensure that data reduction has been performed properly and that the reported results are correct. Calculations and results for field measurements shall be independently reviewed. The reviewer shall initial and date the applicable field results reporting forms (Sections 5 and 6 and Appendices J and K).

11.1.2 Data Reduction Procedures

Analysis-specific calculations and statistical methods are dependent on the methods identified in Appendix G.

Raw instrumental data shall be reduced to the final data package and certificate of analysis when required in accordance with the following steps.

1. Generate data for a particular sample using a specific analytical instrument. If a sample is tested for several analytes, perform data reduction individually for each analyte unless several analytes can be identified at the same time [e.g., metals by Inductively Coupled Plasma (ICP)].

2. For a particular group of analytes (e.g. metals), gather raw data generated for a particular sample. For example, raw data from ICP, graphite furnace, flame atomic absorption, and cold vapor analyses for a particular sample may be used to generate results sheets for all analytes.

3. Gather results sheets from all sections (metals, wet chemistry, gas chromatography, gas chromatography/mass spectrometry, and radiometrics) and forward them to the laboratory project manager or designee for compilation and generation of certificates of analysis.

Reduction of field data shall be performed as described in the field methods. Data reduction shall be done on data sheets specified for the field method or in the field notebook. Equations and other information required to reduce field data shall be specified in the individual field methods.

Records management shall be in accordance with guidelines in Section 4. Sections 5 and 6 and Appendices J and K provide discussions of reporting and data reduction requirements for field measurements.

11.2 DATA VALIDATION

Data validation is a process performed independently of the laboratory or field personnel generating analytical data. The Data Validation Plan (Appendix D) describes the validation process requirements, responsibilities for performing data validation, and detailed technical requirements for review and qualification (flagging) of the analytical data.

Data will be validated according to the ASL at which it was analyzed. Samples analyzed by ASL A methods will be validated against ASL A method criteria, ASL B data against ASL B method criteria, etc. Data used to calculate upper confidence limits (UCLs) for risk assessment by any new method requires full validation to ASL D criteria until completeness requirements for the initial stage or phase of use have been met. Continued use of the method in generating data for quantitative risk assessment requires a minimum of ten percent of the data to be validated to ASL D.

Procedures are included for validation of field data generated for ASL A, inorganic (non-metals) data, radiological data, organic analyses by gas chromatography and Gas

Chromatography/Mass Spectrometry (GC/MS), and metals analysis by inductively coupled plasma spectroscopy and atomic absorption. Requirements for validation of user-defined ASLs B and E data are mentioned and will be specifically defined in the applicable PSP.

Data qualifiers, or flags, are defined in Appendix D along with the procedures on how they are assigned to the validated data. Data validation criteria are based on the method performance and QC acceptance criteria specified for each method in Appendix G.

Data validation procedures presented in Appendix D are applicable only to data collected under the Sitewide CERCLA Quality Assurance Project Plan (SCQ). Data collected prior to implementation of the SCQ shall be considered historical data and its validation will be handled on a project-specific basis as outlined in subsection 11.4.

11.3 DATA REPORTING

A certificate of analysis and summary sheets shall be generated by the analytical laboratory. The sheets shall contain information about analytical tests performed, date and condition of sample received, results, methodology, and quality of data reported. Field measurements shall be reported on applicable forms specified in Sections 5 and 6 and Appendices J and K.

Electronic data transfer information shall be generated from a certificate of analysis. Data shall be verified for accuracy by a person other than the one responsible for entering the data. The FEMP project manager or designee shall be responsible for checking and approving the final presentation of reported data to ensure that project-specific requirements are met.

11.3.1 ASL A Data Reporting

Field-generated data reports for ASL A shall include field logs and report forms specified in Sections 5 and 6 and chain-of-custody records specified in Section 7.

11.3.2 ASL B Data Reporting

For ASL B analyses, when methods, performance requirements, and deliverable items are specified by the user, the deliverable data package shall be specified in applicable PSPs.

For predefined ASL B analyses, the deliverable data package shall include, as a minimum, reports of the following applicable analysis results.

- Samples and dilutions
- Method blanks
- Laboratory control samples

- Matrix spike/matrix spike duplicate samples
- Laboratory replicate samples
- Surrogate recoveries

11.3.3 ASL C Data Reporting

The deliverable data package for ASL C analyses shall include, but not be limited to, the following items for the analytical methods to which they apply.

- **All laboratory analyses**
  - Analysis results of samples and dilutions
  - Analysis results of laboratory control samples
  - Analysis results of matrix spike/matrix spike duplicate samples
  - Analysis results of method blank samples
  - Analysis results of laboratory replicate samples
  - Injection logs of instruments used
  - Analysis results of initial and continuing calibration samples including calibration curve calculations
  - Internal standards and tracer results
  - Analyst bench notes for inorganic, geotechnical, and radiochemical analyses
- **Organic Analyses**
  - Reports of compounds detected in Gas Chromatography (GC) and Gas Chromatography/Mass Spectrometer (GC/MS) analyses including reported retention times, integrated area counts, and compound identification
  - Library search results to tentatively identify non-target analytes in GC/MS analyses
  - Surrogate recoveries
  - Results of GC/MS tuning samples for instruments used

3. Compare results for samples and QA/QC analyses to protocol and method performance criteria in effect at the time data were generated or to data validation criteria of this SCQ if no such protocols are readily available.

4. Review field records, audit and surveillance reports, and training records for personnel performing sampling and analysis.

5. Assign the data set a level of useability that indicates uses the data are suitable for based on the level of performance achieved and the quality of the supporting data package.

If sufficient supporting QA/QC documentation is not available or if the raw data package is not available, a data set may be assigned a more restrictive level of useability than it was originally intended for, or it may be classified as unuseable.

Validation procedures for historical data shall be included in the PSP, and a summary report of data validation shall be prepared. The report shall discuss validation findings and assigned useability of the historical data.

11.5 REFERENCES

U.S. Department of Energy. 1988a. ***Quality Assurance Project Plan, Remedial Investigation and Feasibility Study, Feed Materials Production Center, Fernald Ohio.*** Prepared by Advanced Sciences, Inc., for DOE Oak Ridge Operations. March 1988.

U.S. Department of Energy. 1988b. ***Data Management Plan, Remedial Investigation and Feasibility Study, Feed Materials Production Center, Fernald Ohio.*** Prepared by Advanced Sciences, Inc., for DOE Oak Ridge Operations. March 1988.

U.S. Environmental Protection Agency. 1986. ***Test Methods for Evaluating Solid Waste.*** SW-846, third edition.

U.S. Environmental Protection Agency. 1990. ***Users Guide to Contract Laboratory Program.*** EPA/540/P-91/002.

12.4.3 Quality Requirements

Laboratories shall have an acceptable quality assurance plan that is in accordance with the requirements of the SCQ (paragraph 12.4.6) and shall be audited prior to receiving FEMP samples as follows.

12.4.3.1 **Administrative.** The following administrative items shall be addressed during audits.

- Documentation of laboratory organizational hierarchy
- QA program
- Assignment of responsibility for establishing, maintaining, and verifying an appropriate QA program
- Facility design for applicable analytical work meeting EPA requirements as applicable
- A training and certification program for laboratory analysts
- Tracking system for documents, equipment, parts, and supplies
- Use of current, controlled copies of operating procedures
- Use of current labeled and dated standards
- Internal chain-of-custody process meeting requirements of Section 7
- Procedures and records for equipment calibration, maintenance, and evaluation
- Facilities for receiving, checking, and storing samples prior to analysis and a routine that ensures compliance with preservation requirements
- Tracking system for samples that ensures holding-time requirements are met
- Process for documenting, reporting, and recording nonconforming items or actions, including corrective actions
- Process for storage that ensures record security including a records tracking system
- System for scheduling and documenting internal audits of the analysis system and its components using checklists and reports and a means of addressing audit findings in a timely manner

- Laboratory copies of the SCQ are properly controlled and up-dated

12.4.3.2 Technical. The following technical items shall be addressed during audits.

- Analyses are performed in accordance with written procedural requirements, including calibration and use of proper standards, blanks, and other QC checks
- Demonstration that technical expertise and equipment meet FEMP methods requirements
- Verification and reporting of analytical results as required

12.4.4 Performance Evaluation Samples

Laboratories shall provide documentation of successful analyses of performance evaluation samples prior to approval for FEMP sample analyses.

Laboratories that perform ASL D analyses shall document successful analyses of the EPA Contract Laboratory Program performance evaluation samples, or equivalent, covering the four previous quarters.

For analyses at other ASLs, performance evaluation samples supplied by FEMP or the EPA Contract Laboratory Program shall be successfully analyzed and documented using methods identified in Appendix G.

12.4.5 Continuing Satisfactory Performance

Implementation of quality requirements shall be continually verified through on-site audits conducted by FEMP annually as a minimum (See Appendix E).

Laboratory performance shall be evaluated through data validation (Appendix D) and performance evaluation sample analysis.

12.4.6 Quality Assurance Plan

Analytical laboratories shall be required to have a written internal QA plan and applicable standard operating procedures in place that include the following items. Adherence to the elements of the plan shall be documented in audits.

- Laboratory management structure including individual responsibilities
- Documentation of laboratory personnel qualifications

Section 14

SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

14.1 FIELD DATA

Field data shall be assessed by the data user for accuracy, precision, and completeness taking into account overall project objectives, background data points, and field Quality Assurance (QA) samples as defined in Section 4. Requirements for field documentation are included in Section 5, 6, and 7. If additional requirements are required for a specific project, they shall be defined in Project-Specific Plans (PSP).

14.2 ANALYTICAL LABORATORY

Analysts, in consultation with the laboratory project manager or designee, are responsible for evaluating recoveries of surrogates and matrix spikes and ensuring precision of duplicates. Quality Control (QC) acceptance criteria for recoveries and relative percent difference are included in the applicable method in Appendix G.

Those recoveries and/or Relative Percent Differences (RPD) that are found to be "out-of-control" according to QC acceptance criteria shall be evaluated using all information pertinent to the recoveries/RPDs in question. Pertinent information includes, but is not limited to, preparation blanks, laboratory control samples, any matrix interferences present, concentration of the spiking compound present in the original sample, homogeneity of the sample, and the matrix of the sample.

Assessment of data precision and accuracy is an integral part of the laboratory data verification process.

After data have been generated by an analyst or instrument, they shall be submitted to a qualified peer (another analyst, group supervisor or equivalent) for review. This initial review is for transcription errors, calculation errors, holding times, and a check for completeness, which shall include the following elements.

- Required samples and analyses have been processed
- Complete records exist for each analyte and associated QC samples
- Specified procedures have been implemented

- Electronic data packages have been checked for completeness

A secondary review is conducted by the laboratory group supervisor or equivalent, laboratory project manager, or laboratory quality control personnel or equivalent.

A tertiary review is a QA function that is performed on a minimum of five percent of analytical data. This QA review includes technical and editorial QA reviews. All data shall be reviewed by laboratory project manager or designee for accuracy, precision, and completeness prior to transmittal to the data requestor.

14.3 PRECISION

To determine precision of the method, a routine program of duplicate analyses shall be performed (Section 4). The results of the duplicate analyses are used to calculate the RPD, which is the governing QC parameter for precision.

$$\text{RPD } \% = 100 * \frac{(D_1 - D_2)}{(D_1 + D_2) / 2}$$

Where:

D_1 = the larger of the two observed values

D_2 = the smaller of the two observed values

14.4 ACCURACY

Accuracy shall be estimated based on results of laboratory control sample (LCS) analyses or matrix spike recoveries (Section 4). Accuracy is expressed in terms of percent recovery as expressed in the following formulas.

For LCS

$$\text{Percent Recovery} = 100 * \left(\frac{\text{measured value}}{\text{true value}}\right)$$

For matrix spikes

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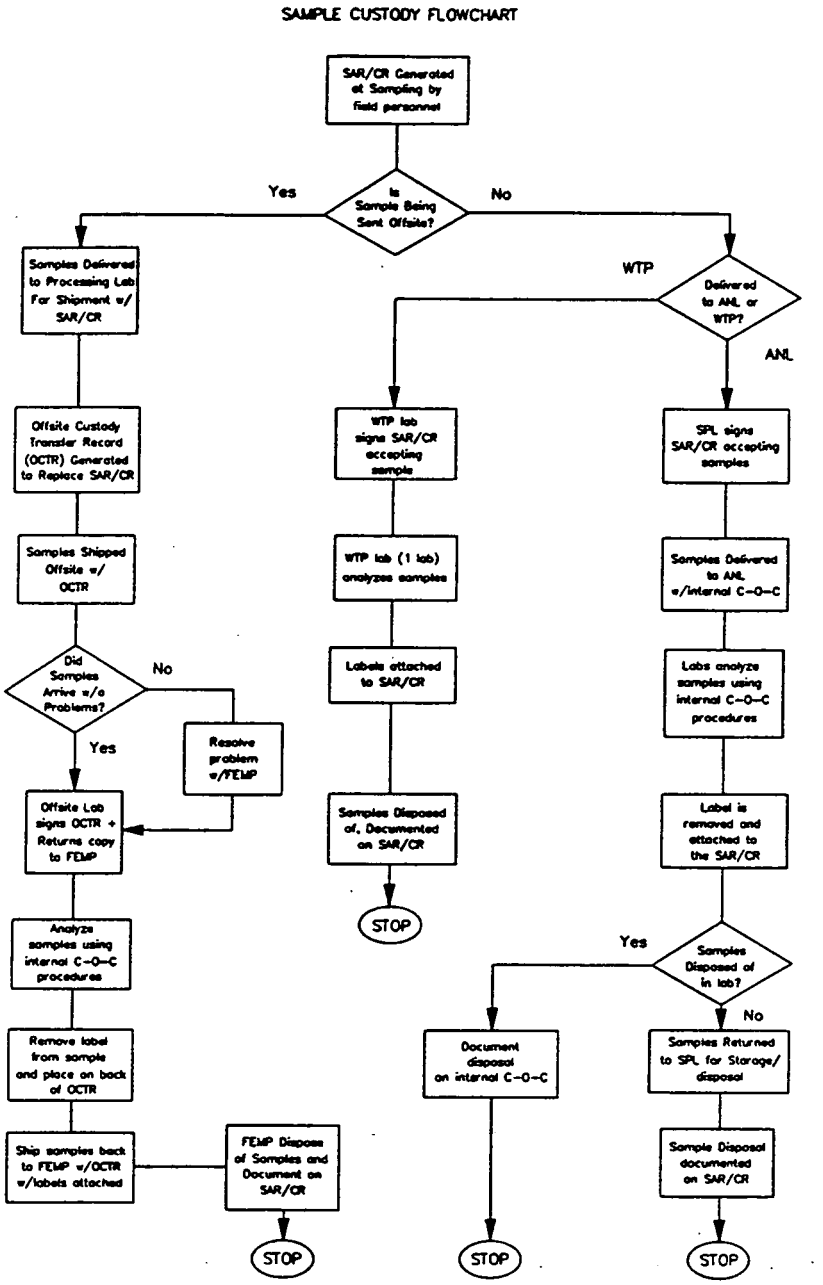
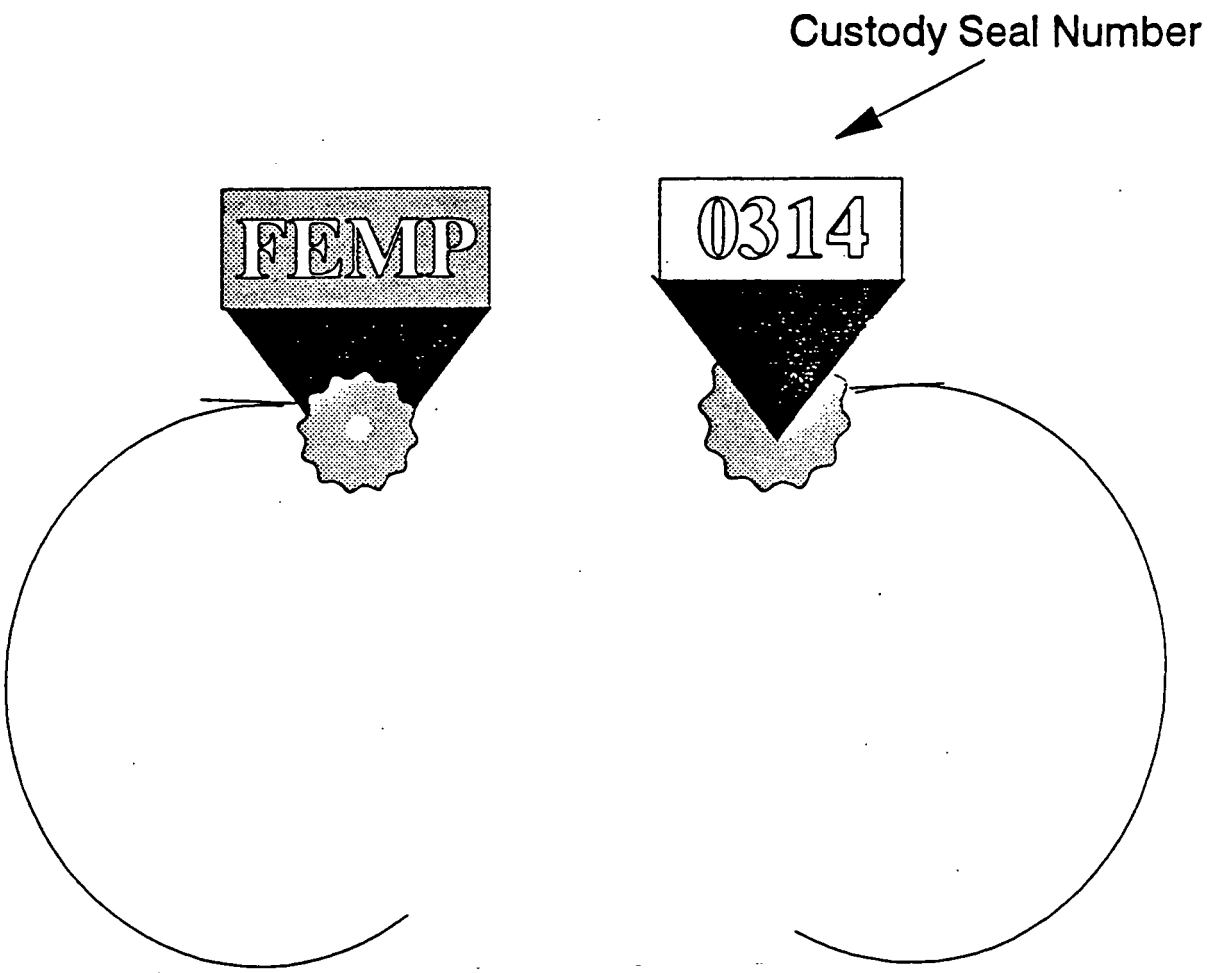


Figure 7-1. Sample Custody Flow Chart

LAB SAMPLE DATE _____

DO NOT TAMPER INITIALS _____

Figure 7-2. Example Custody Tape



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Wire Custody Seal Attachment

1. Wind the wire end of the custody seal around and/or through the locking ring and bolt or the latch of the container being sealed.

2. Slide the end of the wire through the seal portion of the custody seal till the wire is bound tightly around the container.

3. Crimp the seal to lock the wire in place.

NOTE

There may be several varations of how the seal is attached. However, the seal must be attached in a way so it will be broken or destroyed when the container is opened.

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Table 2-2 INTERNAL QUALITY CONTROL REQUIREMENTS

	ANALYTICAL SUPPORT LEVELS	
	B	C & D
Inorganic Analytical QC Samples		
Laboratory control samples	1 per analytical batch if applicable	1 per analytical batch if applicable
Method blanks	1 per analytical batch of samples	1 per analytical batch of samples
Matrix spikes	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method
Laboratory replicate samples	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method
Interference check	A/S	1 per analytical batch per matrix
Dilution check	A/S	1 per analytical batch per matrix
Initial Calibration	As required by method	A/S
Continuing Calibration	As required by method	A/S

Table 2-2 INTERNAL QUALITY CONTROL REQUIREMENTS (cont.)

	ANALYTICAL SUPPORT LEVELS	
	B	C & D
Organic Analytical QC Samples		
Method (reagent) blanks	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method
Matrix spikes	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method
Matrix spike duplicates	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method	1 per 20 samples or 1 per analytical batch, whichever is more frequent (per matrix) if applicable to the method
Surrogates	Present in every determination	Present in every determination
DFTPP and BFB performance results	Daily	Once every 12 hours
Internal standard (GC/MS)	A/S	In every determination

Table 2-2 INTERNAL QUALITY CONTROL REQUIREMENTS (cont.)

	ANALYTICAL SUPPORT LEVELS	
	B	C & D
Organic Analytical QC Samples - (cont.)		
Performance evaluation standard (pesticides/PCBs)	A/S	1 per 10 samples
Initial calibration	A/S	A/S
Continuing calibration	A/S	A/S
Second column confirmation (GC analyses)	A/S	For all positive hits
Review of compound identification for target analytes	A/S	For all positive hits by GC/MS methods
Review tentatively identified compounds	A/S	For GC/MS methods
Radiochemical Analytical QC Samples[1]		
Reagent Blank	1/20 or 1/batch whichever is more frequent	1/20 or 1/batch whichever is more frequent
Laboratory control samples	1/20 or 1/batch whichever is more frequent	1/20 or 1/batch whichever is more frequent
Duplicate	N/A	1/20 or 1/batch per matrix whichever is more frequent
Matrix Spike	Required only when a tracer or carrier is not used	1/20 or 1/batch whichever is more frequent

Table 2-2 INTERNAL QUALITY CONTROL REQUIREMENTS (cont.)

	ANALYTICAL SUPPORT LEVELS	
	B	C & D
Radiochemical Analytical QC Samples - (cont.)		
MSD (^{99}Tc only)	N/A	1/20 or 1/batch whichever is more frequent
^{99}Tc Recovery determination	Use matrix spike	Use average of matrix spike & matrix spike dup.
Tracer or carrier (N/A for ^{137}Cs, ^{99}Tc, Gross Alpha, Gross Beta, and Total U by Laser)	each sample	each sample

Notes

A/S	-	As specified in method or project specific plan
N/A	-	Not applicable
(1)	-	Gross Alpha and Gross Beta are applicable to ASL B only

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Table 2-4 FIELD QUALITY CONTROL SAMPLES

	ANALYTICAL SUPPORT LEVELS		
	B	C	D
INORGANICS, ORGANICS, and RADIONUCLIDES			
Field QC Samples			
Field blanks	A/S	1 per 20 or 1 per sampling event, whichever is more frequent	1 per 20 or 1 per sampling event, whichever is more frequent
Equipment rinsate blanks	A/S	1 per 20 or 1 per sampling event, whichever is more frequent	1 per 20 or 1 per sampling event, whichever is more frequent
Field duplicates	A/S	1 per 20 or 1 per sampling event, whichever is more frequent	1 per 20 or 1 per sampling event, whichever is more, frequent
Preservative blanks	A/S	A/S	A/S
Container blanks	A/S	1 per QC lot of containers	1 per QC lot of containers
Laboratory control samples	1 per analytical batch if applicable	1 per analytical batch if applicable	1 per analytical batch if applicable
Method blanks	1 per analytical batch of samples	1 per analytical batch of samples	1 per analytical batch of samples
Trip blanks (VOAs only)	A/S	1 per shipping container	1 per shipping container

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Table 6-1 SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS (cont.)

Parameter	Container	Preservative	Holding Time	Permissible Sample Type
Liquid - Low to Medium Concentration Samples (cont.)				
Mercury	1-liter polyethylene with polyehtylene-lined closure	50% nitric[3] acid pH<2	28 days	G or C
Metals, dissolved[C] except Hg	1-liter polyethylene with polyethylene lined closure	Filter-on-site[3] 50% nitric acid pH <2	6 months[8]	G
Mercury	1-liter polyethylene with polyethylene-lined	Filter-on-site[3] 50% nitric acid pH <2	28 days	G
Oil and grease[C]	1-liter widemouth glass with Teflon liner	50% sulfuric[3] acid pH <2 Cool[6]	28 days	G
Semi volatile compounds,[C] organochlorine chlorine pesticides PCBs, herbicides, organo-phosphate pesticides in water				
No residual chlorine[C] present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon liner	Cool[6]	7/40 days[1]	G or C
Residual chlorine[C] present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon liner	Add 3 mL 10% sodium thio-sulfate per gallon Cool[6]	7/40 days[1]	G or C
Volatile organic compounds			14 days	
No residual chlorine[C] present	2 40-mL vials with Teflon lined septum caps	4 drops conc. hydrochloric acid Cool[6]	14 days	G

Table 6-1 SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS (cont.)

