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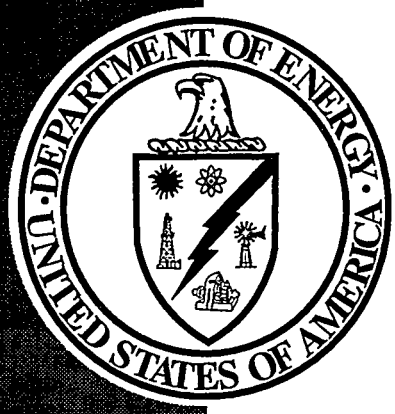

Analytical Laboratory Quality Assurance Guidance

IN SUPPORT OF

EM Environmental Sampling and Analysis Activities

Prepared by:
Analytical Services Division (EM-263)
Office of Compliance and Program Coordination
Environmental Management
U.S. Department of Energy

May 1994

WPI.94-0189

DISCLAIMER

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PART I

PROGRAM DESCRIPTION

1.0 INTRODUCTION

The basis for the Department of Energy's (DOE) Office of Environmental Restoration and Waste Management's (EM) Analytical Services Program (ASP) is contained in the charter and commitments described in Secretary of Energy Notice (SEN) 13-89, EM program policies and requirements, and commitments to Congress and the Office of Inspector General (IG). EM's commitment to the development and implementation of the ASP by the Analytical Services Division (EM-263) is in response to concerns raised by the Chairman of the Congressional Environment, Energy, and Natural Resources Subcommittee on Energy and Commerce regarding the production of environmental data. The development and implementation of an ASP also satisfies the IG's audit report recommendations (IG Reports IG-0293 and IG-0295) on environmental analytical support, including development and implementation of a national strategy for acquisition of quality sampling and analytical services. These recommendations were endorsed in Departmental positions, which further emphasize the importance of the ASP to EM's programs.

This document describes the EM environmental sampling and analysis activities (ESAA) considered to represent the minimum activities necessary to achieve the intended goals.

The Analytical Services Program's ESAA program strategy is designed to comply with DOE 5700.6C (Order) and the EM Quality Assurance and Requirements Description (QARD) document to ensure the production of data readily acceptable to regulatory agencies. The referenced Order and documents establish the quality assurance requirements for EM.

1.1 Requirements to Establish Analytical Laboratory Guidance

Requirements for the establishment of Analytical Laboratory Quality Assurance Guidance originate from several sources: EM's need to address compliance with environmental and safety laws and regulations and to enhance the technical validity of EM programs and projects as part of its overall responsibility to achieve environmental protection; direction from the Secretary of Energy in 1989 to establish an analytical quality assurance program to support environmental restoration and waste management activities in response to DOE/IG findings; DOE 5700.6C, which establishes quality assurance (QA) requirements for DOE; and SEN-6E, which establishes assessment and self-assessment requirements for DOE.

1.2 Purpose

This document introduces QA guidance pertaining to the design and implementation of laboratory procedures and processes for collecting EM ESAA data. The guidance is consistent with and supports DOE (5700.6C) and consensus (ANSI/ASQC E4-1993) QA requirements.

The document addresses several goals:

- identifying key laboratory issues and program elements to EM headquarters and field office managers;
- providing non-prescriptive guidance consistent with regulatory and DOE requirements and a compilation of pertinent references; and
- introducing environmental data collection program elements that are the technical basis for EM-263 assessment documents and programs.

The guidance presented is not prescriptive. However, the elements and processes presented can be integrated into an effective analytical laboratory operation. EM Headquarters management concern is functionality, not form. Laboratory assessments will reflect this emphasis on cost-effective quality and performance of all pertinent analytical procedures and processes.

1.3 Scope

Specific sections of this guidance apply to EM program managers at headquarters and field offices (e.g., laboratory operation issues). Detailed technical guidance applies to DOE contractors and subcontractors in designing and/or reviewing data collection activities.

This guidance describes the implementation of laboratory QA elements within a functional QA program. The development of the QA program and of project-specific Data Quality Objectives (DQOs) are outside the scope of this guidance and not addressed in this document. Additional EM guidance covering these and other technical areas (e.g., Data Quality Assessment) is being developed.

1.4 Relationship to Regulatory Requirements and Existing Programs

This document provides guidance designed to be compatible with existing regulatory QA requirements. However, this guidance may not address all specifications and requirements detailed in various local, state, and other federal programs such as the Department of Transportation (DOT) and the Nuclear Regulatory Commission (NRC). To ensure that all specifications, compliance and regulatory requirements are met, the analytical laboratory organization should consult its specific regulatory program requirements, quality assurance program requirements, project plans, and any other applicable site documents.

The references provided in this document do not confer requirements on EM programs and projects. They are provided to identify existing materials, and as sources of information for use by all levels of EM management.

The Order requires the analytical laboratory's QA plan to be a statement of the laboratory's approach to ensure that quality data are generated and reported. The laboratory QA plan should be designed to cover a single operating facility at a single location. Contracting organizations, corporations or cooperative agreement participants may develop umbrella QA plans, however, it is anticipated that the unique aspects of each facility's

implementation of this plan would be encompassed by a facility-specific QA plan or attachment to the corporate QA plan.

The purpose of this document is not to require a separate QA program. QA elements found in this guidance document may already be incorporated into various existing laboratory documents and need not be located in one document. All items addressed in this guidance document need not be incorporated into laboratory documents; however, documentation as to why items are incorporated or are not incorporated into laboratory documents should be maintained. A summary document identifying where the QA elements are located in existing documents should be developed and maintained, if one does not already exist.

While this document does not cover the detailed development of DQOs or quality assurance programs (QAP), employees and management should be familiar with the specific QA program plans (QAPP), QA project plans (QAPjP) and applicable DQO requirements and concepts. The concepts of Total Quality Management (TQM) and continuous improvement should be applied throughout the planning, implementation, oversight, and assessment phases of the programs and projects. Training should emphasize that the over application of requirements not needed to satisfy project requirements, resulting in excessive project costs, is an important consideration.

1.5 Analytical Contracting and Subcontracting Guidelines

To support integration of needs requirements and to assure the collection of acceptable laboratory products, all local EM program or project management offices should contract for ESAA through a local sample management office associated with the EM's National Sample Tracking System (NSTS). This will support national EM program needs and assure that local EM contract selection and monitoring procedures are consistent with DOE and regulatory standards.

An important aspect of laboratory procurement is to include requirements for participation in regulatory driven and DOE performance evaluation (PE) programs. Participation is defined by site-specific needs. However, free access to all reported analytical results directly from the program sponsor (i.e., U.S. Environmental Protection Agency (EPA)) must be granted to both the contracting organization and to EM-263. This access is necessary to assure timely and accurate monitoring of EM's national program. Both national and program results are distributed to the field offices and to headquarters funding organizations.

In addition to the exchange of data pertaining to performance evaluation sample programs, free access to audit reports and findings resulting from other DOE organizations, Federal agencies, and State's programs is necessary. Contracted laboratories should agree to support free exchange of audit materials between agencies to decrease program redundancy.

1.6 References

1. Bottrell, D.W., Fisk, J.F., Robertson, G.L., Petty, J.D., Dempsey, C.H. and Bartling, M.L. 1991. "Holding Times of Volatile Organics in Water," *Waste Testing and Quality Assurance: Third Volume*, *ASTM STP 1075*, D Friedman (editor), Philadelphia, PA: American Society for Testing and Materials.