Parameter	Container	Preservative	Holding Time	Permissible Sample Type
Liquid - Low to Medium Concentration Samples (cont.)				
No residual chlorine[C] present	2 40-mL vials with Teflon lined septum caps	Cool[6]	7 days	G
Residual chlorine[C] present - drinking water	2 40-mL vials with Teflon lined septum caps	See Note 4	7 days	G
Organic halogens,[E] purgeable (POX)	2 40-mL vials with Teflon lined septum caps	Cool[6]	14 days	G
Organic halogens,[E] total (TOX)	250-mL amber glass with Teflon-lined septum closure	Cool[6]	28 days	G
Total Organic Carbon (TOC)	250 mL amber glass with Teflon lined septum closure	Cool[6]	28 days	G
pH[C]	In-situ (beaker or) bucket	None	Immediate (in field)	G
Total Phenols[C]	1-liter amber glass with Teflon-lined closure	50% sulfuric acid pH <4 Cool[6]	28 days	G
Phosphate-ortho[C]	500-mL or 1-liter polyethylene with polethylene or polyethylene-lined closure	Filter-on-site Cool[6]	48 hours	G
Phosphorus, total[C] dissolved	500-mL or 1-liter polyethylene with polyethylene or polyethylene-lined closure	Filter-on-site 50% sulfuric acid pH <2 Cool[6]	28 days	G
Solids, settleable[C]	1/2-gal. polyethylene with polyethylene closure	Cool[6]	7 days	G or C
Solids (total and[C] suspended, etc.)	500-mL or 1-liter poly-[2] ethylene with polyethylene or polyethylene lined closure	Cool[6]	7 days	G or C

Notes (cont)

6. Cool to the range of 2° to 6° C.

7. Radiochemical holding times are 6 months or 5 half-lives, whichever is shorter.

8. Holding time for Mercury analyses is 28 days.

Table 6-1 SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS (cont.)

Abbreviations

G	-	Grab
C	-	Composite
VOA	-	Volatile organic analysis
VOC	-	Volatile organic compound

References

A U.S. EPA Region IV, Environmental Services Division, ***Analytical Support Branch, Operations and Quality Control Manual***, June 1, 1985, or latest version.

B EPA Method 1310, "Extraction Procedures", 1982, SW 846, U.S. EPA, Office of Solid Wastes, Washington, D.C.

C 40 CFR Part 136, Federal Register, Vol, 49, No. 209, October 26, 1984.

D U.S. EPA Region IV, Environmental Services Division, ***Ecological Support Branch, Standard Operating Procedures Manual***, latest version.

E EPA Interim Method 450.1, "Total Organic Halide" U.S. EPA, ORD, EMSL, Physical and Chemical Method Branch, Cincinnati, Ohio, November, 1980.

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SAMPLE ID: XXXXXXXXXX

(barcode goes here)

XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
SAMPLE ID : XXXXXXXXXX
(barcode goes here)

DATE SAMPLED TIME SAMPLED

MATERIAL NAME / SAMPLE TYPE
XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
PRESERVATIVES
XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
CONTAINER TYPE GROSS WEIGHT
XXXXXXXXXXXXXXXXX

COLLECTORS INITLS TARE WEIGHT

CNT:

COC #:

SAMPLE ID: XXXXXXXXXX

(barcode goes here)

Label A

TEMP SAMPLE ID X-REF SAMPLE ID

DATE SAMPLED TIME SAMPLED

MATL NAME

SAMPLE TYPE

CONTAINER TYPE GROSS WEIGHT
XXXXXXXXXXXXXXXXX

COLLECTORS INITLS TARE WEIGHT

PRESERV:

CNT:

COC #:

Label B

Form 7-2. Example Sample Label

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Section 1C

DQO No. - Enter the number assigned to this particular DQO. It will be assigned by the FEMP sampling and analysis management coordinator of the prime operating contractor.

DQO Reference No. - Identify all other DQOs that are related to the DQO being prepared.

SECTION 2

Media Characterization - Specify the medium being investigated. Only one medium will be considered per DQO. If other media are sampled to support a work phase, a separate DQO for each medium and sampling activity shall be prepared. If **OTHER** is used, include an explanation.

SECTION 3

Data Use with Analytical Support Level (A-E) - Identify data use by activity and the Analytical Support Levels (ASL)s specified for generation of data. (ASLs are described in SCQ Section 2.) More than one activity or ASL may be indicated because an activity may be required to generate data from field measurements to laboratory analysis. Each discrete task requires a separate evaluation of its respective ASL. If **other** is used, include an explanation.

Explanations of terminology

- **Site Characterization** - Determination of the level, extent and location of contamination
- **Risk Assessment** - Endangerment assessment or public health evaluation
- **Evaluation of Alternatives** - Evaluation or screening of remedial alternatives
- **Engineering Design** - Detailed engineering design of remedial actions for the site
- **Monitoring** - During remediation activities, monitoring remedy implementation or establishing baseline conditions for long term monitoring after site remediation

Form C-1. DQO Summary Form (sheet 5 of 8)

SECTION 4

Section 4A

Regulatory Drivers - Identify regulatory drivers and Applicable or Relevant and Appropriate Requirements (ARAR) associated with the task.

Section 4B

Objective - Provide a clear, concise statement of the reason for the sampling activity (e.g., 1991 amended Consent Agreement requirement, RCRA monitoring, waste characterization). Include imminent health risks associated with sampling effort.

SECTION 5

Site Information - Identify information required to gain an overview of the site and the relative complexity and extent of data requirements. Briefly describe the physical setting, dimensions, and current use of the site.

SECTION 6

Section 6A

Data Types, Analytical Support Level, Equipment Selection, and SCQ Reference - Specify data requirements for establishing the type, degree, extent, and migration characteristics of the contaminants and the required site characteristics.

Explanation

- **Analytical Parameters 1 - 6** - Describe the necessary analysis to acquire data necessary to satisfy task requirements by data quality level and analysis activity. (Full radiological includes uranium.)

  The list of analytes and **other** category are completed according to data requirements.

Section 6B

Equipment Selection and SCQ Reference (ASLs A through E with SCQ) - Identify equipment required to analyze sample parameters and corresponding reference to that equipment by analytical method in Appendix G.

Form C-1. DQO Summary Form (sheet 6 of 8)

- Drinking Water Data Validation Procedures for ASL B (subsection D.10)
  - Internal Standard (paragraph D.10.1)
  - Surrogate Analyte (paragraph D.10.2)
  - Laboratory Duplicates (paragraph D.10.3)
  - Field Duplicates (paragraph D.10.4)
  - Laboratory Reagent Blanks (paragraph D.10.5)
  - Field Reagent Blanks (paragraph D.10.6)
  - Laboratory Performance Check Solution (paragraph D.10.7)
  - Matrix Spike/Matrix Spike Duplicates (paragraph D.10.8)
  - Calibration Standards (paragraph D.10.9)
  - Compound Quantitation and Reported Detection Limits (paragraph D.10.10)
  - Overall Assessment of Data for a Case (paragraph D.10.11)
- Data Validation Procedure for Radiological Analyses (subsection D.11)
  - Completeness Checks (paragraph D.11.1)
  - Calibration (paragraph D.11.2)
  - Blanks (paragraph D.11.3)
  - Detection Limits and Sample Results (paragraph D.11.4)
  - Radiometric and Gravimetric Yields (paragraph D.11.5)
  - Duplicate Samples and Analyses (paragraph D.11.6)
  - Laboratory Control Samples (paragraph D.11.7)
  - Holding Times (paragraph D.11.8)
  - Alpha-Emitting Ra Isotopes Using Scintillation Counting (paragraph D.11.9)

- Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting (paragraph D.11.10)
- Supplemental Requirements for Fluorometric Analysis of Uranium (paragraph D.11.11)
- Other Quality Control (paragraph D.11.12)

- Inorganic (Non-metals) Data Validation Procedures (subsection D.12)
  - Holding Times (paragraph D.12.1)
  - Calibration (paragraph D.12.2)
  - Blanks (paragraph D.12.3)
  - Laboratory Control Samples (paragraph D.12.4)
  - Duplicate Sample Analyses (paragraph D.12.5)
  - Matrix Spike Sample Analysis (paragraph D.12.6)
  - Sample Result Verification (paragraph D.12.7)
  - Field Duplicates (paragraph D.12.8)
  - Overall Assessment of Data for a Case (paragraph D.12.9)

D.2 TECHNICAL APPROACH

The following technical approach shall be applied to ensure that data validation activities are cost-effective and technically sound.

D.2.1 Data Validation Procedures

Procedures herein meet technical, regulatory, and Quality Assurance (QA) requirements and guidance of the documents listed in D.12.

D.2.2 Checklists

Checklists are provided for reviewing data. Checklists may either be on hardcopy or automated. When possible, data validation shall be conducted electronically.

Data validation checklists were developed based on the following documents.

- FEMP DQOs (Appendix C)
- ***Laboratory Data Validation Functional Guidelines for Evaluating Organic and Inorganic Analysis***, U.S. Environmental Protection Agency, 1988a and 1988b
- Radiochemical and other QA acceptance criteria for other methods

Data review checklists contain questions and specific guidance on information to be provided for each major measurement parameter. Completion of the checklist will reveal missing data, anomalous data or lack of criteria compliance that may threaten data integrity.

D.2.2.1 **Field Checklist Development.** Validity of the sample collection process is as important as sample analysis. The field data package shall be available for validation and shall include, but not be limited to, the following as applicable.

- Soil boring logs or drillers logs
- Water sampling logs
- Soil/sediment sampling logs
- Custody records
- Field instrument calibration records
- Shipping records
- Field logs or daily log forms
- Health and safety logs
- Data sheets for temperature correction adjustments (e.g., pH and conduction with measurements)

These items are portions of the daily log specified in Sections 5 and 6 (field activities and sampling procedures).

D.2.2.2 Laboratory Checklist Development. Checklists for validating chemical analyses shall be directly traceable to appropriate requirements and industry standards [e.g., American National Standards Institute (ANSI), American Society for Testing and Materials (ASTM), American Society of Mechanical Engineers (ASME), EPA]. Laboratory data validation criteria are determined by analytical methods and ASLs specified for the data. Checklists shall include, but not be limited to, the following criteria.

- **Organic Materials**
  - Holding times
  - Gas chromatograph/spectrometer tuning
  - Calibration
  - Blanks
  - Percent Surrogate recovery
  - Matrix Spike/Matrix Spike Duplicates (MS/MSD)
  - Field duplicates
  - Internal standards
  - Mass spectra/chromatograms
- **Inorganic Materials**
  - Holding times
  - Calibration
  - Blanks
  - Field duplicates
  - MS/MSD
  - AA
  - Serial dilution analysis

W Post-digestion spike for furnace AA analysis is out of control limits (85 to 115 percent), while sample absorbance is less than 50 percent of spike absorbance.

* Duplicate analysis was not within control limits.

+ Correlation coefficient for the MSA was less than 0.995.

No combination of "S", "W", or "+" shall appear in the same field for an analyte. Use of these qualifiers is mutually exclusive.

D.2.5 Sequence of Data Validation Activities

Validation activities for field sample collection and laboratory analysis data shall be accomplished in the following sequence. Subsection D.5 provides complete field data validation procedures. Subsections D.6 through D.12 provide complete laboratory analysis data validation procedures.

D.2.5.1 **Field Data Validation.** Validation of field activities, including results of field analytical methods for ASL A, shall be performed in the following sequence.

1. Prior to beginning data validation, ensure that the custody record and daily log (defined in Sections 5 and 7) are available as specified in paragraph D.5.2. Sample numbers on the custody record should be compared to the two part sample label to make sure the numbers are identical.

2. Review completed sampling data in the field log and associated documentation to ensure that forms specified in PSP and the SCQ have been completed and that required instrument calibration documentation exists.

3. If checklist requirements are not met, initiate the discrepancy/deficiency resolution process specified in paragraph D.2.6.

4. Report data validation results to the FEMP project manager by hardcopy or electronic data transfer. Provide a hardcopy summary that lists sample numbers, flag discrepant/deficient data samples, and include copies of data validation Data Deficiency Reports (DDR) without review checklists.

5. Retain copies of completed review checklists in Data Validation Team (DVT) files. Place original field data forms and records in FEMP files.

D.2.5.2 **Laboratory Analysis Validation.** Laboratory analysis data validation activities shall be performed in the following sequence.

1. Obtain original, completed laboratory certificate-of-analysis data packages from the FEMP project manager.

2. Review data sets using laboratory analysis review instructions, procedures, and checklists.

3. Check that sample numbers on custody record matches those reported on the laboratory data package.

4. If laboratory analyses or results do not meet review requirements, see applicable ASL procedure.

5. Report laboratory data review results to the FEMP project manager. List sample numbers, flag discrepant, deficient, or questionable samples and include copies of data validation DDRs without review checklists.

6. Retain copies of completed review checklists in DVT files. Replace original laboratory documentation in FEMP files.

D.2.6 Discrepancy/Deficiency Resolution

The data validation process may raise questions as to useability of some data because of failure to comply with one or more of the following data quality requirements.

- PSP
- Sample collection/tracking procedures
- Holding times
- Field and analytical instrument calibration requirements
- Quality control procedures
- Compliance with method procedures or requirements (Appendix G)
- Laboratory contamination evaluation

If so, the following formal evaluation process shall be conducted to resolve status of questionable data.

- Data package items shall be identified so that the sample number can be correctly associated with all parts of the reported data package.

- The independent validator shall review the data package for the following.

  - Appropriateness of equations

  - Correctness of numerical output, including correct united and consistent rounding of numerical values

  - Numerical correctness of calculations (by repeating computations)

  - Correct interpretation of strip charts

  - Appropriate detection limits

- The validator shall submit checklists and reports to the designated FEMP QA organization representative.

Entries and calculations reviewed by validator shall be marked in ink with a check mark. If validator disagrees with any part of the computations, a single line shall be drawn through the number, a revised number placed above or beside it, and the change initialed and dated.

If data have been processed by computer, validator shall check input only. Agreement shall be indicated by a check mark on each line. If validator disagrees with input, the number shall be lined through, the correction entered above or beside it, and the change initialed and dated. At least one percent of raw data that were automatically acquired and processed shall be manually checked. These data do not require validation beyond that previously discussed. The validator shall sign and date in ink the corrected original pages of the data package.

D.4.2 Data Validation Report Requirements

Data validation reports shall be prepared to comply with the following requirements.

- Data shall be presented in a tabular format whenever possible.

- Each table shall be identified with project number and name and date of report issue.
- Tables shall include the following.

  - Sample IDentification (ID) number used by laboratory and sample identification provided to laboratory if different than laboratory ID

  - Analyte parameters, reported values, and units of measurement reported with consistent significant figures for samples

- Detection limit of analytical procedure if reported value is less than quantitation limit
- Analytical method used (Appendix G)
- Results of QC sample analyses including calibration standards
- Achieved accuracy, precision, and completeness of data when appropriate
- Footnotes to specific data if required to explain reported values
- Data qualifiers

D.4.3 Review of Data Validation Reports

Review of data validation reports is required to verify that validated results correspond to processed analytical results reported by laboratory. This review is performed on data as it is presented for issuance in the report.

After the draft data report is prepared, results shall be checked against validated data to ensure against transcription errors. The checking procedure is performed by a DVT team member as follows.

1. Using draft report, check data entries to ensure that items cited in paragraph D.4.2 are complete and correct.
2. Place a check mark in ink beside correct entries on draft report.
3. Draw a single line through entries needing correction and write correct entry beside it in ink. Initial and date each correction. Do not erase or use white-out.
4. Indicate that corrections were made in final report by placing a check mark by correction after comparing change with revised copy.
5. Sign and date in ink every page of draft data report used to verify corrections.
6. Maintain draft data reports used to verify corrections on file as a record to prove that review occurred.

NOTE

Step 7 is not intended to verify reported data; rather, it is intended to determine that the report meets project requirements.

- Surface-water and sediment sampling (Form D-11, Appendix B)
- Radiation measurement (node survey) (Form D-12, Appendix B)
- Radiation measurement (walkover survey) (Form D-13, Appendix B)

D.5.1.3 ASL B Data Validation. For user-defined ASL B analyses, the QA/QC sample type and frequency, method performance criteria, and data package deliverables shall be specified in applicable PSPs. The PSP shall also specify data validation requirements and evaluation criteria. Validation may range from minimal checks of method performance to a level similar to ASL C or D validation. Intended use of the data shall determine the nature and level of data validation. Checklists (Forms D-14 through D-17, Appendix B) indicate the types of information that may be required for validation of user-defined ASL B data.

Laboratory QC sample type and frequency for specified ASL B analysis shall be specified in Appendix G. Data package deliverable items for specified ASL B analyses are discussed in Section 11. Analytical results and QC sample performance that is within QC acceptance criteria specified in the Appendix G method shall be reported as received with no flags or qualifiers added. Reported QC sample results that are outside the method-specified acceptance criteria shall be reviewed and qualified unuseable (R), (J), (UJ), or other, as applicable. Qualification shall be based on intended use of the analytical data and the reviewer's judgement.

D.5.2 Procedures for Data Validation

The following procedures have been established to implement the DVP.

D.5.2.1 Preliminary Procedures. The following steps shall be performed to initiate the validation review process.

1. Obtain the field daily log and associated documentation, master sample list, SCQ, and applicable validation checklist. Only one checklist is required for the data package.
2. Complete header information at top of checklist.
3. Consult PSP to determine required ASL.
4. Using custody records, list collected samples in column 1 of master sample list form.
5. Review water or soil sampling log for each sample and verify that each log is completed in ink and signed and dated.

D.5.2.2 Checklist Review. Use checklist to determine if data meet the following criteria.

1. Verify that checklist information is included in each sampling log and check off each checklist item if it is correctly entered in the log.

2. If an error is detected, line through incorrect entry, write correct information beside it in ink, and initial and date correction. Do not erase or use white-out.
3. If information is omitted or a log is missing, obtain information from sampling team, verify its accuracy by signature of the team leader, and incorporate it as follows.

   a. Correct omissions by filling in required information. Initial and date correction.

   b. Replace missing logs by filling out a form indicating that the record was created "after the fact."

4. If errors are identified on sampling logs that cannot be corrected, record this fact in comments section of checklist.

5. If data were developed from samples collected without acceptable documentation or from samples that were not collected in accordance with the SCQ, classify the data as either "unuseable" or "qualitative" as applicable.

6. If no documentation is available to assess sample collection, classify data as unuseable. Data may be classified as "qualitative" if limited documentation exists and it is possible to demonstrate that samples were collected in a manner consistent with SCQ requirements.

D.5.2.3 Organizing and Binding Field Records. Field records shall be organized and bound as follows.

1. Attach sampling logs to back of their respective custody records and arrange in order according to sample collection dates.

2. Assign a number (1, 2, 3, etc.) to each record starting with the one with the earliest sample collection date.

3. Include a table of contents for each volume (may be a copy of the master sample list).

4. In column 2 of master sample list (step 4, paragraph 5.2.1), indicate, next to applicable sample ID, the assigned number (step 2) of each sample.

3. If holding times are exceeded, either on the first analysis or upon re-analysis, use professional judgement to determine reliability of data and effects of additional storage on sample results.

D.6.2 Gas Chromatography/Mass Spectroscopy Tuning

Tuning and performance criteria for Gas Chromatography/Mass Spectroscopy (GC/MS) are established to ensure mass resolution, identification, and, to some degree, sensitivity. The criteria are from the EPA Contract Laboratory Program (CLP) Statement of Work (U.S. Environmental Protection Agency, 1990 and SW-846, Sept. 1986) and are subject to review and change. The most recent set of criteria shall be used by the laboratories if the listed criteria are superceded. Appendix G method modifications shall be submitted to DOE if this occurs.

D.6.2.1 Criteria. Criteria are not sample-specific; conformance is determined using standard materials; therefore, criteria shall be met in all circumstances.

- **DecaFluoroTriPhenylPhosphine (DFTPP) for Semivolatile Organic Compounds**

m/z	Ion Abundance Criteria	
	ASL Level B	ASL Levels C and D
51	30.0 - 60.0% of m/z 198	30.0 - 80.0% of m/z 198
68	less than 2.0% of m/z 69	Same
69	present (record % relative abundance)	Same
70	less than 2.0% of m/z 69	Present
127	40.0 - 60.0% of m/z 198	25.0 - 60.0% of m/z 198
197	less than 1.0% of m/z 198	Same
198	base peak, 100% relative abundance	Same
199	5.0 - 9.0% of m/z 198	Same
275	10.0 - 30.0% of m/z 198	Same
365	greater than 1.0% of m/z 198	greater than 1.0% of m/z 198
441	present, but less than m/z 443	Same
442	> 40.0 of m/z 198	40.0 - 110.0% of m/z 198
443	17.0 - 23.0% of m/z 442	15.0 - 24.0% of m/z 442

- **BromoFluoroBenzene (BFB) for Volatile Organic Compounds**

m/z	Ion Abundance Criteria	
50	15.0 - 40.0% of m/z 95	8.0 - 40.0% of m/z 95
75	30.0 - 60.0% of m/z 95	30.0 - 66.0% of m/z 95
95	base peak, 100% relative abundance	Same
96	5.0 - 9.0% of m/z 95	Same
173	less than 2.0% of m/z 174	Same
174	> 50.0 of m/z 95	50.0 - 120.0% of m/z 95
175	5.0 - 9.0% of m/z 174	4.0 - 9.0% of m/z 174
176	95.0 - 101.0% of m/z 174	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176	Same

D.6.2.2 Procedure for ASL D. The following procedure applies to ASL D data.

1. Verify from raw data that mass calibration is correct.

2. Compare data presented for each tuning to each mass listing submitted.

3. Ensure the following.

   a. Forms are available for each 12-hour period that samples are analyzed as specified in the Laboratory Services Contract (LSC)

   b. Laboratory made no transcription errors

   c. Appropriate number of significant figures has been reported (number of significant figures given for each ion in ion abundance criteria column).

   d. Laboratory made no calculation errors (e.g., percent mass of m/z 443 relative to mass of m/z 442 was calculated using the following equation)

$$\text{percent abundance} = \frac{\text{relative abundance of m/z 443}}{\text{relative abundance of m/z 442}} \times 100\%$$

4. If possible, verify that spectra were generated using appropriate background subtraction techniques. DFTPP and BFB spectra are obtained from chromatographic peaks that should be free from co-elution problems, so background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting contract specifications are contrary to QA objectives and are unacceptable.

5. If mass calibration is in error, classify associated data as unuseable (R).

D.6.2.3 Procedure for ASLs C and D. The following procedure applies to ASLs C and D data and shall be performed if tuning acceptance criteria in paragraph D.6.2.2 are not met.

1. If ion abundance criteria are not met and the data in question are needed on a priority basis, use the following guidelines and apply professional judgement to determine extent of data that may be used.

   a. **DFTPP** - The most critical factors in DFTPP criteria are non-instrument-specific requirements that are not unduly affected by location of the spectrum on the chromatographic profile. The m/z 198/199 and 442/443 ratios are based on natural abundance of carbon 12 and carbon 13 and are critical. They shall always be met.

   Similarly, m/z 68, 70, 197, and 441 relative abundance ratios indicate condition

6. For surrogate spike recoveries out of specification, proceed as follows.

   a. If at least two surrogates in a base/neutral or acid fraction or one surrogate in the volatile fraction are out of specification but have recoveries greater than ten percent, proceed as follows.

      (1) Flag positive results for that fraction as estimated (J).

      (2) Flag negative results for that fraction with the sample quantitation limit as estimated (UJ).

   b. If a surrogate in a fraction shows less than ten percent recovery, proceed as follows.

      (1) Flag positive results for that fraction as estimated (J).

      (2) Flag negative results for that fraction as unuseable (R).

   c. Do not qualify data with respect to surrogate recovery unless at least two surrogates are out of specification in the base/neutral or acid fraction, one is out of specification in the volatile fraction, or unless recovery of a surrogate is less than ten percent. If reanalysis was performed results of the original anaylsis and reanalysis should be compared to assess whether the results are due to matrix effects or to a problem with the analytical process.

   d. For a blank analysis with surrogates out of specification, give special consideration to validity of associated sample data and determine whether problems are isolated to the blank alone or if there is a fundamental problem with the analytical process.

NOTE

For example, if one or more samples in the batch show acceptable surrogate recoveries, the blank problem may be considered an isolated occurrence. However, even if this judgement allows some use of the affected data, analytical problems remain, which shall be reported to and corrected by the laboratory.

D.6.6 Matrix Spike/Matrix Spike Duplicates

MS/MSD data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate precision and accuracy of individual samples.

D.6.6.1 Criteria. Spike recoveries and Relative Percent Differences (RPD) between MS/MSD recoveries shall be within advisory limits in the applicable method (Appendix G).

D.6.6.2 Procedure for ASLs C and D Data. The following procedure is applicable to both ASLs C and D data.

1. Inspect data results for MS/MSD recovery.

2. Verify transcriptions from raw data for ASL D and verify calculations.

3. Do not use MS/MSD data alone to qualify an entire case. Use informed professional judgement and MS/MSD results in conjunction with other QC criteria to determine the need for some qualification of the data.

4. Try to determine effect of MS/MSD results on associated data with regard to the MS/MSD sample and specific analytes for samples associated with the MS/MSD.

5. If it can be determined that results of the MS/MSD affect only the spiked sample, limit qualification to this sample alone.

6. If it is determined through MS/MSD results that a laboratory is having a systematic problem in analysis of one or more analytes, apply qualification to associated samples.

D.6.7 Field Duplicates

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision; therefore, results may have more variability than laboratory duplicates, which measure only laboratory performance. It is also expected that solid matrix duplicate results will have a greater variance than water matrices because of difficulty in collecting identical samples.

D.6.7.1 Criteria. There are no specific review criteria for field duplicate analyses comparability.

D.6.7.2 Procedure.

1. Identify samples that are field duplicates using FEMP forms specified in LSC or PSP.

2. Compare results reported for each sample and calculate the RPD.

3. Provide evaluation of field duplicates with reviewer comments.

- Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include the following.
  - High Reconstructed Ion Chromatograph (RIC) background levels or shifts in absolute retention times of ISs
  - Excessive baseline rise at elevated temperature
  - Extraneous peaks
  - Loss of resolution suggested by factors such as non-resolution of 2,4- and 2,5-dinitrotoluene
  - Peak tailing or peak splitting may result in accurate quantitation

Continued analytical activity with degraded performance suggests lack of attention or professional experience. Using instrument performance indicators, data reviewer shall decide if the system has degraded to the point of affecting data quality or validity. If data quality may have been affected, data shall be qualified using reviewer's best professional judgement.

D.6.14 Overall Assessment of Data for a Case

It is appropriate for data reviewer to make professional judgements and express concerns and comments on validity of the overall data package for a case. This is particularly true when there are several QC criteria out of specification.

The additive nature of QC factors out of specification is difficult to assess in an objective manner, but reviewer has a responsibility to inform users concerning data quality and data limitations in order to avoid inappropriate use of data while not precluding consideration of the data. The reviewer is greatly assisted if DQOs are provided.

D.7 PESTICIDES DATA VALIDATION PROCEDURES FOR ASLs C AND D

Data validation procedures for pesticides are based on the *Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis*, 1 July 1988 (U.S. Environmental Protection Agency, 1988b).

This subsection describes general procedures for data validation from Gas Chromatography (GC) analysis of pesticides (e.g., herbicides, purgeable halocarbon, organo-phosphate pesticides) for ASLs C and D. Specific performance criteria, surrogates, spike compounds, instrument performance requirements, calibration, and standards are provided in Appendix G and shall be used as validation criteria. The following procedures shall be performed for GC data validation in the order indicated.

D.7.1 Holding Times

The objective of validating holding time data is to establish validity of analysis results by ensuring that sample holding times from receipt to analysis or preparation were in compliance with the specified method in Appendix G. This procedure applies to both ASLs C and D data.

D.7.1.1 Criteria.

- Extraction of water samples by the separatory funnel methods shall be completed within seven days of sample collection date.
- Extraction of water samples by continuous liquid/liquid extraction shall be started within seven days of sample collection time.
- Extraction of soil or sediment samples by sonication shall be completed within fourteen days of sample collection time.
- Analysis of samples shall be completed within forty days following start of extraction.

D.7.1.2 Procedure.

1. Verify holding time by comparing sample collection date with dates of extraction and analysis on LSC-specified FEMP form.
2. Examine sample records to determine if samples were preserved as specified in the Project-Specific Plan (PSP).
3. If holding times were exceeded, proceed as follows.
   a. Flag positive results as estimated (J).
   b. Flag sample quantitation limits as estimated (UJ).
   c. Document that holding times were exceeded.
4. If holding times are grossly exceeded either on first analysis or re-analysis, proceed as follows.
   a. Use professional judgement to establish reliability of data and effect of additional storage on sample results.
   b. If non-detect data are unuseable, flag data as (R).

D.7.2 Instrument Performance

D.7.2.1 Criteria. Criteria are established to ensure that adequate chromatographic resolution and instrument sensitivity are achieved by the chromatographic system. These criteria are not sample specific. Conformance is determined using standard materials; therefore, criteria shall be met in all circumstances.

D.7.2.2 Procedure for ASL D.

1. Check raw data to verify that the following conditions exist.

   a. Retention-time windows are reported and pesticide standards are within established windows.

   b. Percent breakdown for DDT or endrin does not exceed 20 percent in evaluation standard analyses.

$$\text{\% Breakdown (DDT)} = \frac{\text{Total (DDE + DDD)}}{\text{Total (DDE+DDD+DDT)}} \times 100$$

$$\text{\% Breakdown (Endrin)} = \frac{\text{Total(Endrin ketone + Endrin Aldehyde)}}{\text{Total (Endrin ketone + Endrin Aldehyde + Endrin)}} \times 100$$

   c. Percent breakdown for combined endrin and DDT does not exceed 30 percent in evaluation standard analyses.

   d. If the retention time shift for DiButylChlorendate (DBC) is greater than 0.3 percent for narrow-bore capillary columns or greater than 1.5 percent for wide-bore capillary columns, the analyses may be flagged unuseable (R) for that sample, but the reviewer shall use professional judgement to qualify data.

2. Check affected sample chromatograms for peaks within an expanded window surrounding expected retention-time window of analyte of interest and proceed as follows to ensure that standards fall within retention-time windows. Retention-time windows are used for qualitative identification. If standards do not fall within the windows, associated sample results shall be evaluated. Samples injected after the last in-control standard may be affected.

   a. If no peaks are present within or close to the window of the deviant target compound, there is usually no effect on data. (Non-detected values can be considered valid.)

b. If affected sample chromatograms contain peaks that may be of concern (i.e., above PQL and either close to or within expected retention-time window of analyte), two options (steps c and d) are available to determine affect on data.

c. If no additional effort is warranted, flag positive results and quantitation limits as unuseable (R). In the comments, emphasize the possibility of either false negatives or false positives as appropriate.

d. If additional effort is warranted (e.g., if data are needed on a priority basis and if peaks may represent a level of concern for that particular analyte), proceed as follows to determine a useable window for affected samples.

(1) Examine data package for presence of three or more standards containing analyte of interest that were run within a 24-hour period during which sample was analyzed.

(2) If three or more such standards are present, re-evaluate mean and standard deviation of retention-time window.

(3) If all standards and matrix spikes fall within revised window, determine valid positive or negative sample results using this window.

(4) Record additional efforts taken and resultant impact on data useability.

(5) Include calculations and comparisons generated in support documentation.

D.7.3 Calibration

Calibration requirements ensure that measuring instruments are capable of producing acceptable quantitative data. Initial calibration ensures that instruments are capable of specified performance in the beginning. Continuing calibration ensures that instruments are adjusted at specific time periods and that required calibration documentation is maintained.

D.7.3.1 Initial Calibration Criteria for ASLs C and D Data.

- Retention-time windows are specified for compounds in the applicable method (Appendix G).

- All single component analytes except surrogates shall have a %RSD less than or equal to 20.0 percent except that up to two single column analytes per column may fall between 20.0 and 30.0%.

- Surrogates shall have a %RSD less than or equal to 30.0 percent.

NOTE

Blank analyses may not involve the same weights, volumes, or dilution factors as associated samples. These factors shall be taken into consideration when applying the five-times criteria so that a comparison of the total amount of contamination may be made.

Additionally, there may be instances where little or no contamination was present in associated blanks, but qualification of sample was deemed necessary (e.g., contamination introduced through dilution water).

Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in a diluted sample result, but are absent in the undiluted sample result. Because both results are not routinely reported, it may be impossible to verify this source of contamination. However, if reviewer determines that contamination is from a source other than the sample, data shall be qualified. In this case, the 5-times criterion does not apply; the sample value shall be reported as a non-detect.

Examples of applying blank qualification guidelines follow. Certain circumstances may warrant deviations from these guidelines.

Case 1 Sample result is greater than RQL but is less than required amount (5 times) of blank result.

	5-Times
Blank result	1.0
RQL	0.5
Sample result	4.0
Qualified sample result	4.0U

In this case, sample results less than 5.0 (5 x 1.0) are qualified as non-detects.

Case 2 Sample result is greater than required amount (5-times) of blank result.

	5-Times
Blank result	1.0
RQL	0.5
Sample result	6.0
Qualified sample result	6.0

D.7.4.3 **Procedure for ASL D.** The following procedure applies only to ASL D data and shall be performed in addition to procedure in paragraphs D.7.4.2.

1. Review results of associated blanks and raw data (chromatograms, quantitation reports or data system printouts).

D.7.5 Percent Surrogate Recovery

Quality of laboratory analysis of individual samples is established by spiking samples with a surrogate compound prior to sample preparation and evaluating the percent recovery. However, evaluation of results of surrogate spikes is not necessarily straightforward. The sample itself may produce effects caused by factors such as interferences and high concentrations of analytes.

The review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement because effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems. Accordingly, this procedure consists primarily of guidelines and, in some cases, several optional approaches are suggested.

D.7.5.1 **Criteria.** The following criterion applies to both ASLs C and D data.

- Sample and blank recoveries of surrogates shall be within advisory limits of the specified method (Appendix G).

D.7.5.2 **Procedure for ASL C.** The following procedure applies to ASL C data only and shall be performed prior to and in addition to procedure in paragraph D.7.5.4.

1. Verify that surrogate recoveries are within advisory limits (paragraph D.7.5.1).

D.7.5.3 **Procedure for ASL D.** The following procedure applies to ASL D data only and shall be performed prior to and in addition to procedure in paragraph D.7.5.4.

1. Check raw data (e.g., chromatograms, quantitation list) to verify recoveries.

2. If recoveries are not within advisory limits, check raw data for possible interferences that may have affected surrogate recoveries.

D.7.5.4 Procedure for ASLs C and D. The following procedure applies to surrogate recovery data for both ASLs C and D and shall be performed after procedures in paragraphs D.7.5.2 and D.7.5.3.

1. Use the following guidelines if surrogate recoveries are outside advisory windows.

   a. If low recoveries are obtained, flag associated positive results and quantitation limits as estimated (J).

   NOTE

   A high bias may be caused by co-eluting interferences.

   b. If high recoveries are obtained, use professional judgement to determine appropriate action.

   c. If zero surrogate recovery is reported, examine sample chromatogram to determine if surrogate may be present, but slightly outside its retention-time window.

   d. If surrogate is present, in addition to assessing surrogate recovery for quantitative bias, investigate qualitative validity of analysis.

   e. If surrogate is not present, flag negative results as unuseable (R).

D.7.6 Matrix Spike/Matrix Spike Duplicates

MS/MSD data are generated to determine long-term precision and accuracy of the analytical method (Appendix G) on various matrices. These data alone cannot be used to evaluate precision and accuracy of individual samples.

This procedure applies to both ASLs C and D data.

D.7.6.1 Criteria.

- Advisory limits are established for spike recovery limits in the applicable method identified in Appendix G and on LSC-specified FEMP forms.

- Advisory limits are established for RPD between matrix spike and matrix spike duplicate recoveries in the applicable method identified in Appendix G and on LSC-specified FEMP forms.

D.7.6.2 Procedure.

1. Inspect results for MS/MSD recoveries.

2. Verify transcriptions from raw data for ASL D evaluation.

3. Verify calculations.

4. Do not use MS/MSD data alone to qualify an entire case, but, using informed professional judgement and MS/MSD results in conjunction with other QC criteria, determine need for qualification of data.

5. First try to determine extent of effects of MS/MSD results on associated data. Make this determination in regard to the sample as well as specific analytes for samples associated with MS/MSD.

6. If it can be determined that results of MS/MSD affect only the spiked sample, limit qualification to sample alone.

7. If it is determined through MS/MSD results that a laboratory is having a systematic problem in analysis of one or more analytes that affect associated samples, laboratory shall be notified and affected samples qualified.

D.7.7 Field Duplicates

Field duplicate samples may be collected and analyzed to evaluate overall precision. These analyses measure both field and laboratory precision; therefore, results may have more variability than laboratory duplicates, which measure only laboratory performance. It is also expected that solid matrix duplicate results will have a greater variance than duplicates of water matrices because of difficulty collecting identical samples. This procedure applies to both ASLs C and D data.

D.7.7.1 Criteria.

- There are no specific review criteria for field duplicate analyses comparability.

D.7.7.2 Procedure.

1. Identify field duplicate samples.

2. Compare results reported for each sample and calculate RPD.

3. Document field duplicate evaluation.

D.7.8 Compound Identification

Qualitative criteria for compound identification were established to minimize erroneous identifications, which can be either a false positive (reporting a compound when it is not present) or a false negative (not reporting a compound that is present).

D.7.8.1 Criteria. The following criteria apply to both ASLs C and D data.

- Retention times of reported compounds shall fall within calculated windows for the two chromatographic columns.

- GC/MS confirmation is required if the concentration of a compound exceeds 10 ng/μL in the final sample extract.

D.7.8.2 Procedure for ASL D Data. The following procedure shall be performed prior to and in addition to procedure in paragraph D.7.8.3.

1. Review compound identification results and associated raw data (chromatograms, data system printouts).

2. Confirm reported positive detects using appropriate retention times and retention-time windows, and verify that compounds listed as "not detected" are correct.

D.7.8.3 Procedure for ASLs C and D Data.

1. Verify that positive identifications have dissimilar column analysis.

2. If qualitative criteria for the two-column confirmation were not met, consider reported positive detects as non-detects; use professional judgement and assign an appropriate quantitation limit based on the following guidelines.

   a. If mis-identified peak was sufficiently outside target pesticide retention-time window, report RQL.

   b. If identified peak interferes with potential detection of target peak, consider reported value and flag it as estimated quantitation limit (UJ).

D.7.9 Compound Quantitation and Reported Detection Limits

The validation objective is to ensure that reported quantitation results and RQLs are accurate.

D.7.9.1 Criteria. The following criterion applies to both ASLs C and D data.

- Compound quantitation, as well as adjustment of the RQL, shall be calculated in accordance with the specified method in Appendix G.

D.7.9.2 Procedure for ASLs C and D. The following procedure applies to both ASLs C and D data.

1. Verify that RQLs were adjusted to reflect sample dilutions, splits, concentrations, clean-up activities, and dry weight factors that were not accounted for in the method.

2. Flag quantitation limits affected by large, off-scale peaks as unuseable (R).

3. If interference is on-scale, provide an estimated quantitation limit (UJ) for each affected compound.

4. Use professional judgement to decide whether a much larger concentration obtained in one column versus the other column indicates presence of an interfering compound.

5. If an interfering compound is indicated, report lower of the two values and qualify it as presumptively present at an estimated quantity (NJ), which will necessitate a determination of an estimated concentration in confirmation column.

6. Document that presence of interferences has obscured attempt at second-column confirmation.

D.7.9.3 Procedure for ASL D. The following procedure applies to ASL D data only and shall be performed in addition to procedure in paragraph D.7.9.2.

1. Examine raw data to verify correct calculation of sample results reported by the laboratory.

2. Compare quantitation reports, chromatograms, and sample preparation logs to reported positive sample results and quantitation limits.

D.7.10 Overall Assessment of Data for a Case

The data reviewer shall make professional judgements, express concerns, and comment on validity of the overall data package. This is particularly appropriate when there are several QC criteria out of specification.

The additive nature of QC factors out of specification is difficult to assess in an objective manner, but reviewer has a responsibility to inform users concerning data quality and limitations. Availability of DQOs is helpful in this review. The information will help the user avoid inappropriate use of data and yet not preclude all consideration of the data.

D.8 INORGANIC DATA VALIDATION PROCEDURES FOR ASLs C AND D

This subsection describes validation procedures for inorganic data for ASLs C and D. Validation procedures for inorganic data for ASL E are provided in subsection D.10. The following procedures are based on the *Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis*, 1 July 1988 (U.S. Environmental Protection Agency, 1988b).

D.8.1 Holding Times

The objective of validating holding times data is to establish the validity of analysis results by ensuring that sample holding times from receipt to analysis or extraction were in compliance with the specified method (Appendix G).

This procedure applies to both ASLs C and D data.

D.8.1.1 **Criteria.** Samples shall be analyzed within the holding times specified in Table 6-1.

D.8.1.2 **Procedure.** The following procedures apply to both ASLs C and D data.

1. Establish holding times by comparing sample collection date with dates of analysis in raw laboratory data (e.g., digestion logs and instrument run logs). Analyte holding time (days) equals analysis date minus sample collection date.

2. Examine digestion and distillation logs to determine if samples were preserved at pH specified in paragraph D.8.1.1.

3. If holding times and preservation requirements are not met, qualify results that are greater than IDL as estimated (J) and results smaller than IDL as estimated (UJ).

4. If holding times are exceeded, use professional judgement to determine reliability of data and effects of additional storage on sample results. The expected bias will be low, so reviewer may determine that results smaller than IDL are unuseable (R).

D.8.2 Calibration

Requirements for instrument calibration are established to ensure that instruments are capable of producing acceptable quantitative data. Initial calibration demonstrates that an instrument is capable of required performance at the beginning of an analysis run. Verification of continuing calibration ensures that the initial calibration remains valid.

D.8.2.1 **Initial Calibration Criteria for ASLs C and D.** The following criteria applies to data for both ASLs C and D.

- **ICP Analysis**
  - A blank and at least one standard shall be used to establish the analytical curve.
  - Analysis results shall fall within control limits of 90 to 110 Percent Recovery (%R) of true value.
- **AA Analysis**
  - A blank and at least three standards, one of which is at specified IDL, shall be used to establish the analytical curve.

NOTE

The correlation coefficient of 0.995 is a technical criterion and not contractual.

  - The correlation coefficient shall be greater than approximately 0.995.
  - Analysis results shall fall within control limits of 90 to 110 %R of true value.
- **Mercury Analysis**
  - A blank and at least four standards shall be used to establish the analytical curve.
  - The correlation coefficient shall be greater than approximately 0.995.
  - Analysis results shall fall within control limits of 80 to 120 %R.

D.8.7 Matrix Spike Sample Analysis

The matrix spike sample analysis provides information about effect of each sample matrix on digestion and measurement methodology.

D.8.7.1 Criteria. The following criteria apply to both ASLs C and D data.

- Samples identified as field blanks shall not be used for spiked sample analysis.
- Spike recovery (%R) shall be within 75 to 125 percent; however, spike recovery limits do not apply when sample concentration exceeds spike concentration by a factor of four or more.

D.8.7.2 Procedure for ASLs C and D. The following procedure applies to both ASLs C and D data.

1. Verify that the field blank was not used for spike analysis.
2. Review and verify that results fall within specified limits.
3. If spike recovery is greater than 125 percent and reported sample result is less than IDL, identify data as acceptable.
4. If spike recovery is greater than 125 percent or less than 75 percent and sample result is greater than IDL, qualify data as estimated (J).
5. If spike recovery is within the range of 30 to 74 percent and sample results are less than IDL, qualify data as estimated (UJ).
6. If spike recovery is less than 30 percent and sample results are less than IDL, qualify data as unuseable (R).
7. If the field blank was used for matrix spike analysis, check other QC data and exercise professional judgement to evaluate data. If matrix spike recovery does not meet criteria (except for silver), a post-digestion spike is required for all methods except furnace, but these data are not used to qualify sample results.

D.8.7.3 Procedure for ASL D. The following procedure applies to ASL D data only and shall be performed in addition to procedure in paragraph D.8.7.3.

1. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

D.8.8 Furnace Atomic Absorption

Duplicate injections and furnace post-digestion spikes establish precision and accuracy of individual analytical determinations.

D.8.8.1 **Criteria for ASLs C and D.** The following criteria apply to both ASLs C and D data.

- For sample concentrations greater than RQL, duplicate injections shall agree within ± 20 percent Relative Standard Deviation (RSD). Otherwise, sample shall be rerun once (at least two additional injections).

$$\text{Standard Deviation (SD)} = \left[\frac{\Sigma (x^i - x)^2}{n - i} \right]^{1/2}$$

Where:

$$\text{Relative Standard Deviation (RSD)} = \frac{SD}{x} \times 100$$

- Spike recovery shall be greater than 75 percent and less than 125 percent.
- The furnace AA method shall be used as specified in Appendix G.

D.8.8.2 **Procedure.** The following procedure applies to both ASLs C and D data.

1. Check raw data for ASL D validation to verify that duplicate injections agree within ± 20 percent of RSD or Coefficient of Variation (CV) for sample concentrations higher than RDL.

2. Review furnace AA raw data for ASL D validation to verify that the method has been followed.

3. If duplicate injections are outside ± 20 percent of RSD or Coefficient of Variation (CV) limits and sample has not been rerun once, qualify data as estimated (J).

4. If rerun sample results do not agree within ± 20 percent of RSD limits, qualify data as estimated (J).

5. If post-digestion spike recovery is less than 40 percent, qualify results higher than IDL as estimated (J).

6. If post-digestion spike recovery is \greater than or equal to 10 percent but less than 40 percent, qualify results less than IDL as estimated (UJ).

7. If post-digestion spike recovery is less than 10 percent, qualify results less than IDL as unuseable (R).

8. If sample absorbance is less than 50 percent of post-digestion spike absorbance, proceed as follows.

   a. If furnace post-digestion spike recovery is not within 75 to 125 percent, qualify sample results higher than IDL as estimated (J).

   b. If furnace post-digestion spike recovery is not within 75 to 125 percent, qualify sample results less than IDL as estimated (UJ).

9. If MSA is required but has not been done, qualify data as estimated (J).

10. If samples run by MSA have not been spiked at appropriate levels, qualify data as estimated (J).

11. If MSA correlation coefficient is less than 0.995, qualify data as estimated (J).

D.8.9 ICP Serial Dilution

The serial dilution determines whether significant physical or chemical interferences exist because of the sample matrix.

D.8.9.1 <u>Criteria</u>. The following criterion applies to both ASLs C and D data.

- If analyte concentration is sufficiently high (concentration in original sample is minimally a factor of 50 above IDL), an analysis of a five-fold dilution shall agree within 10 %D of original results.

D.8.9.2 Procedure for ASLs C and D. The following procedure applies to both ASLs C and D data.

1. Check raw data for ASL D validation evidence of negative interference (i.e., diluted sample results are significantly higher than original sample).

2. When criteria are not met, qualify associated data as estimated (J).

3. If evidence of negative interference is found, use professional judgement to qualify data.

D.8.9.3 Procedure for ASL D. The following procedure applies to ASL D data only and shall be performed in addition to procedure in paragraph D.8.9.3.

1. Check raw data and recalculate %D using the following equation to verify that dilution analysis results agree with reported results.

$$\%D = \frac{|I-S|}{I} \times 100$$

Where:

I = Initial sample result

S = Serial dilution result (instrument reading times five)

D.8.10 Sample Result Verification

The objective is to ensure that reported quantitation results are accurate.

D.8.10.1 Criteria. The following criterion applies to both ASLs C and D data.