2. Federal Register. December 3, 1979. 40 CFR Part 136. Proposed Rules. Vol. 44, No. 233:69534.

3. Federal Register. October 26, 1984. 40 CFR Part 136. Rules and Regulations. Vol. 49, No. 209:145.

4. Federal Register, 10 CFR Part 20, Nuclear Regulatory Commission, Radiation and Protection of the Environment.

5. Federal Register, 49 CFR Part 171, Department of Transportation, General Information, Regulations and Information.

6. Maskarinec, M.P., L.H. Johnson, and S.K. Holladay. 1988. Recommended Holding Times of Environmental Samples, proceedings of U.S. EPA Third Annual Symposium on Solid Waste Testing and Quality Assurance, H29-35.

7. Maskarinec, M.P. and L.R. Moody. 1988. Principals of Environmental Sampling, L.H. Keith (editor), p. 145.

8. Taylor, John K. 1987. "Quality Assurance of Chemical Measurements."

9. U.S. Army Toxic and Hazardous Materials Agency. February 1993. Practical Reporting Times for Environmental Samples. Oak Ridge National Laboratory, Martin Marietta, Oak Ridge, TN. DOE IAG No. 1769-A073-A1

10. U.S. Department of Energy. September 23, 1986. 5700.6C, Quality Assurance, Assistant Secretary for Environment, Safety and Health, Rev. #3, Washington, DC: Government Printing Office/Document Control.

11. U.S. Department of Energy/Office of the Inspector General, IG Report No. 0293 dated December 21, 1990 and IG Report 0295 dated August 12, 1991, Washington, DC: Government Printing Office/Document Control.

12. U. S. Department of Energy. October 31, 1991. Office of Environmental Restoration and Waste Management, Office of Quality Control and Quality Assurance, Quality Assurance Requirements and Description Document.

13. U.S. Department of Energy. November 1992. Secretary of Energy Notices, SEN-6E, Washington, DC: Government Printing Office/Document Control.

14. U.S. Environmental Protection Agency. February 1983. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans - Interim Final, QAMS 005/80, EPA-600/4-83-004

15. U.S. Environmental Protection Agency. 1984. Soil Sampling Quality Assurance Users Guide (1st. Edition). Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. EPA/600/4-84-043

16. U.S. Environmental Protection Agency. March 1987. "Data Quality Objectives for Remedial Response Activities Development Process", OSWER Directive 9355.0-7B.

17. U.S. Environmental Protection Agency. 1988. Sampling for Hazardous Materials. Environmental Response Team, Office of Emergency and Remedial Response, Hazardous Response Support Division, Washington, D.C.

18. U.S. Environmental Protection Agency. 1989. Soil Sampling Quality Assurance Users Guide (2nd. Edition). Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. EPA/600/8/89/046

19. U.S. Environmental Protection Agency. May 1990. A Rationale for the Assessment of Errors in the Sampling of Soils. Environmental Monitoring Systems Laboratory, Las Vegas, NV. EPA/600/4-90/013

20. U.S. Environmental Protection Agency. November 1991. Description and Sampling of Contaminated Soils, A Field Pocket Guide. Technology Transfer. EPA/625/12/91/002

21. U.S. Environmental Protection Agency. July 1992. Test Methods for Evaluating Solid Waste, 3rd Edition, Office of Solid Waste and Emergency Response, EPA-SW-846.

22. U.S. Environmental Protection Agency. 1992. SACM Program Management Update: Identifying SACM Program Management Issues. Office of Solid Waste and Emergency Response. 9203.1-051

23. U.S. Environmental Protection Agency. 1992. SACM Program Management Update: Regional Decision Teams. Office of Solid Waste and Emergency Response. 9203.1-051

PART II

TECHNICAL GUIDANCE

2.0 LABORATORY SYSTEMS GUIDELINES

Applicable EM program or project management (EMPPM), in conjunction with personnel knowledgeable in the relevant analytical criteria, should develop, establish, and update requirements for laboratory organization and personnel, personnel training, facility guidelines, analytical methods, standard operating procedures, corrective actions, document control, and laboratory assessments. Documented procedures should be in place. If the local EMPPM determines that existing laboratory Standard Operating Procedures (SOPs) are sufficient to meet or exceed project needs, new documents need not be developed. For most projects, existing laboratory SOPs should meet or exceed project requirements.

2.1 Laboratory Organization and Personnel

Each analytical laboratory organization supporting DOE EM efforts should clearly define corporate- and facility-specific operational organization and lines of authority. This may be accomplished through an organizational diagram or chart illustrating lines of authority and reporting responsibilities.

Direct and ultimate responsibility for assuring data quality resides with line management (e.g., chief executive officer, laboratory director, section leader), not the QA officer of the laboratory. QA functions provide technical support to management for review and assurance of data quality. Within the organization, every effort should be made to create independent lines of authority and reporting routes for QA functions.

All significant changes in laboratory organization and personnel should be reported to the appropriate EMPPM. Such changes may include facility mergers or acquisitions, expansions, relocation, management adjustments, and changes in primary technical or QA personnel. Regulatory actions toward the facility or its parent corporation, such as suspension of contracts with other federal agencies, as well as all notices of investigations and legal actions against the organization or its personnel should be reported immediately.

2.1.1 Personnel Qualification

Years of analytical experience may often outweigh or gain equivalency to academic achievement. The appropriate corporate, facility or laboratory personnel organization should gauge and document the competency of experienced individuals, and should have in place policy and requirements to establish individual qualifications and competencies for the position in question (e.g., analyst, technician, instrument operator).

The laboratory should maintain comprehensive information on each employee regarding the individual's formal education, training, and experience. This may include such documentation as copies of the individual's up-to-date résumé, degrees earned, certificates of courses completed, and records of in-

house training. It may also include continuing records of the individual's performance related to quality control (QC) and PE effectiveness.

2.2 Personnel Training

Managers should assure that all personnel performing tasks and functions have the needed education, training and experience, and are aware of, and perform, quality work. All pertinent training should be documented through attendance records, individual instruction verified through the instructor's signature, or certificate, or actual written or practical testing sources. Personnel should be provided with continued training to ensure that job proficiency is maintained.

Generally accepted laboratory practice includes personnel training requirements established for the selection and qualification of personnel to assure that:

- Qualification programs are developed, implemented, and documented in an effective and reliable manner consistent with the hazard involved and the risk associated with laboratory operations.
- Qualification programs promote an awareness of the risks involved and a level of proficiency consistent with assigned tasks.
- Personnel receive awareness training regarding the hazards associated with a specific task or procedure to be performed.
- Personnel performing work are capable of performing their assigned tasks. Qualification requirements are to be established for specific job categories. Training includes both education in principles and enhancement of skills.
- Training emphasizes correct completion of work and provides understanding of why specific project quality requirements exist. Training is to provide an understanding of the fundamentals of the work and its context to the QAP and project DQOs. Training instruction is to address potential consequences of improper work, for both over-application of requirements as well as under-application of requirements.

Minimum training requirements include applicable Occupational Safety and Health Administration (OSHA), Site Health, and Safety training. Radiation control worker training may be required if a laboratory handles radioactive materials, or is located on a DOE facility that requires radiation control training.