- Analyte quantitation shall be calculated as specified in the applicable method identified in Appendix G.

D.8.10.2 Procedure for ASLs C and D Data.

1. Examine raw data for ASL D validation and verify correct calculation of sample results reported by the laboratory.

2. Compare digestion and distillation logs, instrument printouts, and strip charts to reported sample results for ASL D validation.

3. Verify that there are no transcription or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.

4. Verify that results fall within linear range of ICP and within calibrated range for non-ICP parameters.

NOTE

When the laboratory provides both ICP and furnace results for an analyte in a sample and concentration is greater than ICP IDL, results may be used to assist in quantitation problem identification.

5. If ICP analysis results are used for arsenic, thallium, selenium, or lead, proceed as follows.

   a. Verify that sample results are greater than five times ICP IDL.

   b. If discrepancies are found, contact laboratory to obtain additional information to resolve differences. If a discrepancy remains unresolved, qualification of data may be warranted.

D.8.10.3 <u>**Procedure for ASL D.**</u> The following procedure applies to ASL D data only and shall be performed in addition to procedure in paragraph D.8.10.3.

1. Examine raw data for ASL D validation for anomalies (e.g., baseline shifts, negative absorbance, omissions, legibility).

D.8.11 Field Duplicates

Field duplicate samples may be collected and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. Therefore, the result may have more variability than laboratory duplicates, which measure only laboratory performance. It is expected that soil duplicate results will have a greater variance than duplicates of water matrices because of difficulties associated with collecting identical soil samples.

D.8.11.1 Criteria. There are no review criteria for field duplicate analyses comparability.

D.8.11.2 Procedure for ASLs C and D.

1. Identify field duplicate samples on field sample sheets.

2. Compare reported results for each sample and calculate RPDs if appropriate.

3. Provide reviewer comments with evaluation report of field duplicates.

D.8.12 Overall Assessment of Data for a Case

This procedure is applicable to ASL D data only. The data reviewer shall make professional judgements, express concerns, and comment on validity of the overall data package for a case. This is particularly appropriate when there are several QC criteria out of specification.
The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform users concerning data quality and limitations. Availability of DQOs is helpful in this review. The information will help the user avoid inappropriate use of data and yet not preclude all consideration of the data.

D.9 DATA VALIDATION PROCEDURES FOR GAS CHROMATOGRAPHY OF ORGANIC COMPOUNDS FOR ASLs B, C, AND D

Gas chromatography procedures for organic compounds are adapted from ***Test Methods for Evaluating Solid Waste***, EPA SW-846, Third Edition, November 1986. The following procedures apply to data for ASLs B, C, and D.

D.9.1 Validation Guidelines for Gas Chromatography Data

D.9.1.1 Guidelines for ASL B Data. There are two sub-levels of ASL B data, and they require different validation procedures.

If the samples taken are user-defined as ASL B, they shall be validated in accordance with requirements in the PSP for that sampling event. When the data user specifies the QC requirements, the validation requirements shall also be specified in the PSP.

If ASL B analysis is specified, QC information shall be reviewed and compared to the QC acceptance criteria of the individual methods. The portions of ASLs C and D procedures that are applicable (e.g., matrix spike/matrix spike duplicate, blanks, laboratory control samples) shall be used as the outline for review. The specific acceptance criteria from the Appendix G method shall be used.

surrogate recoveries, the blank problem may be an isolated occurrence. Even if this judgement allows some use of the affected data, analytical problems remain, which shall be reported to and corrected by the laboratory.

D.9.6 Matrix Spike/Matrix Spike Duplicate.

MS/MSD data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate precision and accuracy of individual samples.

D.9.6.1 Criteria. Spike recoveries and RPDs between MS/MSD recoveries shall be within advisory limits in the applicable method table in Appendix G.

D.9.6.2 Procedure for ASLs C and D Data.

1. Inspect data results for MS/MSD recovery.

2. Verify transcriptions from raw data and verify calculations for ASL D validation.

3. Do not use MS/MSD data alone to qualify an entire case. Use informed professional judgement and MS/MSD results in conjunction with other QC criteria to determine the need for some qualification of the data.

4. Assess effect of results of MS/MSD on associated data with regard to the MS/MSD sample itself plus specific analytes for samples associated with the MS/MSD.

5. If it can be determined that results of the MS/MSD affect only the spiked sample, limit qualification to this sample alone.

6. If it is determined through MS/MSD results that a laboratory is having a systematic problem in analysis of one or more analytes, apply qualification to associated samples.

D.9.7 Compound Identification

D.9.7.1 Criteria.

- Retention times of reported compounds shall fall within the calculated window for two chromatographic columns.

- Second-column confirmation is mandatory at ASLs C and D. If qualitative criteria for two-column confirmation are not met, reported positive detects shall be considered non-detects.

D.9.7.2 Procedure.

1. Use professional judgement to assign an appropriate quantitation limit based on the following guidelines.

   a. If misidentified peak was sufficiently outside target compound retention-time window, RQL may be reported.

   b. If misidentified peak poses an interference with potential detection of a target peak, reported value shall be considered and flagged as estimated quantitation limit (UJ).

D.9.8 Laboratory Control Samples

D.9.8.1 Criteria. Internal QC limits set by the applicable method (Appendix G) for a given sample matrix shall be applied.

D.9.8.2 Procedure.

1. If LCS exceeds method limits for a given sample matrix, inspect data from the associated sample batch.

2. If no analytical problems are found, compare data analyzed with the out-of-control point in the QC section of the case narrative provided with the data package by the laboratory performing the analyses.

3. If problems are found in analytical data, re-analyze samples associated with the batch and report data from the re-analysis.

4. If holding times are exceeded during re-analysis, include both sets of data in the data package.

5. If LCS and matrix spike results are outside method limits, either re-analyze sample within holding times or flag data as unuseable (R).

D.9.9 Compound Quantitation and Reported Detection Limits

The objective is to ensure that reported quantitation results and RQLs are accurate.

D.9.9.1 Criteria. The following criterion applies to both ASLs C and D data.

- Compound quantitation, as well as adjustment of the RQL, shall be calculated in accordance with the method specified in Appendix G.

D.9.9.2 Procedure for ASLs C and D. The following procedure applies to both ASLs C and D data.

1. Verify that RQLs were adjusted to reflect sample dilutions, splits, concentrations, clean-up activities, and dry weight factors there were not accounted for in the analytical method.

2. Flag quantitation limits affected by large, off-scale peaks as unuseable (R).

3. If interference is on-scale, provide an estimated quantitation limit (UJ) for each affected compound.

4. Use professional judgement to decide whether a much larger concentration obtained in one column versus the other column indicates presence of an interfering compound.

5. If an interfering compound is indicated, report lower of the two values and qualify it as presumptively present at an estimated quantity (NJ), which will necessitate a determination of an estimated concentration in confirmation column.

6. Document that presence of interferences has obscured attempt at second-column confirmation.

D.9.9.3 Procedure for ASL D. The following procedure applies to ASL D data only and shall be performed in addition to procedure in paragraph D.7.9.3.

1. Examine raw data to verify correct calculation of sample results reported by the laboratory.

2. Compare quantitation reports, chromatograms, and sample preparation logs to reported positive sample results and quantitation limits.

D.9.10 Overall Assessment of Data for a Case

The data reviewer shall make professional judgements, express concerns, and comment on validity of the overall data package. This is particularly appropriate when there are several QC criteria out of specification.

The additive nature of QC factors out of specification is difficult to assess in an objective manner, but reviewer has a responsibility to inform users concerning data quality and limitations. Availability of DQOs is helpful in this review. The information will help the user avoid inappropriate use of data and yet not preclude all consideration of the data.

D.10 DRINKING WATER DATA VALIDATION PROCEDURES FOR ASL B

Drinking water data validation procedures are based on the EPA method dated December 1988 for GC/MS analysis of volatile organic compounds.

D.10.1 Internal Standards

D.10.1.1 Criteria. The following IS performance criteria will ensure that GC/MS sensitivity response is stable during every run.

- IS area counts shall not vary by more than a factor of two (- 50 percent to + 100 percent) from associated calibration standard.
- IS retention time shall not vary more than ± 30 seconds from associated calibration standard.

D.10.1.2 Procedure.

1. Check raw data to verify recoveries of ISs.
2. Verify retention times.
3. If ISs are outside windows, consider a partial or total rejection of data for that sample fraction.

D.10.2 Surrogate Analytes

D.10.2.1 Criteria. Sample and blank surrogate recoveries shall be within specified limits.

D.10.2.2 Procedure.

1. Check raw data to verify surrogate recovery.
2. If surrogates are outside limits, flag positive results for that fraction as estimated (J).

D.10.3 Laboratory Duplicates

Analysis of laboratory duplicates gives a measure of the precision associated with laboratory procedures.

D.10.3.1 Criteria.

- Specific criteria for laboratory duplicate analyses comparability are specified in the applicable method (Appendix G).

D.10.3.2 Procedure.

1. Compare results for each sample and calculate RPD.

2. If laboratory duplicate samples are outside control limits, re-analyze them.

D.10.4 Field Duplicates

Analysis of field duplicates gives a measure of precision to sample collection, preservation, and storage as well as to laboratory procedures. Field duplicate samples are collected every sampling round or sample delivery group.

D.10.4.1 Criteria. There are no specific criteria for field duplicate analyses comparability.

D.10.4.2 Procedure.

1. Compare results for each sample and calculate RPD.

D.10.5 Laboratory Reagent Blanks

Assessment of blank analysis results identifies existence and magnitude of contamination problems.

D.10.5.1 Criteria. Contaminants should not be present in blanks.

D.10.5.2 Procedure.

1. If gross contamination exists in the blank, flag affected compounds as unuseable (R).

2. If inordinate amounts of target compounds are found at low levels, take corrective action as this is indicative of a laboratory problem.

D.10.6 Field Reagent Blanks

Reagent water is placed in a sample container in a laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and analytical procedures.

D.10.6.1 Criteria. There are no criteria for field reagent blanks.

D.10.6.2 Procedure.

1. If contamination exists, record this fact in data review comments and forward to FEMP project manager.

D.10.7 Laboratory Performance Check Solutions

A laboratory check solution is made up of one or more compounds and used to evaluate performance of the instrument system.

D.10.7.1 Criteria. Criteria are established in the applicable method (Appendix G).

D.10.7.2 Procedure.

1. If check solution is outside control limits, take corrective action (e.g., trouble-shoot instrument and standards preparation).

D.10.8 Matrix Spike/Matrix Spike Duplicates

MS/MSD data are generated to determine long-term precision and accuracy of analytical method on various matrices.

D.10.8.1 Criteria. Spike recoveries shall be within advisory limits established in the applicable method (Appendix G).

D.10.8.2 Procedure.

1. If results are outside advisory limits, use results in conjunction with other QC criteria and establish need for qualification of data.

D.10.9 Calibration Standards

D.10.9.1 Criteria.

- VOA analytes and surrogates are expressed as a percentage of true value and shall be 80 to 120 percent of true value.
- RSD shall be less than 20 percent of true value.
- For continuing calibration, the response factor for each analyte and surrogate shall be within 30 percent of mean value measured in initial calibration.

D.10.10 Compound Quantitation and Reported Detection Limits

The objective is to ensure that reported quantitation results and RQLs are accurate.

D.10.10.1 Criteria. The following criterion applies.

- Compound quantitation, as well as adjustment of the RQL, shall be calculated in accordance with the specified method (Appendix G).

D.10.10.2 Procedure.

1. Examine raw data to verify correct calculation of sample results reported by the laboratory.

2. Compare quantitation reports, chromatograms, and sample preparation logs to reported positive sample results and quantitation limits.

3. Verify that RQLs were adjusted to reflect sample dilutions, splits, concentrations, clean-up activities that were not accounted for in the Appendix G method.

4. Flag quantitation limits affected by large, off-scale peaks as unuseable (R).

5. If interference is on-scale, provide an estimated quantitation limit (UJ) for each affected compound.

6. Use professional judgement to decide whether a much larger concentration obtained in one column versus the other column indicates presence of an interfering compound.

D.10.11 Overall Assessment of Data for a Case

The data reviewer shall make professional judgements, express concerns, and comment on validity of the overall data package. This is particularly appropriate when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but reviewer has a responsibility to inform users concerning data quality and limitations. Availability of DQOs is helpful in this review. The information will help the user avoid inappropriate use of data and yet not preclude all consideration of the data.

D.11 DATA VALIDATION PROCEDURE FOR RADIOLOGICAL ANALYSES

Generally, validation of the data will include examination of the digestion, separation, or other preparation logs, all instrument printouts, including spectra and counting logs for all samples, standards, and QC samples. Chain-of-custody records, calibration data, including certifications of standards, calculations of the detection levels and results, background results, and if available, computer algorithms must also be examined.

Calculations made from the raw data are verified to ensure that no transcription errors were made and that all results are correctly reported in the data package. Verification includes checking the mathematical operations including conversion of units and dilution factors. Other radiological parameters such as the half-lives, decay corrections, branching ratios, dead times for counters, and correlation coefficients for efficiency curves may need verification as well. Requirements to be reviewed during validation are listed below.

1. Completeness Checks
2. Calibration
3. Blanks
4. Detection Limits and Sample Results
5. Radiometric and Gravimetric Yields
6. Duplicate Samples and Analyses
7. Laboratory Control Samples
8. Holding Times
9. Analysis of Alpha-Emitting Radium Isotopes Using Scintillation Counting
10. Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting
11. Supplemental Requirements for Fluorometric Analysis of Uranium
12. Other Quality Control

D.11.1 Completeness Checks

Examine the data package for completeness. Use the checklists included in the Appendices, but also refer to the Statement of Work (SOW) for the analytical laboratory. Items specified in the SOW may supplement or take precedence over the list of items in the checklists.

- The analytical laboratory should provide evidence that interference from cations or anions is negligible, or that steps have been taken to minimize their effects. If evidence is not provided, qualify associated results as estimated (J).

D.11.10.4 **Method Standardization.** The fusion operation is the most critical step in the fluorometric procedure. Small variations in the duration of the fusion temperature of the fusion, and in the method of cooling the fused disk can cause large variations in the fluorescence yield. Each step of the fusion process should be standardized to obtain reproducible results.

- The analytical laboratory should provide a description of the method for fusion standardization. If the fusion process is not standardized, or information is not provided to allow the independent assessment of the standardization process, qualify associated results estimated (J).

D.11.11 Other Quality Control

Other QC checks give the data reviewer an opportunity to provide additional documentation that may be applicable to a particular SDG or useful to data users. The reviewer can also express comments on the overall data quality for an SDG. Other areas that may be addressed under other QC include, but are not limited to, documentation of the following.

- Trends observed in the performance of an instrument, method, or the laboratory over the course of the SDG or past history
- Anomalies associated with the Chain-of-Custody documentation
- Anomalies associated with the shipment or receipt of samples.

It is left to the discretion of the reviewer to evaluate the nature of any problems observed and to attach any qualification which may be necessary to describe the quality of the data. All anomalies and any action taken shall be clearly documented.

D.12 INORGANIC (NON-METALS) DATA VALIDATION PROCEDURES

This subsection describes validation procedures for inorganic (non-metals) data.

D.12.1 Holding Times

The objective of validating holding times data is to establish the validity of analysis results by ensuring that sample holding times from receipt to analysis or extraction were in compliance with the specified method (Appendix G).

D.12.1.1 Criteria. The maximum holding times for completion of laboratory sample analysis and preservation requirements are specified in Table 6-1 (Appendix A) of the SCQ.

D.12.1.2 Procedure.

1. Establish holding times by comparing sample collection date with dates of analysis in raw laboratory data (e.g., digestion logs and instrument run logs).

2. Examine digestion and distillation logs to determine if samples were preserved as specified in Table 6-1 (Appendix A).

3. If holding times and preservation requirements are not met, qualify results that are greater than RQL as estimated (J) and results smaller than IDL as estimated (UJ). Analyte holding time (days) equals analysis date minus sample collection date.

4. If holding times are exceeded, use professional judgement to determine reliability of data and effects of additional storage on sample results.

D.12.2 Calibration

Requirements for instrument calibration are established to ensure that instruments are capable of producing acceptable quantitative data. Initial calibration demonstrates that an instrument is capable of required performance at the beginning of an analysis run. Verification of continuing calibration ensures that initial calibration remains valid.

Requirements for initial and continuing calibration are specified in each method. Results of initial and continuing calibration shall be compared to method requirements. If method requirements are not met the reviewer may qualify the associated data as estimated (J) if the variance is small or unuseable (R) if it is major. Professional judgement shall be used to assess the nature of the variances and whether they are major or minor in effect.

D.12.3 Blanks

Blank analysis results assessment helps determine existence and magnitude of sample contamination problems. Criteria for evaluation of blanks apply to all blanks associated with sample. If problems with blanks data for exist, data associated with the case shall be evaluated to determine whether there is an inherent variability in data for the case or if the problem is an isolated occurrence not affecting other data.

D.12.3.1 Criteria. There shall be no contaminants in blanks.

D.12.3.2 Procedure.

1. Review analytical results as well as raw data (printouts, strip charts, printer tapes, bench sheets) for blanks and verify that results are reported accurately.

Where:

S = first sample value (original)

D = second sample value (duplicate)

3. If duplicate analyses results for an analyte fall outside appropriate control windows, qualify results for the analyte in associated samples of the same matrix as estimated (J).

4. If a field blank was used for duplicate analyses, check other QC data and exercise professional judgement to evaluate data.

D.12.6 Matrix Spike Sample Analysis

The matrix spike sample analysis provides information about affect of each sample matrix on digestion and measurement methodology.

D.12.6.1 **Criteria.** The following criteria apply to methods where matrix spike samples are analyzed.

- Samples identified as field blanks shall not be used for spiked sample analysis.
- Spike recovery (%R) shall be within 75 to 125 percent; however, spike recovery limits do not apply when sample concentration exceeds spike concentration by a factor of four or more.

D.12.6.2 **Procedure.**

1. Verify that the field blank was not used for spike analysis.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

3. Review and verify that results fall within specified limits.

4. If spike recovery is greater than 125 percent and reported sample result is less than RQL, identify data as acceptable.

5. If spike recovery is greater than 125 percent or less than 75 percent and sample result is greater than RQL, qualify data as estimated (J).

6. If spike recovery is within the range of 30 to 74 percent and sample results are less than RQL, qualify data as estimated (UJ).

7. If spike recovery is less than 30 percent and sample results are less than RQL, qualify data as unuseable (R).

8. If the field blank was used for matrix spike analysis, check other QC data and exercise professional judgement to evaluate data.

D.12.7 Sample Result Verification

The objective is to ensure that reported quantitation results are accurate.

D.12.7.1 Criteria. The following criterion applies.

- Analyte quantitation shall be calculated as specified in the applicable method in Appendix G.

D.12.7.2 Procedure. The following procedure applies.

1. Examine raw data and verify correct calculation of sample results reported by the laboratory. Examine raw data for anomalies (e.g., baseline shifts, negative absorbance, omissions, legibility).

2. Compare digestion and distillation logs, instrument printouts, and strip charts to reported sample results.

3. Verify that there are no transcription or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.

4. Verify that results fall within calibrated range.

D.12.8 Field Duplicates

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision; therefore, the result may have more variability than laboratory duplicates, which measure only laboratory performance. It is

APPENDIX E

ANALYTICAL LABORATORY PERFORMANCE REQUIREMENTS

E.1 INTRODUCTION

Analytical performance requirements shall be used as guidelines for evaluating laboratory capability to provide specific analytical services to FEMP. Ability to meet these requirements shall be audited prior to contract award as described in Section 12. Subsequent post-contract-award audits shall be performed to verify laboratory performance using the performance-evaluation sample results specified in subsection E.2 and Section 3.

E.1.1 Purpose

This appendix establishes performance requirements for laboratories doing analytical work for FEMP. Laboratories shall use organic and inorganic methods specified in Tables G-1 and G-2. Radiochemical analysis shall meet performance specifications identified in Tables G-3 and G-4.

E.1.2 Scope

General requirements for laboratories performing analysis for FEMP are provided in the following subsections.

- Laboratory Approval (subsection E.2)
- Requirements (subsection E.3)
    - Equipment (paragraph E.3.1)
    - Sample Receipt and Documentation (paragraph E.3.2)
    - Preparation, Analysis, and Identification of Analytes (paragraph E.3.3)
    - Quality Assurance/Quality Control Procedures (paragraph E.3.4)
    - Reports and Deliverables (paragraph E.3.5)

E.1.3 FEMP Project Contact

The FEMP project contact and the laboratory project manager are project-specific functions, and shall be identified in the project-specific plan. Project correspondence shall be directed through these individuals.

E.1.4 Lab Contract Technical Representative

The lab contract technical representative is a FEMP individual or group tasked with dealing with a specific subcontractor laboratory on contract and organizational issues.

E.1.5 FEMP Manager of Site Sample Management

The FEMP Manager of Site Sample Management is an individual subordinate to the FEMP Sampling and Analysis Management Coordinator responsible for maintaining and updated the FEMP approved laboratory list and for coordinating audits with the designated FEMP QA organization.

E.2 LABORATORY APPROVAL

The FEMP sampling and analysis management coordinator shall maintain a list of analytical laboratories approved for FEMP sample analyses.

E.2.1 Requirements for an Approved Laboratory

A laboratory which demonstrates compliance with the following requirements shall be considered approved to perform work for the FEMP for the ASL and types of analyses considered. An approved laboratory:

1. Has been audited/surveyed by FEMP personnel to ensure compliance with these requirements and to document the compliance.

2. Has the necessary licenses and/or certifications to handle and process FEMP samples.

3. Has standard operating procedures (SOPs) in place which address sample receiving, login, storage, analysis, and disposal. Analysis SOPs shall meet the applicable requirements of Appendix G. Other specific SOPs shall also be required depending on the ASL involved, as dictated by the SCQ.

4. Has adequate building security and Chain-of-Custody system with applicable SOPs.

5. Has a document control system which addresses all SOPs and the Quality Assurance Manual.

6. Has a QA Program which addresses the applicable requirements of the most recent version of ANSI/ASCQ/E4-19xx, and the FEMP SCQ.

7. Can document personnel and laboratory experience in the analysis category (inorganic, organic, asbestos, radiochemical, geotechnical), including acceptable performance in performance evaluation programs. Analytical performance and financial stability will have been verified via reference checks with previous and/or current customers.

8. Can demonstrate the ability to comply with all applicable QC requirements of the SCQ.

9. Can demonstrate the ability to comply with all applicable reporting requirements of the SCQ.

10. Can demonstrate the ability to comply with all other contractual requirements as will be set forth in technical Statements of Work. This shall include the statement that "All contractual requirements shall be met, except for the following:..." in all contract proposals. Any exceptions must be agreed upon by the individuals signing the letter described below. These exceptions shall in no case supersede the requirements of the SCQ.

11. Has a Program Management description which identifies the single point of contact at the laboratory, how FEMP sample will be tracked and processed on a daily basis, and how the lab will ensure compliance with all of the relevant SCQ requirements including QC and reporting.

12. Has laboratory and administrative programs in place which comply with the requirements of OSHA, e.g., use of MSDSs, a Chemical Hygiene Plan, a Radiation Safety Program (as applicable), and a Hazardous Waste Management Program.

E.2.2 Laboratory Performance Review

To assure data comparability, each laboratory must participate in the FERMCO interlaboratory comparability program. This program will consist of analysis, by each laboratory, of blind QC samples, such as split samples, matrix spikes and matrix spike duplicates, and duplicate samples prepared by FERMCO.

Each laboratory will be surveyed, prior to contract award, to ensure compliance with these items. All documentation received with contract proposals and during site visits shall be maintained in a laboratory specific file. The lab contract technical representative (CTR) shall also perform the following activities, at a minimum, to ensure the continued acceptable performance of each lab performing analyses for the FEMP.

1. Follow-up audits will be conducted, subsequent to award, at least annually consistent with Section 12 of the SCQ. All checklists, reports, and corrective action verification shall be maintained in the appropriate file. More frequent audits will be conducted as dictated by lab performance and/or the importance or number of analyses being performed.

2. Monthly performance reports will be submitted to the CTR by all laboratories. These reports will include:

   - A discussion of any problems encountered during the month as related to the processing of FEMP samples.

- A review of samples processed, including identification of samples received, reported on time, reported late, in process, and an indication of holding time compliance.

- Charts or tables which summarize the performance on FEMP supplied blind samples during the month and over the life of the contract. This shall include a narrative which summarizes the performance.

- Copies of round-robin program results received during the month and an analysis of the performance.

- Mention of any reduced (or enhanced) ability to perform under the terms of the contract. This shall include new analysis capabilities, additional or reduced sample capacity, and personnel changes.

The CTR will review these reports, follow-up as necessary, document all resultant conversations with the lab, and file all of this information.

3. Face-to-face contacts between the CTR or designee and contract lab personnel at least semi-annually. This requirement can be met by the annual audit, a visit to the lab to check on samples (announced or unannounced) or a visit to the FEMP by laboratory personnel.

4. Phone calls to each laboratory processing samples shall occur at least weekly and will be documented.

5. Data packages received from the laboratories will be reviewed according to standardized checklists. Compliance with regulatory and contractual requirements shall be confirmed in each case.

E.2.3 Approved List of Laboratories

The approved laboratory list will include labs that are currently approved and whose approval is not current. The list can then be used for historical purposes. Only currently approved laboratories may perform work for FEMP.

This list will contain the following information:

- Date of issuance of the list.

- Revision number of the list.

- Laboratory name and location.

- Analysis category.

- ASL.

- Approval status - examples are currently approved and contracted, currently approved but not contracted, and not currently approved. A "not currently approved lab" is one whose approval has been discontinued or has lapsed. The details for this will have been provided in the letter described in Item D.

- Period of performance.

- Date of last audit.

- Date of next scheduled audit.

- Remarks.

Revision of the approved laboratory list will be accomplished as follows.

Because of changing needs, management or contract changes, or other unforseen circumstances, the approved laboratory list is expected to change over time.

Step 1: A member of the contract Source Evaluation Board (SEB) or the Site Sample Management employee responsible for the contract in question submits a letter to the FEMP Manager of Site Sample Management (SSM). The letter is entitled "Recommendation to Revise the Approved List of Laboratories to Perform Work for the FEMP" and includes what change is required and concurrence lines for the Manager of SSM and a representative of the designated FEMP QA organization (QA representative). The letter must also include a listing of the applicable ASLs, and the category of analyses affected.

Additions must include statements, and all applicable documentation (e.g., audit reports and licenses), that indicate compliance with all of the eleven requirements specified in Item A.

Deletions (designations of discontinued approval) must state reasons why. These include lapsed contract, audit not performed per frequency requirement, and poor performance. Poor performance can include disapproval, by the EPA, of work performed under non-FEMP contracts. A "poor performance" statement must include details.

Step 2: The manager of SSM and the QA representative sign the concurrence line.

Step 3: SSM personnel, by copy of the letter, revise the list.

Step 4: Controlled copies of the list are then distributed. The EPA receives a copy which includes a cover letter which indicates the changes made and which includes all attachments.

The list will be revised each time a change is necessary, but no more frequently than monthly.

A review of this approach to maintaining a list of approved labs shall be conducted as necessary. Changes will be incorporated into the SCQ as dictated by said review.

E.3 EQUIPMENT

Each laboratory must have equipment in top working order capable of performing the analyses for which it bids to perform for FEMP.

E.3.1 Inorganic Compound Analysis. The laboratory shall have equipment capable of performing inorganic compound analyses by specified methods in Appendix G. The following equipment is required for certain methods.

- Inductively Coupled Plasma (ICP) emission spectrometer
- Atomic Absorption (AA) spectrometer with graphite furnace and cold vapor

E.3.2 Organic Compound Analysis. The laboratory shall have equipment capable of performing organic compound analyses by specified methods in Appendix G. The following equipment is required for certain methods.

- Infrared spectrometer
- Gas chromatograph/mass spectrometer
- Gas chromatograph/electron-capture detector
- Gas chromatograph/photo ionization detector
- Gas chromatograph/electrolytic conductivity detector
- Gas chromatograph/flame photometric detector
- Gas chromatograph/flame ionization detector
- High performance liquid chromatograph

E.3.3 Radiological Analysis. The laboratory shall have equipment capable of performing radiological analyses to meet performance specifications identified in Table G-4 (Appendix G). The following equipment is typically used to perform radiochemical analyses.

- Liquid scintillation counting systems

- Alpha spectrometer systems
- Alpha/beta counting systems
- Germanium spectroscopy systems
- Alpha scintillation counting instruments
- Ultraviolet/visible spectrophotometers

E.4 SAMPLE RECEIPT AND DOCUMENTATION

Following are general requirements for sample receipt and preparation. Specific procedures for receipt and preparation of samples are provided in Section 7.

E.4.1 Chain of Custody. Laboratory custody procedures shall be documented and implemented so that the following conditions are met for samples at all times prior to and during analysis. Procedures shall be consistent with Section 7. Documentation of sample custody from time of receipt to final laboratory disposition shall be maintained. A sample is considered in custody when one of the following are met.

- The sample shall remain in one person's possession;
- Or the sample shall be in that custody holder's view after being in holder's possession;
- Or the sample shall be in custody holder's possession and placed in a secure, controlled-access storage area by holder;
- Or the sample shall be in a designated secure area accessible to authorized personnel only.

E.4.2 Document Control. Document control ensures that data for specified sample sets are accounted for after completion of a project. The laboratory shall have written document control measures that shall be specified in the laboratory quality assurance plan in accordance with SCQ Sections 4 and 11. The following document control forms are required.

- Data sheets
- Logs or daily log forms

E.4.3 Standard Operating Procedures. The laboratory shall have written standard procedures for sample receipt, log-in, and storage. These procedures shall be subject to FEMP approval and in accordance with the SCQ.

E.5 PREPARATION, ANALYSIS, AND IDENTIFICATION OF ANALYTES

The laboratory shall demonstrate capability of preparing and analyzing samples and identifying constituents of concern by specified methods and performance specifications in Appendix G. Quantitation limits for analyte targets are provided in the specified method or performance criterion.

E.6 QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

The laboratory shall be responsible for performing Quality Assurance/Quality Control (QA/QC) procedures in strict accordance with Sections 4, 10, and 11 and the laboratory-specific contract, including specified holding times and other criteria. Quality Control (QC) samples for laboratory analysis are defined in Section 4 and listed in Table 2-2 in Appendix A.

Analytical laboratories shall be required to have an internal quality assurance plan and applicable standard operating procedures in place as specified in Section 12. Adherence to the elements of the plan shall be documented in audits.

The SCQ shall be a contract-specified attachment to the laboratory-specific quality assurance plan. Compliance with the SCQ shall be verified through project performance audits. Additional QA/QC requirements may be specified in individual contract statements of work.

E.7 REPORTS AND DELIVERABLE ITEMS

Requirements for reports and deliverable items depend upon the specified Analytical Support Level (ASL) (Section 2). The following paragraphs summarize laboratory requirements.

E.7.1 Inorganic Compound Samples. The following report forms are required for inorganic sample reporting for ASL C data.

- Inorganic analysis data sheet
- Radiation Detection Limit (RDL) standard for Atomic Absorption (AA) and ICP
- Blanks
- Spike sample recovery
- Duplicates

The following data are required for inorganic sample reporting for ASL D data in addition to the preceding requirements for ASL C data.

- Initial and continuing calibration verification

- ICP interference check sample
- Post-digest spike sample recovery
- Laboratory control sample
- ICP serial dilution
- Analysis run log
- Preparation log

Requirements for ASLs B and E data shall be specified in project-specific plans.

E.7.2 Organic Compound Samples. The following report forms are required for organic sample reporting for ASL C data.

- Organic analyte data sheet
- Surrogate recovery forms
- Matrix spike/matrix spike duplicate forms
- Method blank summary
- Analyte resolution summary (pesticides only)
- Identification summary for single or multicomponent analytes (pesticides only)

The following data are required for organic sample reporting for ASL D data in addition to the preceding requirements for ASL C data.

- Instrument performance check
- Initial calibration data
- Continuing calibration check
- Calibration verification (pesticides only)
- Florisil check (pesticides only)
- Gel permeation chromatography calibration (pesticides only)
- Internal standard error and retention time summary for volatile organic analysis/base neutral analysis

Requirements for ASLs B and E data shall be specified in project-specific plans.

E.7.3 Radiometric Samples. The following report forms are required for radiometric sample reporting for ASLs C or D data.

- Radiochemical data completeness checklist
- Data assessment summary report form
- Radiochemical analysis results

Requirements for ASLs B and E data shall be specified in project-specific plans.

observations (e.g., temperature, pH, and specific conductance) are also measured and recorded, but no physical samples are sent to the laboratory. Each sample or piece of recorded data is referenced to an on-site or off-site location through the state of Ohio planar coordinate system (Sections 5 and 6).

F.1.2.4 Transfer and Handling of Samples. Samples collected on site for laboratory analysis are identified with a sample number, packaged, and transported to the laboratory. Custody and other records are maintained for sample tracking from time of collection through final disposition (Sections 5, 6, and 7).

F.1.2.5 Laboratory Analysis and Reporting. Sample analysis is performed at an on-site or off-site analytical laboratory. Analysis results, along with supplemental information on analytical techniques, dilutions, and chain-of-custody records, are documented. Laboratory results are transferred in standard hard copy and/or in electronic formats (Sections 7, 8, 9, 10, 11, and Appendix G).

F.1.2.6 Data Verification and Validation. A set of specified, standardized rules and associated Quality Control (QC) measures is used to validate sample results and assign data qualifier flags (Appendix D).

F.1.2.7 Data Repository. The FEMP Data Management System Results Database (DMSRD) supports direct loading of validated data from electronic media as well as manual data entry. The DMSRD is maintained using relational database management software. Validated data are loaded into the data repository, which is the heart of the FEMP environmental data management system. It is what most data users consider when thinking of the environmental database.

Manual data entry shall be performed in duplicate and the two sets of entered data shall be electronically compared. Discrepancies between the two sets will be resolved by comparison to the original data sheets and corrections made as necessary to entered data.

F.1.2.8 Data Analysis. Analysis results data are retrieved or accessed to support a wide range of activities including modeling, statistics, mapping and visual display, and summary tabular data listings. Some data analyses include assessment of the useability of existing data for current applications. The assessments may lead to definition of a need for additional sampling efforts, which connects the data analysis phase of the data life cycle to data requirements and sampling plan phases.

F.1.2.9 Data Archiving and Storage. Each piece of data in the FEMP environmental DMSRD is linked to the original hard-copy documents produced by analytical laboratories. Hard copies are kept in permanent storage and the electronic database is permanently archived in a neutral ASCII file format.

F.2 FEMP ENVIRONMENTAL DATA MANAGEMENT SYSTEM

A collection of integrated environmental data management systems has been designed for FEMP to support the range of data-related activities previously outlined. These systems make up the FEMP environmental data management system to manage the complete set of sampling, project scheduling, QA/QC, and analytical results data, along with site maps and other spatially oriented data.

The data repository that stores analytical and field observation results, related QA/QC information, sampling station information, and cross references to original hard copy documents is central to the FEMP environmental data management system. Each of the other systems interface with this central repository either by using repository data as input or by serving as a data input point to the repository.

In the FEMP system, data are shared among applications, and redundant storage of a piece of data in more than one location in the repository is avoided when possible. Figure F-2 (Appendix A) shows how the various systems are integrated into the overall data management system and how they are interrelated. The following paragraphs contain brief descriptions of each of the computerized systems illustrated in Figure F-2 (Appendix A).

F.2.1 Automated Sampling and Analysis Program System

The Automated Sampling and Analysis Program system assists in reviewing data results and associated qualifiers to help identify data gaps that require additional sampling, aid in determining necessary non-routine samples, and facilitate development of PSPs for non-routine sampling. The automated sampling and analysis program includes the following subsystems.

- **Query and Report Facility** - Tailored to help identify data deficiencies and provide a profile of historical sampling efforts for user-defined locations and time frames

- **Detailed Logic Based on the SCQ and DQOs** - Produces recommendations for non-routine sampling activities when combined with the query and report facility

- **Reporting Facility** - Helps produce project-specific plans, sample analysis request/custody records, and bottle labels for non-routine sampling efforts

- **Interface to FACTS System** - Transfers data requirements identified for non-routine sampling activities and draws on Fernald Analytical Computerized Tracking System (FACTS) ORACLE data tables (subsection F.2.2) to assign unique sample identification numbers to newly required samples

APPENDIX G ANALYTICAL METHODS AND PERFORMANCE CRITERIA

G.1 INTRODUCTION 1

G.2 ABBREVIATIONS 4

Table G-1 SCQ Analytical Methods Selection Table for Organic and Inorganic Analyses

Table G-2 Organic and Inorganic Performance Criteria

Table G-3 Performance Specifications for Radiochemical Analyses

Table G-4 Radiochemical Performance Criteria

APPENDIX G

ANALYTICAL METHODS AND PERFORMANCE CRITERIA

NOTE: THIS APPENDIX IS NEW TO REVISION 0.1

G.1 INTRODUCTION

This appendix gives the methods and/or performance criteria for all analyses performed for the FEMP. Table G-1, the Methods Selection Table, lists the standard methods which may be used for organic and inorganic analyses. The performance criteria associated with the methods in Table G-1 are presented in Table G-2. Table G-3 lists radiochemical analytes and the matrices and ASLs for which there are performance criteria. Table G-4 gives the performance specifications for radiochemical analyses.

G.2 ABBREVIATIONS

AD	-	Absolute Difference
ADC	-	Analog to Digital Converter
CCB	-	Continuing Calibration Blank
CCV(S)	-	Continuing Calibration Verification (Standard)
DR	-	Data are qualified based on results, using the review and validation guidance
DUP	-	Duplicate
DWB	-	Dilution Water Blank
ECV	-	Energy Calibration Verification
EDXRF	-	Energy Dispersive X-Ray Fluorescence
FCV	-	Final Calibration Verification
GAC	-	Glucose-Glutamic Acid Check
HAMDC	-	Highest Allowable Minimum Detectable Concentration
IAP	-	Ion Abundance Pattern
IC	-	Initial Calibration
ICB	-	Initial Calibration Blank
ICS	-	Interference Check Standard
ICV(S)	-	Initial Calibration Verification (Standard)
IDL	-	Instrument Detection Limit
IS	-	Internal Standards
LCS	-	Laboratory Control Sample (second source verification)
MB	-	Method Blank
MDC	-	Minimum Detectable Concentration
MS	-	Matrix Spike
MSA	-	Method of Standard Additions
MSD	-	Matrix Spike Duplicate
PQL	-	Practical Quantitation Limit
RER	-	Relative Error Rate
RMV	-	Reference Monitor Verification
RPD	-	Relative Percent Difference
VOC	-	Volatile Organic Compounds

TABLE G-1
SCQ ANALYTICAL METHODS SELECTION TABLE
FOR ORGANIC AND INORGANIC ANALYSES

Analyte or Class of Analytes	ASL	Matrices and Methods			
		Water & Wastewater		Soil & Solids	
with performance criteria numbers		Prep Method(s)[1,2]	Analytical Method(s)	Prep Method(s)[1,2]	Analytical Method(s)
1a. VOCs	B	W	SW 846-8260	W	SW 846-8260
	C, D	W	CLP[8]	W	CLP[8]
1b. VOCs (Drinking Water)	B	W	EPA 524.2	NA	NA
2. Semi-Volatile Organic Compounds	B	SW 846-3520 or 3510[9]	SW 846-8270	SW 846-3550[10]	SW 846-8270
	C, D	W	CLP[8]	W	CLP[8]
3. Chlorinated Pesticides and PCBS	B	SW 846-3520 or 3510[9]	SW 846-8080	SW 846-3550[10]	SW 846-8080
	C, D	W	CLP[8]	W	CLP[8]
4. Organophosphorus Pesticides	B	SW 846-3520 or 3510[9]	SW 846-8140	SW 846-3550[10]	SW 846-8140
5. Herbicides	B	W	SW 846-8150	W	SW 846-8150
6. Aromatic Volatile Organics	B	SW 846-5030	SW 846-8020	SW 846-5030	SW 846-8020
7. Halogenated Volatile Organics	B	SW 846-5030	SW 846-8010	SW 846-5030	SW 846-8010
8. Purgeable Organic Halogens	B	W	SW 846-9021	W	SW 846-9021
9. Metals by GFAA	B	SW 846-3020 or 7060[6], 7740[6] or 7761[6]	SW 846-7000 series or 3500[4] series	SW 846-3050 or 7761[6]	SW 846-7000 series or 3500[4] series
	C, D	W	CLP[8]	W	CLP[8]

TABLE G-1
SCQ ANALYTICAL METHODS SELECTION TABLE
FOR ORGANIC AND INORGANIC ANALYSES (cont.)

Analyte or Class of Analytes with performance criteria numbers	ASL	Matrices and Methods: Water & Wastewater Prep Method(s)[1,2]	Water & Wastewater Analytical Method(s)	Soil & Solids Prep Method(s)[1,2]	Soil & Solids Analytical Method(s)
10. Metals by AAS (Flame)	B	SW 846-3010 or 7760[7]	SW 846-7000 series or 3500[4] series	SW 846-3050 or 7760[7]	SW 846-7000 series or 3500[4] series
	C,D	W	CLP[8]	W	CLP[8]
11. Metals by ICP	B	SW 846-3010 or 7760[7]	SW 846- 6010 or 3500[4] series	SW 846-3050 or 7760[7]	SW 846-6010 or 3500[4] series
	C, D	W	CLP[8]	W	CLP[8]
12. Mercury by Cold Vapor AAS	B	W	SW 846-7470	W	SW 846-7471
	C, D	W	CLP[8]	W	CLP[8]
13. Cyanide (Tot)	B	W	335.2[3]	W	335.2[3]
14. Cyanide (Low)	B	W	335.3[3]	W	335.3[3]
15. Soil pH	B	NA	NA	W	SW 846-9045
16. pH (electrometric)	B	W	SW 846-9040 or 4500B[4]	NA	NA
17. Nitrogen, Nitrate/Nitrite	B	W	353.1[3], 353.2[3], 4500D[4],E[4]	NA	NA
18. Conductivity	B	W	120.1[3] or 2510B[4]	NA	NA
19. TKN	B	W	351.2[3]	NA	NA
20. TOC	B	W	SW 846-9060	NA	NA
21. Alkalinity	B	W	310.1[3] or 2320B[4]	NA	NA

TABLE G-1
SCQ ANALYTICAL METHODS SELECTION TABLE
FOR ORGANIC AND INORGANIC ANALYSES (cont.)

Analyte or Class of Analytes	ASL	Matrices and Methods			
		Water & Wastewater		Soil & Solids	
with performance criteria numbers		Prep Method(s)[1,2]	Analytical Method(s)	Prep Method(s)[1,2]	Analytical Method(s)
22. Chloride	B	W	325.2[3], 300.(all)[3] or 4500B[4]	NA	NA
23. Sulfide	B	W	376.1[3] or SW 846-9030	NA	NA
24. Ammonia	B	W	350.1[3], 350.3[3], 4500C & F[4]	NA	NA
25. Hexavalent Cr	B	W	SW 846-7195	W	SW 846-7195
26. Oil & Grease	B	W	SW 846-9070	W	SW 846-9070 or 9071
27. Temperature	B	W	170.1[3]	W	170.1[3]
28. Percent Solids (Moisture)	B	W	160.3[3]	W	160.3[3]
29. TPH	B	W	418.1[3]	W	SW 846-9073
30. Total Dissolved Solids	B	W	160.1[3] or 2540C[4]	NA	NA
31. Phosphorus	B	W	365.(all)[3] or 4500E[4]	NA	NA
32. Surfactants (MBAS)	B	W	5540C[4]	NA	NA
33. Phenolics, Total Recoverable	B	W	SW 846-9065 or 9066	W	SW 846-9065 or 9066
34. Sulfate	B	W	375.2[3], 300.0[3] or 4500E[4]	NA	NA
35. Fluoride	B	W	340.2[3], 300.0[3] or 4500C[4]	NA	NA

TABLE G-1
SCQ ANALYTICAL METHODS SELECTION TABLE
FOR ORGANIC AND INORGANIC ANALYSES (cont.)

Analyte or Class of Analytes	ASL	Matrices and Methods			
		Water & Wastewater		Soil & Solids	
with performance criteria numbers		Prep Method(s)[1,2]	Analytical Method(s)	Prep Method(s)[1,2]	Analytical Method(s)
36. Total Organic Halides	B	W	SW 846-9020	NA	NA
37. Color	B	W	110.2[3]	NA	NA
38. Red/Ox Potential	B	W	ASTM-1498	NA	NA
39. Total Suspended Solids	B	W	160.2[3] or 2540D[4]	NA	NA
40. Paint Filter Test	B	W	SW 846-9095	W	SW 846-9095
41. COD	B	W	5220D[4]	NA	NA
42. BOD_5 & $CBOD_5$	B	W	5210B[4]	NA	NA
43. Total Fecal Coliforms	B	W	9222D[4]	NA	NA
44. Reactivity	B	W	SW 846-parts 7.3.3 & 7.3.4	W	SW 846- parts 7.3.3 & 7.3.4
45. Corrosivity	B	W	SW 846-1110	W	SW 846-1110
46. Ignitability	B	W	SW 846-1010	W	SW 846-1010
47. Sulfide, Extractable	B	W	SW 846-9031	W	SW 846-9031
48. U & Th in Soil by EDXRF	B	W	EPM 9011[5]	W	EPM 9011[5]
49. U & Th in Concrete by EDXRF	B	W	EPM 7004[5]	W	EPM 7004[5]
50. Thorium, Low Level	B	W	EPM 1080[5], 3059[5], 3063[5]	W	EPM 1080[5], 3059[5], 3063[5]
51. Uranium, Low (ppm) Level	B	W	EPM 3002[5]	W	EPM 3002[5]

TABLE G-1
SCQ ANALYTICAL METHODS SELECTION TABLE
FOR ORGANIC AND INORGANIC ANALYSES (cont.)

Analyte or Class of Analytes	ASL	Matrices and Methods			
		Water & Wastewater		Soil & Solids	
with performance criteria numbers		Prep Method(s)[1,2]	Analytical Method(s)	Prep Method(s)[1,2]	Analytical Method(s)[1,2]
52. Uranium, High Level	B	W	EPM 1039[5]	W	EPM 1039[5]
53. Semi-Quant. Analysis by EDXRF	B	W	EPM 9025[5]	W	EPM 9025[5]
54. Total Hardness	B	W	2340C[4]	NA	NA
55. Methanol by GC	B	W	EPM 2002[5]	W	EPM 2002[5]
56. Dioxins by GC/MS	B	W	SW 846-8280	W	SW 846-8280

1 SW 846-1311 (TCLP) could be a prep, however, it is not necessary in all cases.
2 "W" signifies that the preparation is contained within the analytical method.
3 *Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020.
4 *Standard Methods for the Analysis of Water and Wastewater, 17th ed.* These methods are used for NPDES analyses.
5 *FEMP Environmental Process Monitoring Lab Method.*
6 7060 contains the preparation for As, 7740 for Se, and 7761 for Ag.
7 7760 contains the preparation for Ag.
8 USEPA Contract Laboratory Program Statement of Work, most recent revision.
9 SW 846-3520 is the preferred method, however, some foamy or small samples may require the use of Method 3510.
10 SW 846-3550 is used for uniform soil samples. SW 846-3540 is recommended for special matrices (e.g. oil soaked soil, etc.).

Table G-2

Criterion: 1a
PROTOCOL: SW-846
METHOD: GC/MS for Volatile Organics (8260, Dec. 1987)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IAP	Start each 12 hr. period	Per method Table 4	Retune
2. IC	Begin, following tune	Per method Section 7.3	Recalibrate
3. CCV	Every 12 hrs, following tune	Per method Section 7.3.4	Recalibrate
4. LCS	Begin	Per method 8240[1]	Recalibrate
5. MB	Each batch	< PQL	Reanalyze
6. MS/MSD	Every 20 samples	Per CLP SOW[2]	Advisory
7. Surrogates	All samples	Per method Table 9	Reanalyze
8. IS	All samples	Per method Section 7.3.5	Reanalyze

9. Detection limits ..... Per method tables 1,2 & 3

10. Analyte lists ..... Per method table 1

11. Standards concentrations
    IAP ..... Per method section 5.11
    IS ..... Per method sections 5.10 and 7.4.5
    MS ..... Per method section 5.13
    Surrogate ..... Per method section 5.9

12. Calibration points & ranges
    ICV ..... Per method section 5.12
    CCV ..... Per method section 7.2.8

(1) Since Method 8260 has no limits, SW 846 Method 8240 (Sept. 1986), Table 6, is used as guidance.
(2) Since Method 8260 has no limits, USEPA OLM01.0, page D-50/VOA, Table 7, is used as guidance.