2.3 Facilities Guidelines

Administrative, technical, and operating procedures and safety analysis reports should be developed and implemented that include the following requirements:

- Laboratory facilities should be secure. The building and laboratories should limit access to authorized personnel. Entrance to the building(s) should be monitored and visitors to the facility should be registered.

- Analytical instrumentation, furniture, equipment, and utilities should be maintained to perform the required analyses.

- Analytical standards, reagents, and sample storage areas should be isolated from potential sources of contamination. It is recommended that organic preparation, volatile organic analyses, and semi-volatile organic analyses areas be separated. Sample preparation, storage, and hazardous and/or mixed waste areas should be separate from the instrumentation or analytical facilities. If required, the analytical laboratory should be operated in accordance with the applicable radiation control program. Areas of transition between radiation and non-radiation areas should be established.

- Laboratory design and the actual implementation of analytical programs should address situations or conditions necessary for the controlled use, storage, and disposal of samples, sample remnants, and chemical wastes. Laboratory design should incorporate sample receipt rooms for the inspection and isolation of unknown samples before they are introduced into the analytical areas, and to establish radiation levels associated with the sample. Laboratory design should minimize interactions between high and low concentration areas, as well as minimize common utilization of equipment, instrumentation, and facilities. It is important to stress that an active contamination control program should exist to minimize the potential spreading of contamination between the laboratory and sample storage areas. Specially controlled facilities or areas should be considered for the receipt of highly contaminated materials, preparation of calibration standards, and storage of standards and waste.

2.4 Analytical Methods

Documentation of analytical procedures is critical to the technical defensibility and the legal defensibility/admissibility of the resulting data. Generally accepted laboratory practice is that, whenever possible, industry-recognized analytical methods from agency published source documents such as DOE, EPA, and American Society for Testing Materials (ASTM) should be employed. Analyses should be conducted in accordance with current DOE and EPA methods as detailed in the following sources:

- DOE Methods for Evaluating Environmental and Waste Management Samples, DOE/EM-0089T, October 1992.

- Radiological and Environmental Sciences Laboratory Manual, Current revision; Analytical Chemistry Branch Technical Procedures Manual, Current revision; Laboratory Quality Branch Technical Procedures Manual, Current revision, U.S. Department of Energy, Idaho Falls, Idaho 83402

- Environment Measurement Laboratory Procedure Manual, 1990, 27th Edition, U.S. Department of Energy HASL-300, Edited by Herbert L. Volchok, Gail de Planque, 376 Hudson Street, New York, NY 10014.

- US EPA Methods for Chemical Analysis of Waters and Waste, EPA-600/4-79-020.

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, July 1992. Updates to this publication should be incorporated into laboratory protocol as the updates become finalized.

- US EPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration.

- US EPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration.

- 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Final Rule, October 26, 1984 (with subsequent corrections).

- 40 CFR Part 261 et. al., Hazardous Waste Management System; Identification and Listing of Hazardous Waste; Toxicity Characteristics Revisions, Final Rule, March 29, 1990 and June 29, 1990.

Methods employed that are not found in the above references should be thoroughly reviewed and approved by the appropriate EMPPM prior to implementation. Complete and well documented method references should be available for all such methods. In lieu of specific method references, appropriate chapters of documents, such as suppliers manuals, equipment manufacturer instructions, and instrumentation specifications should be used. Such documents should include adequate descriptions and criteria to assure the required quality of work.

2.5 Standard Operating Procedures

As a general rule, the SOPs should encompass administrative, operational, and analytical aspects of the laboratory. When SOPs are developed or reviewed, the following areas should be considered:

- references to source documents published by agencies such as the ASTM, American National Standards Institute (ANSI), DOE and EPA should be included;

- document control of SOPs; and

- review and revision of SOPs as required to address changes in data quality requirements, technology and equipment changes, and/or changes in regulatory requirements.

Revisions to SOPs previously approved for EM work should be reported to appropriate EMPPM for reapproval and is required prior to laboratory implementation for use on EM samples.

2.5.1 Operational Standard Operating Procedures

The number and type of operational SOPs instituted by a particular analytical laboratory may vary greatly, depending on the focus of the operation.

It is suggested that the following operational SOPs be in place:

- sample identification;
- chain of custody;
- sample receiving;
- sample tracking;
- materials receiving and acceptance;
- laboratory notebooks;
- logbooks (temperature logs, balance logs, instrument maintenance logs, instrument run logs, sample storage logs, standards logs, etc.);
- document control, including the review, approval, and signature authority of both the management and QA function of the laboratory; availability to personnel at the appropriate work stations; manual of all SOPs current and copies of SOPs used in the past;
- laboratory-ware cleaning procedures;
- data management and handling;
- data review and verification;
- QA and QC procedures;
- control of chemicals, storage conditions, and shelf life;
- standards preparation and control;
- instrument operation, if not specified in the analytical methods;
- instrument maintenance;
- facility maintenance;
- software verification and validation;

- corrective actions to contractual deficiencies found during external and/or internal laboratory inspections, surveillance, audits, assessments or other oversight functions; and

- subcontracting procedures for EM samples.

2.5.2 Analytical Standard Operating Procedures

Every analytical method employed in the analyses of EM samples should follow a written procedure. A technical procedure should contain the following :

- a unique number or combination of unique numbers and letters that serve to identify the procedure;
- a title that is concise but complete enough to identify the nature of the procedure and the matrix or material to which it is applied;
- the purpose, a clear, concise description of why the procedure exists and the desired results of the procedure;
- the applicability of the method to the matrix or sample type;
- responsibilities of all personnel who are assigned an action in the body of the procedure;
- unique terms, defined in a definitions section;
- sample preparation procedures, such as subsampling, addressing the universe of sample matrices and heterogeneity encountered by the laboratory;
- accuracy, precision, and sensitivity of the method;
- calibration and calibration verification frequency for all measurement equipment (instrumentation, balances, pipets, etc.);
- calibration acceptance criteria;
- calibration documentation;
- reference standards;
- instrument performance specifications and proper operating conditions;
- examples of calculations required;
- instrument and method detection limits and linear range of analytical procedures, their method of determination, and their frequency of verification; and
- related QC analysis type, frequency, and acceptance criteria.

2.6 Variances to Standard Operating Procedures

Analyses should be performed in accordance with established and approved SOPs unless specific needs dictate a temporary and immediate variation from the approved SOP. Whenever possible, any variations to approved SOPs should be

approved by the appropriate EMPPM in advance of implementation. When advance approval is not possible, EMPPM should be notified of the variation at the earliest possible opportunity. The reason for the variation and all specific actions associated with the variations to the approved SOP should be documented. All data associated with a method variation or a temporarily modified method should be evaluated for useability based on project DQOs.

2.7 Conditions Adverse to Quality and Corrective Action

A procedure should be developed to identify conditions adverse to quality, such as deviations from Technical Procedure or Standard Operating Procedure requirements, and deficiencies, such as data of indeterminate quality, flawed deliverable reports, or faulty computer software. The procedure should include the following:

- the capability to effectively deal with errors or defects at any point in the generation of data;
- protocols for reporting, format and content of reports, timing of reports and actions, individuals responsible for corrective measures, and lines of communication to management should be included;
- the ability to identify, tally, and track defects to their origin should be implemented in the form of a Deviation Report (DR). The DR should provide for the planning and implementation of measures to correct the identified defects, and to document the results of the corrective actions; and
- the DR should be maintained and controlled by the laboratory QA officer, and documentation of events affecting data should be reported with, and archived in, a controlled environment.