Table G-2 (cont.)

Criterion: 1b
PROTOCOL: U.S. EPA
METHOD: GC/MS for Volatile Organics, Drinking Water (524.2, Revision 3)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IAP	Start each 8 hr. period	Per method Table 3	Retune
2. IC	Begin, following tune	Per method Section 9.2	Recalibrate
3. CCV	Every 8 hrs, following tune	Per method Section 9.3	Recalibrate
4. LCS	Begin and each batch	Per method section 10.6	Recalibrate
5. MB	Each batch	< PQL	Reanalyze
6. Surrogates	All samples	Per method Section 10.4	Reanalyze
7. IS	All samples	Per method Section 9.3.4	Reanalyze

8. Detection limits ..... Per method table 4

9. Analyte lists ..... Per method table 1

10. Standards concentrations
    IAP ..... Per method section 7.5
    IS ..... Per method section 7.5
    Surrogate ..... Per method section 7.5

11. Calibration points & ranges
    ICV ..... Per method section 7.8
    CCV ..... Per method section 9.3.2

Table G-2 (cont.)

Criterion: 2
PROTOCOL: SW-846
METHOD: GC/MS for Semivolatile Organics (8270, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IAP	Start each 12 hr. period	Per method Table 3.	Retune
2. IC	Begin, following tune	Per method Section 7.3.4	Recalibrate
3. CCV	Every 12 hrs, following tune	Per method Section 7.4.4	Recalibrate
4. LCS	Begin	Per method Table 6	Recalibrate
5. MB	Each batch	< PQL	Reanalyze
6. MS/MSD	Every 20 samples	Per method Sections 8.6.2 & 8.6.3	Rerun LCS
7. Surrogates	All samples	Per method Table 8	Reanalyze
8. IS	All samples	Per method Section 7.4.5	Reanalyze

9. Detection limits ..... Per method table 2

10. Analyte lists ..... Per method table 1

11. Standards concentrations
    IAP ..... Per method section 5.3
    IS ..... Per method sections 5.2 and 7.4.5
    MS ..... Per method section 5.6
    Surrogate ..... Per method section 5.5

12. Calibration points & ranges
    ICV ..... Per method section 5.4
    CCV ..... Per method section 7.4.2

Table G-2 (cont.)

Criterion: 3
Protocol: SW-846
METHOD: GC for Organochlorine Pesticides and PCB's (8080, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Degradation check sample	Start each 12 hours	Per method Section 7.4.5	Reanalyze
2. IC	Begin	Per method Section 8.2.2	Recalibrate
3. CCV	1/10 samples and end of sequence	Per method Section 8.2.2	Recalibrate
4. LCS	Begin	Per method Table 3	Recalibrate
5. MB	Each batch	< PQL	Reanalyze
6. MS/MSD	Every 20 samples	Per CLP SOW[1]	Advisory
7. Surrogates	All samples	Per method Section 8.3	Reanalyze

8. Detection limits ..... Per method table 1

9. Analyte lists ..... Per method table 1

10. Standards concentrations
    MS ..... Per CLP SOW[2]
    Surrogate ..... Per method Section 5.5

11. Calibration points & ranges
    ICV ..... Per method section 5.3
    CCV ..... Per method section 7.3.1

(1) Since Method 8080 has no limits, USEPA OLM01.0, section 16.4, page D-58/Pest is used as guidance.
(2) Since Method 8080 has no concentrations, USEPA OLM01.0, section 4.9.5, page D-14/Pest is used as guidance.

Table G-2 (cont.)

Criterion: 4
PROTOCOL: SW-846
METHOD: GC for Organophosphorus Pesticides (8140, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Begin	Per method Section 7.3	Recalibrate
2. CCV	1/10 samples and end of sequence	Per method Section 7.3	Recalibrate
3. LCS	Begin	60-150%	Recalibrate
4. MB	Each batch	< PQL	Reanalyze
5. MS/MSD	Every 20 samples	60-150%	Advisory
6. Surrogates	All samples	60-150%	Reanalyze

7. Detection limits ..... Per method table 2

8. Analyte lists ..... Per method table 1

9. Standards concentrations
   IS ..... Per method section 5.4
   MS ..... Per method section 8.2
   Surrogate ..... Per method section 5.5

10. Calibration points & ranges
   ICV ..... Per method section 5.3
   CCV ..... Per method section 7.3

Table G-2 (cont.)

Criterion: 5
PROTOCOL: SW-846
METHOD: GC for Chlorinated Herbicides (8150, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Begin	Per method Section 7.5	Recalibrate
2. CCV	1/10 samples and end of sequence	Per method Section 7.5	Recalibrate
3. LCS	Begin	60-150%	Recalibrate
4. MB	Each batch	< PQL	Reanalyze
5. MS/MSD	Every 20 samples	40-150%	Reanalyze
6. Surrogates	All samples	60-150%	Reanalyze

7. Detection limits ..... Per method tables 1 & 2

8. Analyte lists ..... Per method table 1

9. Standards concentrations
   IS ..... Per method section 5.12
   MS ..... Per method section 8.2.1
   Surrogate ..... Per method section 5.5

10. Calibration points & ranges
   ICV ..... Per method section 5.11
   CCV ..... Per method section 7.5

Table G-2 (cont.)

Criterion: 6
PROTOCOL: SW-846
METHOD: GC for Aromatic Volatile Organics (8020, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Begin	Per method Section 7.3	Recalibrate
2. CCV	1/10 samples and end of sequence	Per method Section 7.3	Recalibrate
3. LCS	Begin	Per method Table 3	Recalibrate
4. MB	Each batch	< PQL	Reanalyze
5. MS/MSD	Every 20 samples	Per method Section 8.2	Advisory
6. Surrogates	All samples	80-120%	Reanalyze

7. Detection limits ..... Per method tables 1 & 2

8. Analyte lists ..... Per method table 1

9. Standards concentrations
   IS ..... Per method section 5.5
   MS ..... Per method section 8.2
   Surrogate ..... Per method section 5.6

10. Calibration points & ranges
   ICV ..... Per method section 5.4
   CCV ..... Per method section 7.3

Table G-2 (cont.)

Criterion: 7
PROTOCOL: SW-846
METHOD: GC for Halogenated Volatile Organics (8010, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Begin	Per method Section 7.3	Recalibrate
2. CCV	1/10 samples and end of sequence	Per method Section 7.3	Recalibrate
3. LCS	Begin	Per method Table 3	Recalibrate
4. MB	Each batch	< PQL	Reanalyze
5. MS/MSD	Every 20 samples	Per method Section 8.2	Advisory
6. Surrogates	All samples	80-120%	Advisory

7. Detection limits ..... Per method table 1

8. Analyte lists ..... Per method table 1

9. Standards concentrations
   IS ..... Per method section 5.5
   MS ..... Per method section 8.2
   Surrogate ..... Per method section 5.6

10. Calibration points & ranges
   ICV ..... Per method section 5.4
   CCV ..... Per method section 7.3

Table G-2 (cont.)

Criterion: 8
PROTOCOL: SW-846
METHOD: Purgeable Organic Halogen (9021, Dec. 1987)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Begin	Per method Section 7.1	Recalibrate
2. CCV	Every 12 hrs	Per method Section 7.1	Recalibrate
3. LCS	Begin	80-120%	Recalibrate
4. MB	Each batch	< PQL	Reanalyze
5. MS/MSD	Every 10 samples	75-125%	Advisory

6. Detection limits ..... Per method section 9.1

7. Standards concentrations ..... Per method section 5

8. Calibration points & ranges ..... Per method section 7.1

Table G-2 (cont.)

Criterion: 9
PROTOCOLS: SW-846 & Standard Methods for the Analysis of Water and Wastewater
METHOD: METALS BY GFAA (7000 series and 3500 series, respectively)
ASLs: B ONLY

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Beginning	80-120%	Recalibrate
2. CCVS	Every 10 Samples and at end	80-120%	Recalibrate
3. ICB/CCB	With ICVS/CCVS	± 3 Std. Dev.	Recalibrate/ Reanalyze last 10
4. Method Blank	Each Batch	<PQL	Redigest Batch
5. LCS	1/20 or 1/batch	80-120%	Redigest Batch
6. Duplicate	Each Matrix	RPD <20% for samples > 10X IDL	Redigest
7. MS	Each Matrix	75-125%	Redigest or Post Digestion Spike or MSA

8. Reporting Limits..... Per method 7000, table 1

Table G-2 (cont.)

Criterion: 10
PROTOCOLS: SW-846 & Standard Methods for the Analysis of Water and Wastewater
METHOD: METALS BY FLAA (7000 series and 3500 series, respectively)
ASLs: B ONLY

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Beginning	80-120%	Recalibrate
2. CCVS	Every 10 Samples and at end	80-120%	Recalibrate
3. ICB/CCB	With ICVS/CCVS	± 3 Std. Dev.	Recalibrate/ Reanalyze last 10
4. Method Blank	Each Batch	<PQL	Redigest Batch
5. LCS	1/20 or 1/batch	80-120%	Redigest Batch
6. Duplicate	Each Matrix	RPD <20% for samples > 10X IDL	Redigest
7. MS	Each Matrix	75-125%	Redigest or Post Digestion Spike or MSA

8. Detection Limits..... Per method 7000, table 1

Table G-2 (cont.)

Criterion: 11
PROTOCOLS: SW-846 & Standard Methods for the Analysis of Water and Wastewater
METHOD: ICP-AES (6010 or 3500 series)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90% - 110%	Recalibrate
2. CCVS	Every 10 and at end	90% - 110%	Recalibrate
3. MB	Each batch	< PQL	Redigest batch
4. ICB/CCB	With ICVS/CCVS	± 3 Std. Dev.	Recalibrate/ Reanalyze last 10
5. ICS	Begin & end or every 8 hrs.	80% - 120%	Reexamine background/ reanalyze
6. LCS	1/20 or 1/batch	80-120%	Redigest Batch
7. MS	Each matrix	75% - 125%	Redigest or Post Digestion Spike or MSA
8. DUP	1/20 samples	RPD < 20% for samples 10 x IDL	DQO driven

9. Detection Limits.....Per method 6010, table 1

Table G-2 (cont.)

Criterion: 12
PROTOCOL: SW-846
METHOD: Mercury by Cold Vapor AAS (7470 or 7471)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90% - 110%	Recalibrate
2. CCVS	1/10 and at end	90% - 110%	Recalibrate
3. MB	Each batch	< PQL	Redigest batch
4. ICB/CCB	With ICVS/CCVS	± 3 Std. Dev.	Recalibrate/ Reanalyze last 10
5. LCS	1/20 or 1/batch	80-120%	Redigest batch
6. MS/MSD	1/10 or each matrix	75% - 125% (MS)	Redigest or M.S.A.
7. Duplicate	1/20	<20% RPD for samples at 10 x IDL	DQO driven

8. Detection Limits.....0.0002 mg/L

9. Standards Concentrations.....Per section 7.2 of 7470 or section 7.3 of 7471

Table G-2 (cont.)

Criterion: 13
PROTOCOL: EPA-600/4-79-020
METHOD: Cyanide, Total (335.2)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	85-115%	Recalibrate
2. CCVS	1/20	85-115%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....1.0 mg/L

Criterion: 14
PROTOCOL: EPA-600/4-79-020
METHOD: Cyanide, Low Level (335.3)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	85-115%	Recalibrate
2. CCVS	1/20	85-115%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....0.005 mg/L

Table G-2 (cont.)

Criterion: 15
PROTOCOL: SW-846
METHOD: Soil pH (9045)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Reanalyze samples since last CCVS
3. Duplicate	1/20	0-20% RPD	Qualify data

Criterion: 16
PROTOCOL: SW-846 or Standard Methods for the Analysis of Water and Wastewater
METHOD: pH, Electrometric (9040 or 4500-H^+ B)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Reanalyze samples since last CCVS
3. Duplicate	1/20	0.2 pH units	Qualify data

Table G-2 (cont.)

Criterion: 17
PROTOCOL: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Nitrogen, Nitrate/Nitrite (353.1, 353.2, 4500-NO_3 D or 4500-NO_3 E)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/10	90-110%	Recalibrate and reanalyze all since last CCVS
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....0.01 mg/L

Criterion: 18
PROTOCOLS: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHODS: Conductivity (120.1 or 2510B)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Reanalyze samples since last CCVS
3. Cell Constant	1/20	between 1 and 2	Recalibrate
4. Duplicate	1/20	0-20% RPD	Qualify data

Table G-2 (cont.)

Criterion: 19
PROTOCOL: EPA-600/4-79-020
METHOD: Total Kjeldahl Nitrogen (351.2)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....0.1 mg/L

Criterion: 20
PROTOCOL: SW-846
METHOD: Total Organic Carbon (9060)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	80-120%	Recalibrate
2. CCVS	1/15	80-120%	Reanalyze samples since last CCVS
3. Method Blank	1/20	DR	Qualify data
4. MS/MSD	1/10	75-125%	Qualify data
5. Duplicate	1/20	0-20% RPD	Qualify data

6. Detection Limit..... 1.0 mg/L

Table G-2 (cont.)

Criterion: 21
PROTOCOLS: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHODS: Alkalinity (310.1 or 2320B)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. LCS	1/20	80-120%	Reanalyze samples since last LCS
2. Duplicate	1/20	0-20% RPD	Qualify data

3. Detection Limit.....10 mg/L as $CaCO_3$

Criterion: 22
PROTOCOLS: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Chloride (300.0, 300.1, 300.2, 325.2 or 4500-Cl B)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....1.0 mg/L

Table G-2 (cont.)

Criterion: 23
PROTOCOL: EPA-600/4-79-020 or SW-846
METHOD: Sulfide (376.1 or 9030)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/15	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/10	0-20% RPD	Qualify data

7. Detection Limit.....1.0 mg/L

Criterion: 24
PROTOCOLS: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Ammonia (350.1, 350.3, 4500-NH_3 C or F)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Duplicate	1/20	0-20% RPD	Qualify data

6. Detection Limit.....0.1 mg/L

Table G-2 (cont.)

Criterion: 25
PROTOCOL: SW-846
METHOD: Hexavalent Chromium (7195)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	80% - 120%	Recalibrate
2. CCVS	1/10 and at end	80% - 120%	Recalibrate and Reanalyze samples since last CCVS
3. ICB/CCB	With ICVS/CCVS	± 3 Std. Dev.	Recalibrate/ Reanalyze last 10
4. Method Blank	Each batch	< PQL	Redigest batch
5. LCS	1/20	80-120%	Recalibrate/ Redigest batch
6. MS/MSD	1/10 or each matrix	75% - 125% (MS)	Redigest or M.S.A.
7. Duplicate	1/20	<20% RPD for samples at 10 x IDL	DQO driven

8. Detection Limits.....10 μg/L

Table G-2 (cont.)

Criterion: 26
PROTOCOL: SW-846
METHOD: Oil and Grease (9070 or 9071)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. LCS	1/20	80-120%	Recalibrate
2. Method Blank	1/20	DR	Qualify data
3. MS/MSD (soil only)	1/10	75-125%	Qualify data
4. DUP (soil only)	1/20	0-20% RPD	Qualify Data

5. Detection Limit.....0.1 mg/L

Criterion: 27
PROTOCOL: EPA-600/4-79-020
METHOD: Temperature (170.1)
ASLs: B only

REQUIREMENT

Quality control requirements are determined by the corresponding analytical methods or the project specific plan.

Criterion: 28
PROTOCOL: EPA-600/4-79-020
METHOD: Percent Solids (Moisture) (160.3)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Method Blank	1/20	DR	Qualify data
2. Duplicate	1/20	0-20% RPD	Qualify data

3. Detection Limit.....10 mg/L

Table G-2 (cont.)

Criterion: 29
PROTOCOLS: EPA-600/4-79-020 or SW-846
METHOD: Total Petroleum Hydrocarbons (418.1 or 9073)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	Per Method Section 6.5	Recalibrate
2. CCVS	Every 12 Hours	Per Method Section 6.5	Recalibrate/ Reanalyze all samples since last CCVS
3. LCS	1/20	80-120%	Reanalyze
4. Method Blank	Each batch	<PQL	Reanalyze
5. MS/MSD (soil)	1/20	75-125%	Advisory
6. DUP (soil)	1/20	0-20% RPD	Qualify data

7. Detection Limit.....1.0 mg/L

Criterion: 30
PROTOCOLS: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHODS: Total Dissolved Solids (160.1 or 2540C)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Method Blank	1/20	DR	Qualify data
2. Duplicate	1/20	0-20% RPD	Qualify data

3. Detection Limit.....10 mg/L

Table G-2 (cont.)

Criterion: 31
PROTOCOL: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Phosphorus (365.1, 365.2, 365.3, 365.4 or 4500-P E)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....0.05 mg/L

Criterion: 32
PROTOCOL: Standard Methods for the Analysis of Water and Wastewater
METHOD: Surfactants (MBAS) (5540C)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	80-120%	Recalibrate
2. CCVS	1/20	80-120%	Reanalyze samples since last CCVS
3. Method Blank	1/20	DR	Qualify data
4. Duplicate	1/20	0-20% RPD	Qualify data

5. Detection Limit.....0.025 mg/L calculated as LAS

Table G-2 (cont.)

Criterion: 33
PROTOCOL: SW-846
METHOD: Phenolics, Total Recoverable (9065 or 9066)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/15	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....5.0 μg/L

Criterion: 34
PROTOCOL: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Sulfate (300.0, 375.2, or 4500-SO_4 E)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....1.0 mg/L

Table G-2 (cont.)

Criterion: 35
PROTOCOL: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Fluoride (300.0, 340.2 or 4500-F C)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Reanalyze samples since last CCVS
3. Method Blank	1/20	DR	Qualify data
4. Matrix Spike	1/20	75-125%	Qualify data
5. Duplicate	1/20	0-20% RPD	Qualify data

6. Detection Limit.....0.01 mg/L

Criterion: 36
PROTOCOL: SW-846
METHOD: Total Organic Halides (9020)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	Per Method Section 7.2	Recalibrate
2. CCVS	1/10 pyrolyses	Per Method Section 7.2	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	Each batch	<PQL	Reanalyze
5. MS/MSD	1/15	75-125%	Qualify data
6. Duplicate	1/15	0-20% RPD	Qualify data

7. Detection Limit.....5.0 μg/L

Table G-2 (cont.)

Criterion: 37
PROTOCOL: EPA-600/4-79-020
METHOD: Color (110.2)
ASLs: B only

REQUIREMENT

Since Color is a semiquantitative measure, it is not necessary to analyze QC samples. Duplicate analyses are of little value since the sample result is based on visual comparison and is subject to individual variability.

Criterion: 38
PROTOCOL: ASTM
METHOD: Oxidation/Reduction Potential (D-1498)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Recalibrate
3. Duplicate	1/20	0-20% RPD	Qualify data

Table G-2 (cont.)

Criterion: 39
PROTOCOLS: EPA-600/4-79-020 or
Standard Methods for the Analysis of Water and Wastewater
METHOD: Total Suspended Solids (160.2 or 2540D)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Method Blank	1/20	DR	Qualify data
2. Duplicate	1/20	0-20% RPD	Qualify data

3. Detection Limit..... 10 mg/L

Criterion: 40
PROTOCOL: SW-846
METHOD: Paint Filter Test (9095)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Duplicate	1/20	Results must agree	Qualify data

Criterion: 41
PROTOCOL: Standard Methods for the Analysis of Water and Wastewater
METHOD: Chemical Oxygen Demand (COD) (5220D)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/20	90-110%	Reanalyze samples since last CCVS
3. Method Blank	1/20	DR	Qualify data
4. Duplicate	1/20	0-20% RPD	Qualify data
5. LCS	1/20	80-120%	Redigest & Reanalyze

6. Detection Limit.....5 mg/L COD

Table G-2 (cont.)

Criterion: 42
PROTOCOL: Standard Methods for the Analysis of Water and Wastewater
METHOD: Five Day Biochemical Oxygen Demand (BOD_5 and $CBOD_5$) (5210B)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. DWB	1/batch	<0.2 mg/L	Qualify data
2. GAC	1/batch	200±37 mg/L	Qualify data
3. Method Blank	1/20	DR	Qualify data
4. Duplicate	1/20	0-20% RPD	Qualify data

5. Detection Limit.....1.0 mg/L

Criterion: 43
PROTOCOL: Standard Methods for the Analysis of Water and Wastewater
METHOD: Total Fecal Coliforms (9222D)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Method Blank	1/20	DR	Qualify data
2. Duplicate	1/20	0-20% RPD	Qualify data

Table G-2 (cont.)

Criterion: 44
PROTOCOL: SW-846
METHOD: Reactivity (parts 7.3.3 and 7.3.4)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. LCS	1/20	>50%	Reanalyze batch
2. Method Blank	1/20	DR	Qualify data
3. Duplicate	1/20	0-20% RPD	Qualify data

Criterion: 45
PROTOCOL: SW-846
METHOD: Corrosivity (1110)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Duplicate	Every Sample	0-20% RPD	Qualify data

Table G-2 (cont.)

Criterion: 46
PROTOCOL: SW-846
METHOD: Ignitability (1010)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. Duplicate	1/20	0-20% RPD	Qualify data
2. Xylene Std.	1/20	Per method	Qualify data

Criterion: 47
PROTOCOL: SW-846
METHOD: Sulfide, Extractable (9031)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. LCS	1/20	80-120%	Reanalyze samples since last LCS
2. Method Blank	1/20	DR	Qualify data
3. Matrix Spike	1/20	75-125%	Qualify data
4. Duplicate	1/20	0-20% RPD	Qualify data

5. Detection Limits.....Liquids, 1.0 mg/L; Solids, 1.0 mg/kg

Table G-2 (cont.)

Criterion: 48
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: URANIUM & THORIUM IN SOIL BY EDXRF (9011)
ASLs: B ONLY

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICV	Beginning	90-110%	Recalibrate
2. MB	Each Batch	<IDL	Check for cross contamination; Regrind & press, then rerun batch
3. LCS	Each Batch	80-120%	Check for cross contamination; Reanalyze batch
4. DUP	Each Batch	RPD <20% for samples > 10X IDL	Void batch analysis due to inhomogeneity and submit to be run by other method
5. MS	Each Batch	75-125%	Void batch analysis due to possible matrix problem and submit to be ran by other method
6. FCV	Batch End	90-110%	Recalibrate
7. ECV	Weekly	Fe K-alpha 6.40 +/- 0.01 keV Mo K-alpha 17.44 +/- 0.02 keV	Recalibrate ADC
8. RMV	Weekly	95-105%	Perform X-ray tube stability test. Establish reference monitor ratio correction factor and input into calibration equations

Table G-2 (cont.)

Criterion: 49
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: URANIUM & THORIUM IN CONCRETE BY EDXRF (7004)
ASLs: B ONLY

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICV	Beginning	90-110%	Recalibrate
2. MB	Each Batch	<IDL	Check for cross contamination; Regrind & press, then rerun batch
3. LCS	Each Batch	80-120%	Check for cross contamination; Reanalyze batch
4. DUP	Each Batch	RPD <20% for samples > 10X IDL	Void batch analysis due to inhomogeneity and submit to be run by other method
5. MS	Each Batch	75-125%	Void batch analysis due to possible matrix problem and submit to be ran by other method
6. FCV	Batch End	90-110%	Recalibrate
7. ECV	Weekly	Fe K-alpha 6.40 +/- 0.01 keV Mo K-alpha 17.44 +/- 0.02 keV	Recalibrate ADC
8. RMV	Weekly	95-105%	Perform X-ray tube stability test. Establish reference monitor ratio correction factor and input into calibration equations

Table G-2 (cont.)

Criterion: 50
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: THORIUM, LOW LEVEL (1080, 3059, and 3063)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/10	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit . . . . . 45 ppm

Criterion: 51
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: URANIUM, LOW (ppm) LEVEL (3002)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/10	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate	1/20	0-20% RPD	Qualify data

7. Detection Limit.....0.1 mg/L, 1 ppm

Table G-2 (cont.)

Criterion: 52
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: URANIUM, HIGH LEVEL (1039)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ICVS	Begin	90-110%	Recalibrate
2. CCVS	1/10	90-110%	Recalibrate
3. LCS	1/20	80-120%	Reanalyze samples since last LCS
4. Method Blank	1/20	DR	Qualify data
5. Matrix Spike	1/20	75-125%	Qualify data
6. Duplicate - solids	1/20	AD < 1%	Qualify data
7. Duplicate - liquids	1/20	AD < 5 g/L	Qualify data

8. Detection Limit..... Solids, 1.00%; Liquids, 10.0 g/L

Criterion: 53
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: QUALITATIVE/SEMI-QUANTITATIVE ANALYSIS OF UNKNOWNS BY EDXRF (9025)
ASLs: B ONLY

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. ECV	Weekly	Fe K-alpha	Recalibrate ADC 6.40 +/- 0.01 keV Mo K-alpha 17.44 +/- 0.02 keV
2. RMV	Weekly	95-105%	Perform X-ray tube stability test. Establish reference monitor ratio and correction factor input into calibration equations

Table G-2 (cont.)

Criterion: 54
PROTOCOL: Standard Methods for the Analysis of Water and Wastewater
METHOD: Total Hardness (2340C)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. LCS	1/20	80-120%	Reanalyze samples since last LCS
2. Duplicate	1/20	0-20% RPD	Qualify data

Criterion: 55
PROTOCOL: FEMP ENVIRONMENTAL PROCESS MONITORING LAB
METHOD: Methanol by GC (2002)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Weekly	90-110%	Recalibrate
2. CCV	Begin and end shift	90-110%	Recalibrate
3. LCS	1/shift	Determined by QC	Reanalyze
4. MB	1/shift	< PQL	Reanalyze

5. Detection limits ..... 10 mg/L

6. Standards concentrations ..... Per method section 4.2

7. Calibration points & ranges
   IC ..... Per method section 8.2
   CCV ..... Per method sections 11.1, 11.2

Table G-2 (cont.)

Criterion: 56
PROTOCOL: SW-846
METHOD: GC/MS for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans (8280, Sept. 1986)
ASLs: B only

REQUIREMENT	FREQUENCY	ACCEPTANCE LEVELS	CORRECTIVE ACTION
1. IC	Begin	Per method Sections 6.3-6.7	Recalibrate
2. CCV	Every 12 hrs	Per method section 6.9	Recalibrate
3. LCS	As provided	Per method section 7.3	Recalibrate
4. MB	Each batch	< PQL	Reanalyze
5. Duplicates	Every 20	Per method section 7.5	Advisory
6. IS	All samples	Per method section 10.5	Reanalyze

7. Detection limits ..... Per method section 1.2

8. Analyte lists ..... Per method table 1

9. Standards concentrations
   IS ..... Per method section 9.1

10. Calibration points & ranges
    ICV ..... Per method section 6.2
    CCV ..... Per method section 6.9

TABLE G-3

HIGHEST ALLOWABLE MINIMUM DETECTABLE CONCENTRATIONS (HAMDCs[4]) FOR RADIOCHEMICAL ANALYSES

Analyte or Class of Analytes (with performance criterion numbers)	ASL	MATRICES[3] Water pCi/L	Fly Ash pCi/g	Soil/Sediment pCi/g	Air Filters pCi/ filter	Contaminated Liquid[1] pCi/L
1. U234, U235/236, U-238	B	0.4	0.4	0.2	9.0	1.0
2. U-234, U-235/236, U-238	C, D	0.2	0.2	0.1	4.0	0.5
3. Th-227, Th-228, Th-230, Th-232	B	0.4	0.4	0.2	9.0	1.0
4. Th-227, Th-228, Th-230, Th-232	C, D	0.2	0.2	0.1	4.0	0.5
5. Pu-238, Pu-239/240	B	1.0	0.4	0.4	4.0	1.0
6. Pu-238, Pu-239/240	C, D	0.5	0.2	0.2	2.0	0.5
7. Np-237	B	1.0	0.4	0.4	4.0	1.0
8. Np-237	C, D	0.5	0.2	0.2	2.0	0.5
9. Po-210	B	1.0	0.4	0.4	4.0	1.0
10. Po-210	C, D	0.5	0.2	0.2	2.0	0.5
11. Am-241	B	2.0	2.0	0.4	11	11
12. Am-241	C, D	1.0	1.0	0.2	5.0	5.0
13. Ra-226	B	2.0	0.4	0.6	4.0	2.0
14. Ra-226	C, D	1.0	0.2	0.25	2.0	1.0

TABLE G-3 (cont.)

HIGHEST ALLOWABLE MINIMUM DETECTABLE CONCENTRATIONS (HAMDCs[4])
FOR RADIOCHEMICAL ANALYSES

Analyte or Class of Analytes (with performance criterion numbers)	ASL	MATRICES[3] Water pCi/L	Fly Ash pCi/g	Soil/Sediment pCi/g	Air Filters pCi/ filter	Contaminated Liquid[1] pCi/L
15. Ra-228	B	7.0	1.0	1.0	11	7.0
16. Ra-228	C,D	3.0	0.5	0.5	5.0	3.0
17. Pu-241	B	1.0	1.0	0.2	11	2.0
18. Pu-241	C,D	0.5	0.5	0.1	5.0	1.0
19. Pb-210	B	7.0	2.0	1.0	20	20
20. Pb-210	C,D	3.0	1.0	0.5	10	10
21. Sr-90	B	2.0	1.0	1.0	4.0	2.0
22. Sr-90	C, D	1.0	0.5	0.5	2.0	1.0
23. Tc-99	B	30	20	2.0	110	70
24. Tc-99	C, D	15	10	1.0	50	30
25. Cs-137	B	9.0	2.0	0.4	20	20
26. Cs-137	C, D	4.0	1.0	0.2	10	10
27. Total U[2] (laser)	B	0.5 µg/L	0.5 µg/g	0.5 µg/g	10 µg/ filter	5.0 µg/L
28. Total U[2] (laser)	C, D	0.1 µg/L	0.1 µg/g	0.1 µg/g	2.0 µg/ filter	1.0 µg/L
29. Gross Alpha	B	2.0	N/A	15	4.0	N/A
30. Gross Beta	B	4.0	N/A	30	30	N/A

1 Contaminated liquids are two phased systems containing about 90% water and 10% organic liquid.
2 Note the different units for this analyte.
3 Does not account for waste matrices. Performance criteria for waste matrices will be specified through the DQO process, documented in the PSP, and treated as ASL E.
4 The HAMDCs are representative values derived from input from seven radiochemical laboratories for routine operating conditions. These values may be refined, pending EPA review, on the basis of measurements of these or other laboratories on FEMP matrices under actual operating conditions.

Table G-4

Criterion: 1

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: U-234, U-235/236, U-238

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.4 pCi/L	0.2 pCi/g	9.0 pCi/Filter	0.4 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	85-115%	85-115%	85-115%	85-115%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 2

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: U-234, U-235/236, U-238	SAMPLE MATRIX				
PERFORMANCE PARAMETERS	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.2 pCi/L	0.1 pCi/g	4.0 pCi/Filter	0.2 pCi/g	0.5 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	85-115%	85-115%	85-115%	85-115%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} \cdot \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 3

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Th-227, 228, 230, 232

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.4 pCi/L	0.2 pCi/g	9.0 pCi/Filter	0.4 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	85-115%	85-115%	85-115%	85-115%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} \cdot \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 4

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Th-227, 228, 230, 232

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.2 pCi/L	0.1 pCi/g	4.0 pCi/Filter	0.2 pCi/g	0.5 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	85-115%	85-115%	85-115%	85-115%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} \cdot \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 5

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Pu-238, 239/240

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	1.0 pCi/L	0.4 pCi/g	4.0 pCi/Filter	0.4 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	85-115%	85-115%	85-115%	85-115%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 6

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Pu-238, 239/240

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.5 pCi/L	0.2 pCi/g	2.0 pCi/Filter	0.2 pCi/g	0.5 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	85-115%	85-115%	85-115%	85-115%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} + \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 7

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Np-237

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	1.0 pCi/L	0.4 pCi/g	4.0 pCi/Filter	0.4 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	85-115%	85-115%	85-115%	85-115%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 8

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Np-237

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.5 pCi/L	0.2 pCi/g	2.0 pCi/Filter	0.2 pCi/g	0.5 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	85-115%	85-115%	85-115%	85-115%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(6)

(1) $MDC = \frac{4.65\ SBLK}{K} \cdot \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 9

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Po-210

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	1.0 pCi/L	0.4 pCi/g	4.0 pCi/Filter	0.4 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	85-115%	85-115%	85-115%	85-115%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} \cdot \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 10

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Po-210

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.5 pCi/L	0.2 pCi/g	2.0 pCi/Filter	0.2 pCi/g	0.5 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	85-115%	85-115%	85-115%	85-115%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} + \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

00258

4562-

Table G-4 (cont.)

Criterion: 11

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Am-241

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	2.0 pCi/L	0.4 pCi/g	11 pCi/Filter	2.0 pCi/g	11 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	85-115%	85-115%	85-115%	85-115%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 12

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Am-241

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	1.0 pCi/L	0.2 pCi/g	5.0 pCi/Filter	1.0 pCi/g	5.0 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	85-115%	85-115%	85-115%	85-115%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} + \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

00260

- 4562-

Table G-4 (cont.)

Criterion: 13

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Ra-226

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	2.0 pCi/L	0.6 pCi/g	4.0 pCi/Filter	0.4 pCi/g	2.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	75-125%	75-125%	75-125%	75-125%	75-125%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

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4562

Table G-4 (cont.)

Criterion: 14

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Ra-226

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	1.0 pCi/L	0.25 pCi/g	2.0 pCi/Filter	0.2 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	75-125%	65-130%	65-130%	65-130%	65-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} \cdot \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / |(TPU_1)^2 + (TPU_2)^2|^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 15

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Ra-228

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	7.0 pCi/L	1.0 pCi/g	11 pCi/Filter	1.0 pCi/g	7.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	75-125%	75-125%	75-125%	75-125%	75-125%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

00263

45625

Table G-4 (cont.)

Criterion: 16

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Ra-228

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	3.0 pCi/L	0.5 pCi/g	5.0 pCi/Filter	0.5 pCi/g	3.0 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	70-125%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER≤2(5)	RER≤2(5)	RER≤2(5)	RER≤2(5)	RER≤2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} , \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 16

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Ra-228

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	3.0 pCi/L	0.5 pCi/g	5.0 pCi/Filter	0.5 pCi/g	3.0 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	70-125%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} + \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 18

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVELS (ASL) C & D

ANALYTE: Pu-241

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.5 pCi/L	0.1 pCi/g	5.0 pCi/Filter	0.5 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	70-125%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} \cdot \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 19

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Pb-210

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	7.0 pCi/L	1.0 pCi/g	20 pCi/Filter	2.0 pCi/g	20 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	75-125%	75-125%	75-125%	75-125%	75-125%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

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Table G-4 (cont.)

Criterion: 20

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Pb-210

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	3.0 pCi/L	0.5 pCi/g	10 pCi/Filter	1.0 pCi/g	10 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	70-125%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} + \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

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Table G-4 (cont.)

Criterion: 21

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Sr-90

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	2.0 pCi/L	1.0 pCi/g	4.0 pCi/Filter	1.0 pCi/g	2.0 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%	45-100%	45-100%	45-100%	45-100%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	75-125%	75-125%	75-125%	75-125%	75-125%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} \cdot \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained, If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

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Table G-4 (cont.)

Criterion: 22

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Sr-90

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	1.0 pCi/L	0.5 pCi/g	2.0 pCi/Filter	0.5 pCi/g	1.0 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%	45-100%	45-100%	45-100%	45-100%
Percent Matrix Spike Recovery (6)	70-125%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$
Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 23

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Tc-99

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	30 pCi/L	2.0 pCi/g	110 pCi/Filter	20 pCi/g	70 pCi/L
Percent Overall Tracer/Chemical Recovery (5)	50-100%(6)	45-100%(6)	45-100%(6)	45-100%(6)	45-100%(6)
Percent Matrix Spike Recovery (5)	65-120%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	70-130%	70-130%	70-130%	70-130%	70-130%

(1) $MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$

Where S_{BLK} is the standard deviation of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

(6) Use recovery of matrix spike for batch chemical recovery correction.

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Table G-4 (cont.)

Criterion: 24

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Tc-99

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	15 pCi/L	1.0 pCi/g	50 pCi/Filter	10 pCi/g	30 pCi/L
Percent Overall Tracer/Chemical Recovery (6)	50-100%(7)	45-100%(7)	45-100%(7)	45-100%(7)	45-100%(7)
Percent Matrix Spike Recovery (6)	65-120%	60-130%	60-130%	60-130%	60-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	70-130%	70-130%	70-130%	70-130%	70-130%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} + \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, $RER = |C_1 - C_2| / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

(7) Use average recovery of matrix spike and matrix spike duplicate for batch chemical recovery correction.

Table G-4 (cont.)

Criterion: 25

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Cs-137

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	9.0 pCi/L	0.4 pCi/g	20 pCi/Filter	2.0 pCi/g	20 pCi/L
Percent Overall Tracer/Chemical Recovery	N/A	N/A	N/A	N/A	N/A
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	85-115%	80-120%	80-120%	80-120%	80-120%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained, If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

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Table G-4 (cont.)

Criterion: 26

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: Cs-137

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	4.0 pCi/L	0.2 pCi/g	10 pCi/Filter	1.0 pCi/g	10 pCi/L
Percent Overall Tracer/Chemical Recovery	N/A	N/A	N/A	N/A	N/A
Percent Matrix Spike Recovery	N/A	N/A	N/A	N/A	N/A
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	85-115%	80-120%	80-120%	80-120%	80-120%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) $MDC = \frac{4.65\ SBLK}{K} \cdot \frac{2.71}{T \times K}$

Where SBLK is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, RER = $| C_1 - C_2 | / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 27

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: U-Total (Pulsed Laser Phosphorimetry)

PERFORMANCE PARAMETERS	SAMPLE MATRIX				
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.5 ug/L	0.5 ug/g	10 ug/Filter	0.5 ug/g	5.0 ug/L
Percent Overall Tracer/Chemical Recovery	N/A	N/A	N/A	N/A	N/A
Percent Matrix Spike Recovery (5)	75-125%	70-130%	70-130%	70-130%	70-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (5)	75-125%	75-125%	75-125%	75-125%	75-125%

(1) MDC = 4.65 S_{BLK}

Where S_{BLK} is the standard deviation of the results for the most recent 20 method blanks.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent bassi, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

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Table G-4 (cont.)

Criterion: 28

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) C & D

ANALYTE: U-Total (Pulsed Laser Phosphorimetry)

	SAMPLE MATRIX				
PERFORMANCE PARAMETERS	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)	FLY ASH	CONTAMINATED LIQUID (3)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	0.1 ug/L	0.1 ug/g	2.0 ug/Filter	0.1 ug/g	1.0 ug/L
Percent Overall Tracer/Chemical Recovery	N/A	N/A	N/A	N/A	N/A
Percent Matrix Spike Recovery (6)	75-125%	70-130%	70-130%	70-130%	70-130%
Method Blank Concentration	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)	<HAMDC(4)
Laboratory Control Sample: Percent of Known Value (6)	75-125%	75-125%	75-125%	75-125%	75-125%
Precision Requirements for Duplicate Samples	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)	RER ≤ 2(5)

(1) MDC = 4.65 SBLK

Where SBLK is the standard deviation of the results for the most recent 20 method blanks.

(2) Glass Fiber 8" X 10".

(3) Two phase system containing 90% Water + 10% Organic liquid.

(4) Less than HAMDC or 5% of sample concentration whichever is greater.

(5) Relative Error Ratio, RER = $| C_1 - C_2 | / [(TPU_1)^2 + (TPU_2)^2]^{1/2}$ where C_1 and C_2 are the measured concentrations for the sample and duplicate and TPU_1 and TPU_2 are the respective total propagated uncertainties. Measurements are acceptable if RER ≤ 2. If RER is greater than 2 but less than or equal to 3, investigate the cause and take corrective actions if RER is consistently greater than 2. If RER > 3, take corrective actions and reanalyze the batch of samples.

(6) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent basis, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 29

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS
ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Gross Alpha Activity

PERFORMANCE PARAMETERS	SAMPLE MATRIX		
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	2.0 pCi/L	15 pCi/g	4.0 pCi/Filter
Percent Overall Tracer/Chemical Recovery	N/A	N/A	N/A
Method Blank Concentration	<HAMDC(3)	<HAMDC(3)	<HAMDC(3)
Laboratory Control Sample: Percent of Known Value (4)	40-160%	40-160%	40-160%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} \cdot \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Less than HAMDC or 5% of sample concentration whichever is greater.

(4) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent bassi, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

Table G-4 (cont.)

Criterion: 30

RADIOCHEMICAL ANALYSIS PERFORMANCE SPECIFICATIONS ANALYTICAL SUPPORT LEVEL (ASL) B

ANALYTE: Gross Beta Activity

PERFORMANCE PARAMETERS	SAMPLE MATRIX		
	WATER	SOIL/ SEDIMENT	AIR FILTERS (2)
Highest Allowable Minimum Detectable Concentration (HAMDC) (1)	4.0 pCi/L	30 pCi/g	30 pCi/Filter
Percent Overall Tracer/Chemical Recovery	N/A	N/A	N/A
Method Blank Concentration	<HAMDC(3)	<HAMDC(3)	<HAMDC(3)
Laboratory Control Sample: Percent of Known Value (4)	50-150%	50-150%	50-150%

(1) $$MDC = \frac{4.65\ S_{BLK}}{K} + \frac{2.71}{T \times K}$$

Where S_{BLK} is the standard deviation of the count rate of an appropriate method blank and, K is the correction factor that includes units conversion and typical values for the volume or weight of sample, decay correction factor, detector efficiency and the chemical recovery. T is the counting time of the sample.

(2) Glass Fiber 8" X 10".

(3) Less than HAMDC or 5% of sample concentration whichever is greater.

(4) Recoveries or percentages of known values which are 15% above or below the ranges listed are acceptable on an infrequent bassi, i.e., less than 15% of the time. These occurrences must be investigated and explained. If more than 15% of the recoveries are outside the ranges listed, take corrective actions and reanalyze samples.

APPENDIX I

FIELD CALIBRATION REQUIREMENTS

I.1 TYPE OF EQUIPMENT

Calibration requirements for the following types of field instrumentation are discussed in this appendix.

- pH meter
- Specific conductance meter
- Dissolved oxygen meter
- Redox Potential
- M-Scope (water level indicator)
- Thermometers
- Photo-ionization detector (HNu, TIP, OVM)
- Flame-ionization detector (OVA)
- Explosimeter
- Pressure transducers
- Hand-held radiological survey instruments

Table I-1 (Appendix A) provides a summary of calibration frequency and accuracy requirements for these measuring devices.

I.2 CALIBRATION REFERENCE STANDARDS

Calibration standards represent materials traceable to National Institute of Standards and Technology (NIST), EPA-certified standards, or the best quality materials available. Calibration certification verifies that measurement equipment is working properly in accordance to the applicable standard.

The SCQ user is directed to refer to the manufacturer instruction manual, or to call the manufacturer when uncertain about the calibration requirements and/or procedures.

I.2.1 Geophysical Instruments

Instruments for quantitative geophysical measurement shall be calibrated by the manufacturer or authorized representative at least annually. Field calibration shall be performed or response checked, as applicable, in accordance with manufacturer instructions or Project-Specific Plans (PSP) each day of field use for both quantitative and qualitative instruments.

I.2.2 Flow Meters and Gauges

Instruments that measure flow rate and pressure shall be calibrated by the manufacturer or authorized representative at least annually. Field checks of calibration shall be performed each day of use in accordance with manufacturer instructions or PSP.

I.2.3 Colorimetric Indicator Tube Pumps

Colorimetric indicator tube pumps shall be calibrated by the manufacturer prior to purchase. Calibration shall be checked as recommended by manufacturer instructions or PSP.

I.2.4 Automatic Air Sampling Pumps

Air sampling pumps shall be calibrated by the manufacturer prior to FEMP use. Pump calibration shall be checked as recommended by manufacture instructions or PSP. The power source shall be within manufacturer specifications and checked at least annually.

I.3 CALIBRATION FREQUENCY

Field instruments shall be calibrated at frequencies specified in Appendix G.

I.4 CALIBRATION PROCEDURES

I.4.1 pH

Calibrate pH meters in accordance with manufacturer instructions. Meters shall be direct-reading and temperature-compensating and capable of responding within 0.1 pH unit over a temperature range of - 2 to + 40 degrees Centigrade. Response time of the instrument shall not be greater than two minutes (Manigold, et.al., 1982).

- Proper pressure
- Outage

Standard general requirements for packages used to ship hazardous materials are contained in 49 CFR 173 (1991). In general, packages shall be designed and constructed so that, under normal transportation conditions, there will be no release of the hazardous material and the effectiveness of the packaging will not be reduced. 49 CFR 173 (1991) states that, where there are no detailed specifications for packaging, the hazardous substance shall be enclosed in strong, tight packaging that does not leak or have its effectiveness reduced.

K.10.5.6 **Shipping Documentation.** Basic requirements for preparing shipping documentation for hazardous materials include proper shipping name, hazard classification, ID number, total quantity, and the shipper certification. Provide information on shipping papers required by 49 CFR 172 (1991). See 49 CFR 172 through 172 (1991) for additional requirements.

K.10.5.7 **Certification.** Authorized personnel will certify, by printing on the shipping papers, that the materials being offered for shipment are properly classified, described, packaged, marked, and labeled and are in proper condition for transport as specified in the applicable DOT regulation [49 CFR 172 (1991)]. See 49 CFR 172 (1991) for surface shipment and for air shipments.

K.10.5.8 **Marking the Package.** Apply markings specified in 49 CFR 172 (1991). Requirements for packaging of 110 gallons or less are identified in 49 CFR 172 (1991) and include the following.

- Shipping name
- Identification number
- FEMP name and address
- Laboratory name and address
- Inhalation hazard if required [49 CFR 173 (1991)]
- Name and address required on all packages

K.10.5.9 **Labeling.** See the table in 49 CFR 172 for required labeling, additional labels, location of labels, and packaging of samples.

The Hazardous Materials Tables in 49 CFR 172 (1991) identify proper labels for hazardous materials. Labeling requirements in at 49 CFR 172 (1991) are in specific sections as follows.

- **49 CFR 172.400(a) (1991)** - Labels specified for hazardous materials shipment
- **49 CFR 172.401 (1991)** - Labels affixed to packages even though not required by regulations, provided they represent hazards of material in the package
- **49 CFR 172.406(a) (1991)** - Label location on the package
- **45 CFR 172.406(c)** - Positioning of two or more labels
- **49 CFR 173.3a (1991)** - Inhalation hazard/poison label requirements
- **49 CFR 172.404(b) (1991)** - Label requirements when two or more packages containing compatible hazardous materials are packaged within the same overpack

K.10.6 Radioactive Samples

In general, most samples collected at FEMP are classified as radioactive for transport purposes; however, certain samples may fall into categories for which special packaging and shipping restrictions are mandated. Guidelines for determining the category to which a particular sample shipment belongs and for selecting a suitable mode of shipment and appropriate packaging follow.

Potentially radioactive samples shall be screened as specified by individual laboratory licensing requirements before they can be accepted for analysis. The method for screening in Appendix G shall be followed when applicable. Laboratory-specific license requirements shall take precedence over this requirement.

Regulations impose limits on the total radioactivity (i.e., specific activity times the weight of the package) contained within a package of radioactive material. With respect to type A packages, the limits are expressed as two quantities, A1 and A2, which refer to the maximum permissible activity for radionuclides in special form and normal form radioactive materials, respectively. The samples from FEMP fall into the latter category so the A2 value sets the activity limits for packages of samples. In those cases where contaminated material shipments are designated "low specific activity" or "limited quantity," some fraction of the A2 value will normally apply.