The DRs should contain at least the following information:

- when and where the deviation or event occurred;
- who discovered the deviation or event;
- the name of the individual responsible for the corrective action;
- an explanation for the deviation or event. Copies of relevant information, control charts, sample data, etc. may be included as part of the corrective action report;
- identification of all samples affected. Sample problems and possible effects should be discussed;
- corrective actions should be described and the appropriate EMPPM notification and approval of proposed corrective action obtained;
- corrective action should be implemented, and measures enacted to prevent a recurrence of the condition or event enacted; and

- a tracking system that allows the DR to be brought to closure.

2.8 Document and Record Control Guidelines

A uniform method for the distribution and retention of controlled documents should be established. Distribution is controlled to ensure that only current documents are in use at the work location.

2.8.1 Documentation Control System

A procedure should be developed and implemented to prepare and maintain a controlled distribution list for each controlled document. The procedure should include the following;

- assignment of responsibility for preparing, reviewing, approving, and issuing documents;
- review process for adequacy, completeness, and correctness prior to approval and issuance of document;
- definition of the scope of the document control system;
- documentation of the document control system itself;
- identification of documents to be controlled and their distribution, archival, and disposal;
- control of superseded documents to ensure that only current documents are in use;
- review and approval of major changes to documents by the same organizations/personnel that performed the original review and approval;
- provision of pertinent background data or information to reviewing organizations;
- definition of minor and major changes to documents (i.e. editorial corrections that do not require the same review and approval as the original documents); and
- identification of personnel who can determine what constitutes minor and major changes.

2.8.2 Records Control System

A procedure should be developed to provide a uniform method for the identification, maintenance, storage, disposition and final disposal of records generated by EM programs and projects. The procedure should include the following:

- specification of records of items, data, and processes to be controlled;

- all records which have been designated as quality records by EMPPM;
- preparation, review, approval, and maintenance of records accurately reflecting completed work and fulfilling statutory requirements;
- requirements and responsibilities for record transmittal, distribution, change, retention, protection, preservation, traceability, archival, retrieval, and disposal;
- identification of a records custodian;
- preparation of storage procedures prior to records storage;
  - assignment of responsibility for funding and enforcing of requirements;
- description of the storage facility and the filing system to be used;
- verification that records received are legible and are in agreement with the transmittal document;
- rules governing access to and control of the files;
- procedures for the control of and accountability for records removed from the storage facility;
- procedures for filing of supplemental information and disposing of superseded records;
- storage of records in a manner approved by the organization or organizations responsible for the records;
- construction and maintenance of records storage facilities in a manner that minimizes the risk of damage or destruction from natural disasters; and
- replacement, restoration, or substitution of lost or damaged records.

2.8.3 Documents and Records Retention

The laboratory should establish a procedure that requires that the originals and copies of all data packages, calibration records, and other QA/QC-related records be maintained until such time as they can be destroyed or designated as controlled documents or records.

2.8.4 Data Correction Guidelines

The laboratory should establish a procedure that defines a consistent and approved method of data correction. The procedure should delineate responsibility and the authority required to modify a quality record,

including data, previously accepted as final and complete. Changes or corrections to information, including data entries, notebook and log entries, and computer or data systems output should be corrected by drawing a single line through the incorrect information and initialing and dating the new entry. Correction tape or fluid should not be used. Changes to computerized data records are be identified such that original and corrected entries are retrievable, and the individual initiating the changes can be identified.

2.9 Laboratory Assessments

During the actual performance of laboratory activities, in-process audit/assessment should be performed to assure that the laboratory's activities are being conducted according to approved procedures by qualified personnel using specified equipment. The audit/assessment of the laboratory activities should evaluate, at a minimum, the following subjects:

- Equipment: Measuring and test equipment should meet the applicable standards (e.g., ASTM) or have been evaluated as being acceptable to the procedures, requirements, and specifications.

- Verification of Laboratory Activities: The audits/assessments should be performed to verify that the elements of the laboratory analytical program are in compliance with the applicable technical and quality standards, specifications, and the QAPP and QAPjP requirements. The elements to be verified should include, but are not limited to, the following:

  - implementation of the laboratory QA Program;
  - qualification of laboratory personnel;
  - control and calibration of measuring and test equipment;
  - identification, control, and storage of samples and project documents;
  - implementation and effectiveness of corrective actions;
  - implementation of methods or procedures conforming to applicable specifications and Work Plan requirements; and,
  - documentation and verification of test data, results, conditions, and observations.

- Completeness of Laboratory Records The audit/assessment should determine whether:

  - all samples and analyses required by the QAPP or QAPjP have been processed;
  - complete records exist for each analysis and the associated QC samples;
  - the procedures specified in the QAPP or the QAPjP have been implemented and that changes have been noted according to the established procedures; and,
  - the results of the internal completeness check have been documented, and data affected by incomplete records have

been identified. An assessment of the utility of the analytical results is recorded.

- Evaluation of Data with Respect to Detection Limits: The audit/assessment should compare analytical results to the required detection limits and documents any detection limits that exceed regulatory limits, action levels, or specific project limits, as specified in the QAPP or the QAPjP.

- Evaluation of Data with Respect to Control Limits: The audit/assessment should compare the results of QC and calibration check samples to control criteria. An examination of the deviation reports including the corrective action plans and the results of any re-analyses should be completed for all data not within the control limits. The audit/assessment should determine whether samples associated with ambiguous QC data are identified in a written record of the data review, and whether a review of the utility of such analytical results is recorded.

- Review of Holding Time Data: The audit/assessment should compare sample holding times to those required by the QAPP or the QAPjP. The audit/assessment should determine whether samples associated with deviation from holding time requirements are identified in a written record of the data review, and whether an assessment of the utility of such analytical results is recorded.

- Review of Performance Evaluation Results: The audit/assessment should review documents on internal and external PE studies.

2.10 References

1. American Society of Mechanical Engineers. Quality Assurance Program Requirements for Nuclear Facilities, Supplement 25-1,25-4. Appendix 2A-1 Nonmandatory Guidance on the Qualifications of Inspection and Test Personnel. (ANSI/ASME NQA-1-1986 Ed.), pp. 42-43.

2. U.S. Department of Energy. March 2, 1981. 1324.3, Files Management, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

3. U.S. Department of Energy. September 23, 1986. 5700.6C, Quality Assurance, Assistant Secretary for Environment, Safety and Health, Rev. #3, Washington, DC: Government Printing Office/Document Control. D.C.

4. U.S. Department of Energy. January 6, 1987. 1324.5, Records Management Program, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control. D.C.

5. U.S. Department of Energy. June 8, 1987. HQ 1324.1A, Records Management, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

6. U.S. Department of Energy. September 13, 1988. 1324.2A, Records Disposition, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

7. U.S. Environmental Protection Agency. February, 1983. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans - Interim Final, QAMS 005/80, EPA-600/4-83-004.

8. U.S. Environmental Protection Agency. 1990. Contract Laboratory Program,Statement of Work for Organics Analysis. Document ILM 01.8.

9. U.S. Environmental Protection Agency. 1990. Contract Laboratory Program, Statement of Work for Inorganics Analysis. Document OLM 01.0.

10. U.S. Nuclear Regulatory Commission. February 1979. Quality Assurance for Radiological Monitoring Program (Normal Operations) Effluent Streams and the Environment, Office of Standard Development, Regulatory Guide 4.15, Rev. 1.