Table K-2 (Appendix A) lists A1 and A2 values cited in 49 CFR 173 (1991) for radionuclides of the uranium decay series. Values for radionuclides not listed in the regulations (e.g., lead-

D.11.1.1 Evaluation Criteria. Verify that the items listed in the SOW, if available, and the Method Specific Appendices are included in the data package. These items may be recorded in various ways, and the data package may be organized differently than this procedure, so some searching may be required. Information that pertains to a requirement that is associated with an **R** qualifier, e.g., initial calibration, is classed as major, and its lack would be a major deficiency. A minor deficiency would result from a lack of information associated with a **J** qualifier, e.g., duplicates. The validator may have to use professional judgement to classify some deficiencies.

D.11.1.2 Procedure. If minor deficiencies are encountered and can not be rectified by the laboratory, then generally all affected (associated) data must be qualified as estimated (**J**). Major deficiencies that can not be rectified will require that all affected data be qualified as unusable (**R**). The validator may use other qualifiers, but their definition must be included in the data validation report. If the validator concludes that no qualification is necessary, the reason must be included in the data validation report.

D.11.1.3 Supplemental Initial Calibration Requirements for Analyses Using Gas Proportional Counters Procedure. Use checklist (Form D-20, Appendix B) to assure completeness of the analysis.

D.11.2 Calibration

Instruments must be calibrated in accordance with laboratory standard operating procedures and/or manufacturer's instructions initially and when a detector or other major system component is changed. Frequently thereafter, less extensive continuing calibration checks, which consist of background and check source counts, must be done.

D.11.2.1 Initial Calibration Evaluation Criteria. Review the data package to verify that the instrument was calibrated within the time period specified in the laboratory standard operating procedure or manufacturer's instructions, but not less than annually.

D.11.2.2 Procedure.

1. If the instrument was not calibrated within the specified time period qualify the associated data as unusable (**R**). Associated data means, in this case, results for all the analyses for each run or day during the period for which no calibration is valid.

2. Each detector in multiple counting systems must be calibrated. Compare the identifications of detectors calibrated against those used for all analyses to verify that each detector used was calibrated.

3. If the detector was not calibrated qualify all associated data as unusable (**R**).

4. Use National Institute of Standards and Technology (NIST), NIST-traceable, or equivalent certified standards for calibration.

5. Review the certifications, including identification numbers, of the standards. Compare the identification numbers on the certificates with identification numbers on the instrument printouts.

6. If the standards used for calibration are not certified or traceable, or cannot be positively identified, qualify all associated data as unusable (R).

NOTE

The standards must not have decayed away by the time they are used for calibration.

7. Review the expiration or issue (assay) dates, and activities of the standards.

8. If the standards were used past their expiration dates, or past five half-lives of the radionuclide of interest if no expiration date is provided, qualify all associated data as unusable (R).

D.11.2.3 <u>Continuing Calibration</u>. The check source should be identified by activity and radionuclide(s).

D.11.2.4 <u>Procedure</u>.

1. If the activity and identity of the radionuclide(s) used in the check source(s) are not provided qualify all associated data as estimated (J).
2. Check source(s) shall be counted daily or as specified in the Appendices.

3. If the daily check source is not performed, qualify associated results as unusable (R).

4. The check source counts shall be within the control limits provided by the laboratory but no greater than plus or minus 3 standard deviations of the mean. Review the results, including raw data, of all daily check source counts.

5. If the check source counts are outside of the control limits, qualify all associated data as unusable (R). Associated data means here all the results for all the counts within the time period covered by the out of control counts. Use the raw data, or compare the raw data with the count log, to determine the affected time periods. Note any bias or trend in the data validation report.

6. Background counts shall be performed at least weekly and before and after all the field and QC samples associated with the Sample Delivery Group (SDG). Review the instrument printouts and counting logs to verify that the background counts were taken on the correct frequency.

7. If the background counts were not performed qualify all associated results as unusable (R).

8. The background counts shall be within the control limits provided by the laboratory but no greater than plus or minus 3 standard deviations of the mean. Review the results, including raw data, of all background counts.

9. If the background counts are outside of the control limits, qualify all associated data as unusable (R). Associated data means all the results for all the field and QC samples counted within the time period covered by the out of control background counts. Use the raw data, or compare the raw data with the count log, to determine the affected time periods. Note any bias or trend in the data validation report.

D.11.2.5 <u>Supplemental Initial Calibration Requirements for Analyses Using Gas Proportional Counters Evaluation Criteria</u>. Depending upon the type of counter/system used review the results, as applicable, of the plateau determination, amount of alpha-beta crossover, random coincidence counts, and/or energy calibration. Review the efficiency determinations and self-absorption curves. Compare the range of the self-absorption curve to the amounts of field and QC samples counted. Self-absorption curves shall be generated for each radionuclide of interest.

D.11.2.6 <u>Supplemental Initial Calibration Requirements for Analyses Using Gas Proportional Counters Procedure</u>.

1. If the field and QC sample preparations are outside the range of the self-absorption curves, qualify all associated data as estimated (J).

2. If the efficiency calculation shows less than 20 percent efficiency, qualify all data as unusable (R).

D.11.2.7 <u>Supplemental Continuing Calibration Requirements for Analyses Using Gas Proportional Counters Evaluation Criteria</u>. Verify that chi-square or other appropriate statistical tests were done for the counters on a routine frequency at least weekly.

D.11.2.8 <u>Supplemental Continuing Calibration Requirements for Analyses Using Gas Proportional Counters Procedure</u>.

1. If a chi-square test was not performed, or results of the test show non-random behavior, then qualify all data as estimated (J).

2. Stability verifications, e.g., plateau(s) or response(s) to the check sources shall be made after each gas change. Specific verifications may not be needed if check sources are used daily.

3. If stability verifications were not performed qualify all data as estimated (J).

D.11.2.9 **Supplemental Initial Calibration Requirements for Analyses Using Alpha Spectroscopy Criteria.** The calibration of the detector system must cover the energy range of interest, that is, the radionuclides (peaks) used for calibration must be identical to and/or bound the energy range of the radionuclides of interest.

D.11.2.10 **Supplemental Initial Calibration Requirements for Analyses Using Alpha Spectroscopy Procedure.**

1. If the energy of the alpha particle(s) of the radionuclide(s) of interest falls outside the calibrated range of the detector, qualify all results as unusable (R).

2. Review the calibration spectrum or printout to verify that the resolution of the detector system provides accurate identification of each peak centroid, i.e., the peaks have sufficient counts and are distinct and separate from each other.

3. If the centroids of the peaks used for calibration can not be determined from the spectrum or printout, qualify all results as unusable (R).

4. A nominal value of 20 keV (or number of channels if detector gain is available) FWHM is used to gauge resolution for each peak used to calibrate the detector system.

5. If the resolution of the system is greater than 20 keV (or corresponding number of number of channels) FWHM for any of the peaks used for calibration, qualify all results as estimated (J).

D.11.2.11 **Supplemental Continuing Calibration Requirements for Analyses Using Alpha Spectroscopy Criteria.**

1. Compare the efficiency obtained from the calibration to the efficiency obtained from the check source count(s) for the SDG. The efficiencies should be within 5 percent.

OR

2. Compare the efficiency from the check source count for the SDG with the control charts. The efficiency should be within the control limits or 3 sigma.

D.11.2.12 Procedure for Supplemental Continuing Calibration Requirements for Analyses Using Alpha Spectroscopy.

1. If the check source efficiency is not within the limits, qualify all associated results as unusable (R).

D.11.2.13 Criteria for Supplemental Calibration Requirements for Analyses Using Gamma Spectroscopy. Efficiency calibration does not approximate a smooth semi-log curve.

D.11.2.14 Procedure for Supplemental Calibration Requirements for Analyses Using Gamma Spectroscopy.

1. Review the efficiency calibration curve and/or raw data, and, if the efficiency calibration does not approximate a smooth semi-log curve, qualify results as unusable (R).

2. Verify that geometry and matrix factors were accounted for in the analyses of all field and QC samples.

3. If geometry and matrix factors are not used, qualify all results as unusable (R).

4. The calibration of the detector system must cover the energy range of interest, but at least 0 to 2 MeV. Review the energy calibration and verify that the radionuclides (peaks) used for calibration are within and/or bound the energy range of the radionuclides of interest.

5. If the energy of the radionuclides falls outside the calibrated range of the detector, qualify all results as unusable (R).

6. Review the calibration data to verify that the resolution of the detector system is sufficient for the radionuclides of interest, i.e., that accurate identification of peak centroid can be made, and the peaks are distinct and separate from each other. A nominal value of five channels (full width, half maximum) is used to gauge resolution.

7. If the resolution of the system is greater than five channels (full width, half maximum) for any peaks used for calibration, qualify all results as estimated (J).

D.11.2.15 Criteria for Supplemental Calibration Requirements for Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting. A counting system consists of a scintillation cell and associated photomultiplier tube, electronics and scaler. Each counting system should be calibrated as a unit. Calibration consists of determining a calibration constant using a NIST traceable Ra-226 standard. The calibration constant includes the de-emanation

efficiency of that system and the counting efficiency of the scintillation cell. The calibration constant should be established for a system at the beginning of each day that samples will be analyzed and each time the scintillation cell is replaced. The scintillation cell should be uniquely identified to allow its association with a specific counting system to be identified.

D.11.2.16 Procedure for Supplemental Calibration Requirements for Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting.

1. If calibration data cannot be definitely associated with the specific counting system, qualify associated sample results unusable, (R).

2. If the counting system is not calibrated each day that samples are analyzed, qualify associated results as estimated, (J).

3. If the counting system is not calibrated upon replacing the scintillation cell, qualify associated results as estimated, (J), if the cell has a previously determined calibration constant or is unusable, qualify as unusable (R), if no constant is available for the replacement cell.

D.11.2.17 Supplemental Calibration Requirements for the Analysis of Tritium Using Liquid Scintillation Counting. Liquid scintillation counting systems are calibrated using NIST traceable external or internal standards. The chemical and physical matrix of the standards should resemble the samples as closely as possible in order to match light emission (scintillation) and quenching properties.

- If the matrix of the sample is not representative of that for the samples, qualify all associated results as estimated, (J).

Most automated liquid scintillation systems are capable of processing many more samples than is normally contained in an SDG. As a minimum, one calibration standard should be included with every analytical run of samples. An analytical run can be comprised of more than one SDG.

- Qualify results associated with runs lacking calibration standards as unusable, (R).

The efficiency for detecting the tritium beta particle must be established for each counting system. The counting system is comprised as the liquid scintillation counter, scintillation solution, and sample matrix. The laboratory should provide detailed explanation of the method and results for determining the counting system efficiency. For automated systems employing computerized algorithms, copies of applicable pages from the instrument manuals should submitted with the analytical results.

- If the method and results for counting efficiency determination are not provided, qualify all associated results as unusable, (R).

D.11.2.18 Supplemental Calibration Requirements for Fluorometric Analysis of Uranium. In addition to the internal standard added to each sample, a calibration should be performed prior to sample analysis to confirm the linear relationship between the fluorometer readings and uranium concentrations.

- If a calibration is not performed prior to sample analysis to verify linear instrument response, qualify associated results as estimated, (J).

D.11.3 Blanks

D.11.3.1 General Criteria. A reagent (or method) blank of the same aliquot size as the samples must be processed like a sample and analyzed with each SDG on the same detectors or detector system, or a field blank must be analyzed with each SDG on the same detectors or detector system, or both. As a minimum one blank must be analyzed with the SDG.

D.11.3.2 General Procedure.

1. If no blanks were analyzed with the SDG, or if the blanks were not analyzed on the same detectors or detector system, qualify all the results greater than Lower Limit of Detection (LLD) as estimated (J).

2. The net blank value, i.e., the results from the analysis of the blank corrected for background, should be less than the Minimum Detectable Activity (MDA), which is the quantity of radioactive material that can be detected in the sample at the 95 percent confidence level. Review the data and verify that no contaminants were detected.

3. If contaminants are detected in any blank, associated sample results that are reported as statistically greater than background but less than the MDA, are qualified as nondetects (U). Any other sample result is qualified as an estimated detect (J) unless the sample result is 10 times the contaminant in the blank. Generally, no action is taken for radionuclides detected in a blank but not in a sample, although the validator must be vigilant for situations when a radionuclide in a blank but not in a sample may cause interference with other radionuclides of interest in the sample.

4. Verify calculation or method of calculating the net blank value.

5. Any blank with a negative result whose absolute value is greater than LLD must be carefully evaluated to determine its effect on sample data. Review all the QC data specific to the method to evaluate the possibility of false negatives.

D.11.3.3 Supplemental Blanks Requirements for Analysis of Alpha-Emitting Radium Isotopes Using Scintillation Counting. Most chemical reagents contain some levels of radium. Generally, it is prudent to analyze additional blank samples in the event that the batch or lot number of a reagent should change in the course of preparing a group of samples for analysis.

- If additional blank samples are not analyzed to check potential contamination of reagents with a different lot number from those used for previous blanks, qualify associated results as estimated (J).

D.11.3.4 **Supplemental Blanks Requirements for Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting.** Most chemical reagents contain some levels of radium. Generally, it is prudent to analyze additional blank samples in the event that the batch or lot number of a reagent should change in the course of preparing a group of samples for analysis.

- If additional blank samples are not analyzed to check potential contamination of reagents with a different lot number from those used for previous blanks, qualify associated results as estimated (J).

D.11.3.5 **Supplemental Blanks Requirements for the Analysis of Tritium Using Liquid Scintillation Counting.** Tritium in chemical reagents or water used for background determinations will interfere with sample results and reduce the method sensitivity. Tritium levels in reagents and background water should be less than the desired MDA for the method.

- If the tritium levels in reagents or background water exceed the desired MDA, qualify associated results less than 10X the background tritium level as estimated (J).

- If additional blank samples are not analyzed to check potential contamination of reagents with a different lot number from those used for previous blanks, qualify associated results estimated (J).

D.11.4 Detection Limits and Sample Results

D.11.4.1 **Criteria.** Methods must be equivalent to the following.

$$\text{LLD} = (4.66)\ (\text{Background Counts})^{1/2}$$

$$\text{MDA} = \frac{(4.66)\ (\text{Background Counts})^{1/2}}{(\text{Efficiency})\ (\text{Volume})\ (\text{Yield})\ (\text{Conversion Factors})}$$

D.11.4.2 **Procedure.**

1. Verify calculation or method of calculating the LLD and the MDA.

2. Verify that positive results (detects or results not qualified U) reported meet detection limits stated in the SOW and are above the MDA for the analysis or method.

3. If the LLD and MDA cannot be verified, qualify all associated results as estimated detects (J) or estimated nondetects (UJ).

4. Verify the calculation of sample results by examining the raw data, i.e., spectra or printouts, for counts, counting time, efficiencies, and yields or recoveries, and data transcriptions, e.g., sample volumes.

5. Correct errors on the photocopied pages of the data package and include in the data validation report.

D.11.5 Radiometric and Gravimetric Yields

D.11.5.1 Criteria. At least one spike, tracer, or chemical yield must be analyzed in each SDG. The tracer, spike, or carrier must have chemical and radioactive characteristics appropriate for the sample matrix and analytical method.

D.11.5.2 Procedure.

1. If no or an inappropriate spike, tracer, or carrier was used qualify all associated results as unusable (R).

2. Samples identified as field blanks may not be used for spike, tracer, or chemical yield analysis. Verify that the field blank was not used for such analyses. Look at chain-of-custody documents to find identifier.

3. If the field blank was used for spike, tracer, or chemical yield analysis, all other QC data must be carefully checked and professional judgement used when evaluating the data. Document if the field blank was used but don't qualify data on this alone.

4. Spike or tracer per cent recovery, or chemical yield must be within the control limits of 30-105 percent for all radionuclides or as specified in the Appendices. If sample activity is greater than 4x the spike activity, recovery limits do not apply. Review the raw data for counts, activity, and aliquot of the tracer, spike, or carrier used. Verify that the per cent recoveries or yields were correctly calculated and reported and that the results fall within the specified limits.

5. Qualify associated sample results outside of the acceptable limits as estimated detects (J), estimated nondetects (UJ), or unusable (R) according to the following guidelines. Note any bias or trend in estimated results in the data validation report.

Yield (%R):	<30%	30-105%	>105%	>115%
Results < LLD:	R	acceptable		UJR
Results > LLD:	R	acceptable		JR

D.11.5.3 Criteria for Supplemental Requirements for Radiometric Yields for Analyses Using Alpha Spectroscopy. A tracer should be used to spike each sample prior to analysis. The percent recovery of the tracer should be between 30 percent and 105 percent.

D.11.5.4 Procedure for Supplemental Requirements for Radiometric Yields for Analyses Using Alpha Spectroscopy.

1. If a sample did not have a tracer added, qualify the result as unusable (R).

2. Verify that recovery is calculated from the applicable instrument efficiency. Check the calculation of recovery using the following formula.

$$\text{percent recovery} = (\text{net CPM tracer/DPM tracer added}) \times (\text{efficiency in DPM/CPM})\ (100)$$

3. Qualify results outside of the acceptable limits as estimated (J or UJ), or unusable (R) according to the following guidelines.

Yield (%R):	<30%	30-105%	>105%	>115%
Results < LLD:	R	acceptable		UJR
Results > LLD:	R	acceptable		JR

4. Record any bias or trend in estimated results in the data validation report.

D.11.5.5 Gravitimetric Yield Requirements for Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting. The laboratory shall provide information on typical chemical recoveries achieved with the method and evidence that sample results were corrected.

- If information is not provided on the determination of the chemical yield for each sample, qualify the results as estimated (J).

D.11.6 Duplicate Samples and Analyses

D.11.6.1 Criteria. At least one duplicate processing and analysis must be performed for every twenty samples in the SDG.

D.11.6.2 Procedure.

1. If the number of duplicate analyses is not satisfactory, qualify all associated results as estimated (J).

2. Samples identified as field blanks (look at chain-of-custody documents) may not be used for duplicate sample analysis. Confirm that the field blank was not used for the duplicate analysis.

3. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data. Document if the field blank was used, but don't qualify data on this alone.

4. RPD between the sample and duplicate analysis of samples must be within the control limits of ± 35 percent for results greater than 5x the LLD). A control limit of ± 2x the LLD is applied if one or both of the sample values are less than 5x the LLD. If both values are less than LLD, no control limit is applicable. Review the data package and verify that results have been correctly calculated and reported and fall within the established control limits.

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

S = first sample value (original)

D = second sample value (duplicate)

5. If duplicate analysis results for a particular radionuclide are outside specified control limits, qualify results less than LLD as estimated nondetects (UJ), and results greater than LLD as estimated detects (J) for that radionuclide in all samples of the SDG.

D.11.7 Laboratory Control Samples

LCS may be prepared by the same laboratory performing the analyses or by a reference laboratory or agency. Laboratory control samples are equivalent to internal or external control samples. Laboratory Control Samples, or their equivalents, may be identified as QC samples, as samples from a particular agency, or as LCS.

D.11.7.1 General Criteria. All LCS results must be within the control limits. If control limits are not provided by the laboratory then use the control limits of 80-120 %R. Review the data package and verify that all LCS results have been correctly calculated and reported and fall within the specified control limits.

$$LCS\ \%R = \frac{\text{LCS found}}{\text{LCS true}} \times 100$$

Where:

LCS found = concentration or activity for each radionuclide measured in the LCS

LCS true = concentration or activity of each radionuclide in the LCS

D.11.7.2 General Procedure.

1. Qualify affected sample results as estimated detects (J), estimated nondetects (UJ), or unusable (R) for radionuclides that are outside control limits according to the following guidelines. Affected sample results may be all results for the SDG or just the results following an out-of-control LCS.

Radionuclide %R	<50%	50-79%>	120%
Results < LLD:	R	UJ	R
Results > LLD:	R	J	R

2. Note any bias or trend in estimated results.

3. At least one LCS must be analyzed with the SDG.

4. If the required LCS are not analyzed, qualify all results as estimated (J).

D.11.7.3 Supplemental Requirements for Laboratory Control Samples for Analysis of Alpha-Emitting Ra Isotopes Using Scintillation Counting. This method will not be able to achieve the criteria for accuracy specified in Section 6.8. Accuracy is determined by comparing the analytical results from laboratory control samples with the known concentration of Ra isotopes. The two values are used to compute a recovery factor (LCS %R presented in Section 6.8).

- Qualify associated sample results according to the following criteria.

Method Accuracy Criteria

	LCS %R			
	<50%	50-69%	70-130%	>130%
Results <LLD	R	UJ	Acceptable	R
Results >LLD	R	J	Acceptable	R

D.11.8 Holding Times

D.11.8.1 General Criteria. Samples shall be analyzed within the period of 5 half-lives of the radionuclide of interest or within the holding time specified in Table 6-1, whichever comes first. Samples shall be properly contained and preserved (e.g., acidified) in accordance with laboratory standard procedures, to ensure that sample integrity is maintained. Holding times for each radionuclide are established by comparing the sampling date on the chain-of-custody record with the dates of analysis found in the data package.

Analysis date - sample date = radionuclide holding time

D.11.8.2 General Procedure.

1. Review the chain-of-custody documents to determine if the samples were preserved in accordance with the laboratory procedure.

2. If holding time or preservation requirements are not met, qualify all results greater than LLD as estimated detects (J) and results less than LLD as estimated nondetects (UJ). Professional judgement must be used in the case of grossly exceeded holding times. The expected bias would be toward lower results and the reviewer may determine that results less than LLD are unusable (R).

D.11.8.3 Supplemental Holding Times Requirements for Analysis of Alpha-Emitting Ra Isotopes Using Scintillation Counting. Samples are to be preserved by adjusting the pH to less than 2 with Nitric Acid. Depending on to the time of preservation, the following holding time requirements are specified.

1. If the sample is preserved at the time of collection, subsection 6.9 requirements apply.

2. If the sample is not preserved at the time of collection, the following additional requirements apply.

   - Time from sampling to receipt at laboratory cannot exceed 5 days.

   - Laboratory must preserve the sample upon receipt and hold for at least 16 days prior to analysis.

3. Qualify associated sample results estimated (J) if holding time requirements are not met.

D.11.8.4 Supplemental Holding Times Requirements for Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting. Samples are to be preserved by adjusting the pH less than 2 with Nitric Acid. According to the time of preservation the following holding time requirements are specified:

1. If the sample is preserved at the time of collection, Section 6.9 requirements apply.

2. If the sample is not preserved at the time of collection, these additional requirements apply.

   - Time from sampling to receipt at laboratory shall not exceed 5 days.

   - The laboratory shall preserve the sample upon receipt and hold for at least 16 days prior to analysis.

3. Qualify associated sample results as estimated (J) if holding time requirements are not met.

D.11.9 Analysis of Ra-226 Using Scintillation (Lucas) Cell Counting

D.11.9.1 Scope. This Appendix provides supplemental information for validating analytical results from the determination of Ra-226 in water. The Ra-226 is separated from interfering species and concentrated by co-precipitation. After allowing for the ingrowth of Rn-222, the radon is purged and counted in a scintillation (Lucas) cell.

D.11.9.2 Applicability. The validation criteria in this appendix are intended to be applied in addition to those found in the body of the procedure. In cases where discrepancies exist between the procedure and the appendix, the criteria in the appendix shall apply.

D.11.10 Supplemental Requirements for Fluorometric Analysis of Uranium

D.11.10.1 Scope. This Appendix provides supplemental information for validating analytical data from the determination of uranium by fluorometry. The uranium is separated from interferences and concentrated by co-precipitation and purified by solvent extraction. The sample is fused with flux and analyzed on a fluorometer.

D.11.10.2 Applicability. The validation criteria in this appendix are intended to be applied in addition to those found in the body of the procedure. In cases where discrepancies exist between the procedure and the appendix, the criteria in the appendix shall apply.

D.11.10.3 Interferences. The fluorescence of uranium in the fluoride matrix can be either quenched or enhanced by the presence of cations or anions. When uranium is present in low concentrations, the interferences can be removed by various methods.