3.0 LABORATORY OPERATIONAL GUIDANCE

The applicable EMPPM, in conjunction with personnel knowledgeable in the relevant analytical criteria, should develop, establish, and update requirements for management of sample, waste disposal, chain of custody, laboratory subsampling, holding times, sample containers, standards and reagent control, critical analytical equipment, and preventative maintenance. If it is determined that existing laboratory TPs are sufficient to meet or exceed project needs, new documents need not be developed. For most projects, existing laboratory TPs and SOPs should meet or exceed program or project requirements.

3.1 Management of Samples Received from the Field

DOE EM samples may contain hazardous organic, inorganic, and/or radiochemical materials. Laboratories should be aware of potential hazards associated with the handling, analysis, and disposal of these samples. It is the responsibility of the laboratory to take all necessary precautions to ensure the health and safety of its employees, and to meet regulatory requirements. All sample management procedures should be documented in the TPs and SOPs.

3.1.1 Sample Receipt

The laboratory should establish a procedure describing the receipt of samples. The procedure should designate an individual(s) as a sample receipt custodian. The rigor contained within this procedure should be dependent upon the QAPP and QAPjP. The following areas should be considered within the procedure:

- inspect the shipping container(s) upon receipt;
- sign shipping manifests, and retain copies of these for custody transfer purposes;
- verifiy shipping container contents against the chain of custody form;
- inspect the custody seals and documentation of their condition;
- determine and document the levels of activity of the sample and packing material;
- if the analyses require, determine and document the integrity of the coolant and cooler temperature;
- determine and document the condition of the sample containers (e.g., sample containers, sample containers properly closed, volatile organic containers show no evidence of bubbles, containers appropriately labeled);
- require documentation and notification if samples are damaged or missing; and
- verify and document sample preservation;

The laboratory should maintain a designated area for the receipt and screening of samples. Separate sample receipt areas should be maintained for samples of known radioactivity. Laboratories are responsible for compliance with NRC, their State, and their facility radiation license limits for the receipt of radioactive samples.

Radioactive sample shipments should be accompanied by proper documentation and identification from the shipping source. This information should be reviewed and checked in a consistent manner with all sample shipments. Samples received from known or potentially radioactive sources should undergo screening and inspection for emitted radiation upon receipt. Samples should also be scanned prior to, and after removal from, the shipping container. Procedures containing action levels and appropriate actions should be established by the facility for each step in the screening process. Such actions should include segregation of the samples to radiological zones and internal radiation labeling consistent with radiation policies of the laboratory facility. In some instances, additional isotopic determination may be required prior to introduction of the sample into the laboratory analytical system.

Prior to shipping radioactive samples to the laboratory, the organization responsible for shipping the samples should notify the laboratory of the number and approximate levels of radioactivity in the samples. The laboratory is responsible for assuring that its NRC license limits are not exceeded.

Any breakage information, improper packaging, improper preservation, incorrect labeling, or other irregularities should be identified by the sample receipt custodian and documented. Corrective action necessary to maintain safety requirements and contain the material should be initiated immediately. The laboratory should notify the customer of all problems in shipments to assist in the identification of further corrective actions and appropriate disposition of the samples.

All documentation should be cross-referenced for accuracy and completeness. The documentation may include shipping manifests, chain-of-custody records, sample labels, and pre-receipt information (e.g., scope of work, purchase order, project work plan, telephone conversation record). Information on the receipt of samples should then flow back to the field shipping coordinator, field sampling supervisor, and the project manager to confirm that the correct samples have been received and the proper analysis is being initiated.

3.1.2 Sample Identification

The TPs and SOPs for sample identification should describe methods to assure laboratory samples are identified and controlled in a consistent manner. The procedures should define the responsibilities for documenting identification and tracking sample possession from receipt through handling, storage, transfer, analysis, and disposal.

Sample identification should be transferred to each subdivision, which includes sample splits, sample digestates, and extracts. Verification of sample identification and integrity should be performed (1) prior to release of sample to another organization for testing or analysis, or (2) when samples are subdivided and/or split, and identification is transferred. Verification

should be documented, and appropriate records should be maintained and updated.

3.1.3 Sample Handling, Shipping, and Transfer

All samples should be collected in or transferred to appropriate containers. When acceptable, break-resistant containers should be used.

Samples in glass containers should be transported using secondary containment.

Procedures should be established describing sample collection, handling, shipping, and transfer in accordance with accepted regulatory requirements and guidance. Samples should be controlled during handling and transfer to preclude loss of identity, damage, loss of sample and deterioration. Chain of custody with documentation accompanying the samples must be positively maintained at all times.

The procedure should include requirements for marking, labeling, handling, and the storing of samples.

All packaging and transportation of samples along public roads or in the public domain should be in compliance with DOT regulations and DOE requirements. All other packaging and transportation of samples should be in compliance with DOE requirements. All packaging and transportation of samples should adequately protect personnel, the public, and the environment.

3.1.4 Short-Term Sample Storage

Sample storage SOPs should describe and document the storage conditions for all samples, sample extracts, and digestates. These entities should remain in storage in predetermined physical and environmental conditions commensurate with their intended purpose and consistent with regulatory requirements until acceptance of the final data package by EM.

A procedure should be developed that delineates authority to handle samples, sample extracts, and digestates. Verification and documentation of daily storage temperature should be maintained when appropriate. Measures should be taken to avoid sample contamination during storage, such as separate storage of standards and samples, separate storage of samples and extracts, and separate storage of volatile organic samples from all other samples. Measures should also be taken to contain and avoid material spills during storage. Storage blanks should be used as appropriate (e.g., for volatile organics).

3.1.5 Long-Term Sample Storage

Procedures should be developed describing long-term storage/archival of samples and documenting the storage conditions for all samples, sample extracts, and digestates. These entities should remain in storage in predetermined physical and environmental conditions commensurate with their intended purpose until acceptance of the final data package by EM. Samples may need to be stored permanently in a laboratory under controlled conditions and beyond the acceptance of the final data package by EM.

Long-term storage/archival areas should be controlled to prevent damage and loss, and maintain sample container and identification integrity. The procedure should establish authority to authorize the archival of samples and sample aliquots. The procedure should comply with the appropriate environmental safety and health requirements and policies. Removal of samples from long-term storage/archive should be approved and documented. Long-term storage/archival of samples should be maintained until authorized by the EMPPM for removal and/or disposal. Access to the long-term storage/archival area should be controlled.

3.2 Waste Disposal

During the analytical analyses, waste materials can be generated. The method of identification, storage, and disposal of these waste materials and unused samples should be specified. An effective waste management plan that complies with applicable federal, state, and local regulations should be in place. Policies and guidelines should apply to all personnel who generate, handle, manage, and/or dispose of waste in the laboratory. Specific guidance related to the disposal of excess sample and laboratory generated waste associated with EM programs is being developed.

3.3 Chain-of-Custody

A major consideration for the legal credibility of analytical data is the ability to demonstrate that samples were obtained, reached the laboratory, and were analyzed without improper alteration. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal should be documented. Documentation is accomplished through chain-of-custody procedures and records that describe and document how physical custody is maintained, how custody is transferred, the identity of individuals responsible for sample/sample collection, shipment, receipt, analysis, storage, and disposal. A sample is considered in custody if it is in the person's actual possession, in view after being in physical possession, locked so that no one can tamper with it after having been in physical custody, or in a secured area restricted to authorized personnel.

A procedure should be established by the laboratory describing the interface and custody responsibilities for sample receipt, custody transfer, handling, analysis, storage, and disposal.

Chain-of-custody forms should accompany all EM samples. These forms should be signed and dated upon receipt at the facility. Agreement should be reached between the laboratory and customer regarding disposition of the "original" custody form (i.e., should it be retained by the laboratory, returned immediately to the customer, delivered to the customer as part of the final data deliverable). If copies of the chain-of-custody forms associated with the samples are not maintained as part of the formal analytical data package, the reason for this should be documented by the EM project manager.

Internal chain-of-custody may vary from locked sample custodian control utilizing formal sign-out and sign-in documentation to facilities that maintain restricted access and determine that once the sample is in the facility, they maintain custody. Each facility should establish, document, and implement an internal sample custody SOP.

The procedure should include, in step-wise fashion, procedures used in sample receiving, custody transfer, log-in, tracking the sample, extract, and digestate transfer during preparation and analysis, storage, and eventual disposal of samples, extracts, or digestates, or shipment back to the customer.

3.4 Laboratory Subsampling

Subsampling is a key link in the sampling and analytical chain and can have a substantial impact on the reliability of resulting analytical data. Subsampling is commonly the largest source of error associated with laboratory operations. Thus, it is important and necessary that technical procedures be developed, implemented, and monitored to ensure the use of acceptable subsampling methods.

The laboratory should assume field sampling was completed correctly and that the sample received by the laboratory is representative of the sample population. When information concerning samples indicates those received may not be representative of the sample population (i.e., liquid-solid sample identified as water on the chain-of-custody), the laboratory should contact the customer for clarification.

3.5 Holding Times

Holding times identified in each QAPP or QAPjP for each parameter or group of parameters to be analyzed should be met when implementing work for EM projects.

- Sample shipment and delivery should be coordinated between the field supervisor and the laboratory to meet sample holding times, where applicable.

- If the final reported data resulted from a dilution, re-injection, re-preparation, or re-analysis of the sample, this analysis should have been initiated within the holding time.

- If the laboratory exceeds a holding time, EM management should be notified by the laboratory at the earliest possible opportunity and receive instructions regarding variance procedures and documentation. All data associated with a sample which has exceeded a holding time should be flagged. All reported data associated with a sample which has exceeded a holding time should be evaluated for useability based on project DQOs.

- Although current holding times are regulatory requirements, the analyte-specific impact of holding times is a technical issue that can be negotiated with regulators based on the use of preservatives, etc.

3.6 Sample Containers

A procedure should be developed and in place specifying those types of containers, caps, and liners required for a given analysis or suite of analyses.

When containers are cleaned in the laboratory, blanks should be analyzed for each identified lot of containers to verify and document that the containers are free from contamination for the analytes of interest. The laboratory should establish SOPs for container cleaning and verification.

When commercially precleaned containers are purchased, the manufacturer, lot identification, and certification should be retained for documentation. All containers should be capped and stored in a contaminant-free area.

3.7 Standards and Reagent Control

Procedures should be developed that delineate requirements for standards and reagent control. The procedures should include:

- procurement, preparation, and control of standards and reagents;
- defined requirements in the preparation of standards. Information should include identification of manufacturer, specific grades, purity, activity, concentration, lot number, shelf life, receipt date, preparation procedures and dates, storage of materials used in standard and reagent preparation, appropriate glassware and containers for preparation and storage, labeling, and record keeping for stock solutions and dilutions;
- reference standards should be traceable to nationally-recognized standards or accepted values of natural physical constants. If nationally-recognized standards do not exist, the basis for the reference standard should be documented. Reference standards should be used for calibration and be stored separately from samples;
- the laboratory should maintain documentation of standards and reagents traceability, such as calibration standards, interference check standards, internal standards, surrogate standards, and spike solutions. The laboratory should maintain records for all stock, interim, and working standards employed; and
- all purchased reagents should be of known or proven purity consistent with the intended use. Laboratory reagent screening procedures should ensure materials received are of the purity and specifications required for the intended analysis. Such materials should include laboratory blank water, organic solvents, cleanup column material, etc. All material found to be non-acceptable for the intended use should be clearly labeled and disposed of as soon as possible.

3.8 Preventive Maintenance Program

An adequate preventive maintenance program increases the reliability of a measurement system, and minimizes down time of each measurement system. Procedures should be developed that identify requirements that include the following:

- actions to be taken to maintain proper instrument and equipment performance and prevent instruments and equipment from failing during use; and

- a stock of critical spare parts should be maintained and documented. Preventive maintenance should be scheduled and documented and a maintenance record should be maintained for all instruments and equipment used in the laboratory.

3.9 References

1. American Society of Mechanical Engineers. Quality Assurance Program Requirements for Nuclear Facilities, Supplement 25-1,25-4. Appendix 2A-1 Nonmandatory Guidance on the Qualifications of Inspection and Test Personnel. (ANSI/ASME NQA-1-1986 Ed.), pp. 42-43.

2. Bottrell, D.W., Fisk, J.F., Robertson, G.L., Petty, J.D., Dempsey, C.H. and Bartling, M.L. 1991. "Holding Times of Volatile Organics in Water," *Waste Testing and Quality Assurance: Third Volume, ASTM STP 1075*, D Friedman (editor), Philadelphia, PA: American Society for Testing and Materials.

3. Federal Register. December 3, 1979. 40 CFR Part 136. Proposed Rules. Vol. 44, No. 233:69534.

4. Federal Register. October 26, 1984. 40 CFR Part 136. Rules and Regulations. Vol. 49, No. 209:145.

5. Maskarinec, M.P., et al. Stability of Volatile Organics in Environmental Soil Samples, Final Report ORNL/TM-12128, November 1992, Oak Ridge National Laboratory, Oak Ridge, TN. U.S. Department of Energy Contract No. DE-AC05-840R21400.

6. U.S. Department of Energy. March 2, 1981. 1324.3, Files Management, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

7. U.S. Department of Energy. January 6, 1987. 1324.5, Records Management Program, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

8. U.S. Department of Energy. September 13, 1988. 1324.2A, Records Disposition, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

9. U.S. Department of Energy. October 1992. Methods for Evaluating Environmental and Waste Management Samples, DOE/EM-0089T.

10. U.S. Department of Energy. Radiological and Environmental Sciences Laboratory Manual - Current Revision; Analytical Chemistry Branch Technical Procedures Manual - Current Revision; Laboratory Quality Branch Technical Procedures Manual - Current Revision, Idaho Falls, Idaho.

11. U.S. Environmental Protection Agency. February 1983. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans - Interim Final, QAMS 005/80, EPA-600/4-83-004.

12. U.S. Environmental Protection Agency. 1990. Contract Laboratory Program, Statement of Work for Organics Analysis. Document ILM 01.8.

13. U.S. Environmental Protection Agency. 1990. Contract Laboratory Program, Statement of Work for Inorganics Analysis. Document OLM 01 0.

14. U.S. Nuclear Regulatory Commission. February 1979. Quality Assurance for Radiological Monitoring Program (Normal Operations) Effluent Streams and the Environment, Office of Standard Development, Regulatory Guide 4.15, Rev. 1.

15. Volchok, Herbert L, and de Planque, Gail (editors). U.S. Department of Energy. 1990. Environmental Measurement Laboratory Procedure Manual, 27th Edition, HASL-300, New York, NY.

4.0 LABORATORY QUALITY CONTROL

Laboratory QC is required to assure continuing precision, accuracy, and sensitivity of analytical measurements consistent with the data quality objectives. Acceptance limits for QC measures should be specified as part of the data quality objectives, and corrective actions should be required when these limits are exceeded. Examples of such measures should include, but are not limited to: instrument calibration; internal QC samples, such as surrogate samples, spiked samples, replicates, duplicates, blanks, reference control samples, and standards; and external QC samples, such as PE samples, and referee samples. Quality control requirements may come from methods, e.g., SW-846, or from contracting documents.

Technical procedures should be developed that establish requirements for the relevant analytical criteria which should include:

- updated requirements for calibration;
- method batch and method blanks;
- laboratory control samples, laboratory surrogates, internal standards, laboratory spikes, laboratory duplicates, laboratory splits, and interference check samples; and
- identification of false negatives and positive based on project DQOs.

4.1 Calibration

Technical and operating procedures should be developed and implemented that address and include the following:

- Measuring and testing equipment calibrations should be traceable to nationally-recognized standards. When no nationally-recognized standards exist, the basis for calibrations should be documented.

- Equipment should be calibrated and adjusted prior to use, or maintained at prescribed intervals. The protocol and interval of calibration for equipment should be specified, and based on the type of equipment, stability characteristics, required accuracy, intended use, manufacturer's recommendations, and degree of usage. The date of last calibration, the date of the next calibration, and traceability of calibration data of measuring and test equipment should be maintained as a quality record.

- Validation of test results and chemical analyses require confirmation that all aspects of the process were accurate and corect. Out-of-calibration equipment prevents such confirmation. It is thus essential that when equipment is found to be inoperable or out of calibration, test results and analyses made since the last calibration should be validated and the results recorded. Devices that are out of calibration should be recalibrated or tagged and/or segregated, and not used until they have been recalibrated. If any measuring or testing equipment is consistently found to be out of calibration, it should be repaired or replaced. Calibration should also be performed when the accuracy of equipment is suspect.

- Quality records should be prepared and maintained for each piece of equipment subject to calibration. Quality records demonstrating the accuracy of reference standards should also be maintained.

- Continuous monitoring and periodic calibration should be performed for equipment such as pipets, balances, thermometers, radiation survey instruments, refrigerators and freezers, ovens, and furnaces required in analytical methods, but which are not routinely calibrated as part of the analytical procedure.

- Documentation in the form of a quality record for equipment calibration should be maintained for each item. Calibration requirements should be specified by procedure.

- Operations calibration may be performed as part of the analytical procedure. The analysis of a calibration blank and the preparation of a standard response (standard calibration) curve may be included. Operational calibration is dependent upon specific instrumentation within a laboratory.

4.2 Method Batch

A batch is a number of samples of similar matrix that are processed simultaneously through the entire preparation and analytical process.

4.3 Method Blank

Method blanks are used to determine the existence and magnitude of possible contamination encountered during the entire sample preparation and analysis process. They should be carried through the entire analytical procedure with the samples. Procedures should be developed and implemented that determine the frequency and control limits of method blank analysis consistent with the DQOs and/or contract specifications.

4.4 Laboratory Control Samples

A Laboratory Control Sample (LCS) consists of either a certified reference material or a control matrix spiked with analytes representative of the target analytes. LCSs are used to verify that precision and bias of the analytical process are within control limits. The LCS matrix should be comparable to the sample matrix. Procedures should be developed and implemented that determine the frequency and control limits of LCS analysis that are consistent with the DQOs.

The purpose of a LCS program is to demonstrate that the laboratory process for sample preparation and analysis is in control. LCS information, used in conjunction with sample matrix spike recoveries, can be used as a quality control measure. The LCS results should be monitored through the use of control charts. Results of the LCSs may be compared to control limits established for both precision and bias to determine usability of the data.

4.5 Laboratory Surrogate and Internal Standards

A surrogate standard consists of spiking samples and blanks with known concentrations of certified analytes before analysis of samples. Procedures should be developed and implemented that determine the frequency and control limits of surrogate standard analysis that are consistent with the DQOs. The procedures should clearly define all related calculations, acceptance criteria, and implementation required to produce the final quality data result.

The procedures should include:

- surrogate standard determinations should be performed on all samples and blanks for Gas Chromatography/Mass Spectroscopy (GC/MS) analyses;
- all recoveries should meet predetermined acceptance criteria (e.g. DQOs) that are monitored as laboratory results become available; and
- internal standards should be employed in several methods to determine the specific procedural recovery of an analyte group or analyte.

4.6 Laboratory Matrix Spikes and Laboratory Matrix Spike Duplicates

A Matrix Spike (MS) is an aliquot of a sample spiked with known quantities of analytes and subjected to the entire analytical procedure. It is used as a measure of recovery or bias.

A Matrix Spike Duplicate (MSD) is a second aliquot of the same sample as the MS, with the same known quantities of analytes added as the MS. The purpose of the MSD, when compared to the MS, is to estimate method precision. The use of these samples has minimal technical application. They should be considered only if a specific regulatory requirement dictates their use.

4.7 Laboratory Duplicate Analyses

A laboratory duplicate is defined as a subsampling of a homogeneous sample into two separate subsamples for method preparation and analysis, or the initial subsampling of a non-homogeneous sample which has been homogenized and then further divided into two separate subsamples for method preparation and analysis. The purpose of the laboratory duplicate is to test for method precision. Procedures should be developed and implemented that determine the frequency and control limits of laboratory duplicate analysis consistent with DQOs.

4.8 Laboratory Split Analyses

Laboratory splits are two separate, non-homogenized, subsamples of an individual sample analyzed by the laboratory to assess sample homogeneity. The sample should be split in the laboratory prior to sample analytical preparation. Procedures should be developed and implemented that determine the frequency and control limits of laboratory split analyses that are

consistent with the DQOs. Because of the homogenization process, splits and duplicates are distinctly different and must not be confused in their application.

4.9 Interference Check Samples

An Interference Check Sample (ICS) consists of two subsamples of either a certified reference material or a control matrix. One subsample is spiked with analytes representative of the interfering analytes. The second subsample is spiked with the interfering and target analytes. ICSs are used to verify that inter-elemental correction factors applied to the analytical process are within control limits. The ICS matrix should be comparable to the sample matrix. ICSs are used mainly in Inductively Coupled Plasma (ICP) analyses. Procedures should be written and implemented that define the frequency and control limits of ICS analysis.

The purpose of an ICS program is to demonstrate that the laboratory process for sample analysis is in control. ICS information can be used as a quality control measure aid to detect changing instrument conditions. The ICS results should be monitored though the use of control charts. Results of the ICSs may be compared to control limits to monitor instrument performance.

4.10 Identification of False Positive, False Above-Decision-Threshold (ADT), False Negative, and False Below-Decision-Threshold (BDT) Data

Technical and administrative procedures and subject white papers should be prepared discussing the significance of the potential for producing, and means of controlling false negative, false Below-Decision-Threshold (BDT), false positive, and false Above-Decision-Threshold (ADT) data. The procedures should describe corrective actions for dealing with suspected false results.

4.11 References

1. American Society of Mechanical Engineers. 1986. Quality Assurance Program Requirements for Nuclear Facilities. Supplement 25-1,25-4. Appendix 2A-1 Nonmandatory Guidance on the Qualifications of Inspection and Test Personnel. New York, NY. (ANSI/ASME NQA-1-1986 Ed.) pp. 42-43.

2. U.S. Department of Energy. Radiological and Environmental Sciences Laboratory Manual - Current Revision; Analytical Chemistry Branch Technical Procedures Manual - Current Revision; Laboratory Quality Branch Technical Procedures Manual - Current Revision. Idaho Falls, Idaho.

3. U.S. Environmental Protection Agency. 1984. Soil Sampling Quality Assurance Users Guide (1st. Edition). Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. EPA/600/4-84-043

4. U.S. Environmental Protection Agency. 1989. Soil Sampling Quality Assurance Users Guide (2nd. Edition). Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. EPA/600/8/89/046

5. U.S. Environmental Protection Agency. May 1990. A Rationale for the Assessment of Errors in the Sampling of Soils. Environmental Monitoring Systems Laboratory, Las Vegas, NV. EPA/600/4-90/013

6. U.S. Environmental Protection Agency. 1990. Contract Laboratory Program, Statement of Work for Organics Analysis. Document ILM 01.8.

7. U.S. Environmental Protection Agency. 1990. Contract Laboratory Program, Statement of Work for Inorganics Analysis. Document OLM 01.0.

8. U.S. Environmental Protection Agency. November 1991. Description and Sampling of Contaminated Soils, A Field Pocket Guide. Technology Transfer. EPA/625/12/91/002

9. U.S. Environmental Protection Agency. July 1992. Test Methods for Evaluating Solid Waste, 3rd Edition, Office of Solid Waste and Emergency Response, EPA-SW-846.

10. U.S. Nuclear Regulatory Commission. February 1979. Quality Assurance for Radiological Monitoring Program (Normal Operations) Effluent Streams and the Environment, Office of Standard Development, Regulatory Guide 4.15, Rev. 1.

11. Volchok, Herbert L., and de Planque, Gail, Editors. Environment Measurement Laboratory Procedure Manual, 1990, 27th Edition, U.S. Department of Energy HASL-300, New York, NY.

5.0 MONITORING LABORATORY QUALITY CONTROL

Administrative procedures should be developed that establish requirements for the update control criteria requirements for all technical and administrative procedures related to the analytical process based on an approved quality assurance program. Control criteria and charting should be established to evaluate laboratory precision, bias, and trends associated with analyses. Documented procedures should be in place to demonstrate that the laboratory is in control during each data collection activity. Most analytical laboratory's methods have established control limits.

5.1 Control Criteria

Procedures should be developed that contain the following control criteria requirements:

- sample receipt temperature controls;
- storage temperature controls;
- sample preparation temperature controls;
- method-specific blank contamination controls;
- instrument-specific calibration controls;
- measuring equipment calibration controls; and
- QC sample criteria controls including LCS, surrogates, and ICS controls.

Control limits may be based on internal or published external requirements and guidelines, other regulatory criteria where they exist, and/or specific project requirements and DQOs. Laboratory-specific statistically based criteria should be established to ensure quality control. Laboratory-specific criteria should normally be more stringent than those established by multi-laboratory national program criteria.

The procedures should establish the formulas used for calculation of control sample limits; if appropriate, the statistical methods used to derive the limits should be fully referenced.

When QC results or other operating conditions fall outside established control criteria, concurrently generated data are considered suspect and should be repeated or reported with qualifiers. Data generated under these conditions should be communicated to EMPPM for resolution regarding their impact on achieving project data quality objectives and resultant data quality.

If a software program is used that is not capable of monitoring data that are outside these criteria, it is the responsibility of laboratory personnel to establish quality control procedures to monitor these conditions manually.

5.2 Control Charting

Technical procedures should be developed and implemented that establish requirements for control charting and criteria that are consistent with the DQOs. Procedures should be in place to demonstrate that the laboratory is in control during each data collection activity. If existing laboratory control charting requirements and criteria are sufficient to meet or exceed project needs, new control charting documents need not be developed. For most projects, existing laboratory control charting requirements and criteria should meet or exceed project requirements.

Control charts provide a useful tool in assessing analytical performance through graphic display of a parameter's variability over time. The parameter plotted on the chart is related to control sample testing, either directly, in terms of concentration, or indirectly, in terms of derived information (i.e., means, ranges, percent recoveries, relative percent differences, or slopes of least square data fits).

Control charts graphically follow the quality of sample analysis by testing a control sample to determine whether reproducible and accurate results are being obtained. Control charts usually consist of a graph showing time on the abscissa and control results on the ordinate.

The procedures should include the following:

- which control parameters are to be plotted;
- the number of controls to be analyzed per run sequence;
- statistical/mathematical basis for establishing and updating warning and control limits; and
- how to identify shifts and trends that may be revealed by these charts.

Administration of control charts requires consideration of the following aspects:

- the types of activities control charts monitor;
- personnel responsible for maintaining and updating charts;
- personnel responsible for control chart oversight; and
- how changes in personnel, equipment, or processes affect existing charts.

The procedures should include the generation of a QC control chart for each method of analysis and sample matrix. These charts should monitor laboratory measurements obtained from the QC samples. Each control chart should consist of a statistically-derived target value, warning limits, and control limits. Control charts should be maintained on a real-time basis by the analyst performing the analysis.

5.3 References

1. American Society for Testing and Materials. 1977. Standard Practices for Sampling Water, Method D 3370-76, Annual Book of ASTM Standard (Part 31), Water, Philadelphia, PA: ASTM.

2. American Society for Testing and Materials. 1983. Sampling Surface Soils for Radionuclides, ASTM C-998-83.

3. Box, G.E.P. 1978. Statistics for Experimenters. John Wiley & Sons, New York.

4. Cochran, W.G. and G.W. Snedecor. 1977. Statistical Methods. Iowa State University Press, Ames, IA.

5. Duncan, A.J. 1974. Quality Control and Industrial Statistics. Dow Jones Irwin, Homewood, IL.

6. Grant, E.L., and R. Leavenworth. 1988. Statistical Quality Control. McGraw-Hill, New York.

7. Helsel, D.R. 1990. Less than obvious: Statistical treatment of data below the detection limit. Envir. Sci. & Tech. 24:1766-1774.

8. Lambert, D., B. Peterson, and I. Terpenning. 1991. Nondetects, detection limits, and the probability of detection. Jour. Amer. Stat. Assoc. 86:266-277.

9. Taylor, John K. 1988. Quality Assurance of Chemical Measurements. Chelsea, MI: Lewis Publishers, Inc.

10. U.S. Department of Energy. Radiological and Environmental Sciences Laboratory Manual - Current Revision; Analytical Chemistry Branch Technical Procedures Manual - Current Revision; Laboratory Quality Branch Technical Procedures Manual - Current Revision, Idaho Falls, Idaho.

11. U.S. Environmental Protection Agency. July 1992. Test Methods for Evaluating Solid Waste, 3rd Edition, Office of Solid Waste and Emergency Response, EPA-SW-846.

12. Volchok, Herbert L., and de Planque, Gail, editors. Environmental Measurement Laboratory Procedure Manual, 1990, 27th Edition, U.S. Department of Energy HASL-300, New York, NY.

6.0 LABORATORY DATA

Administrative procedures, including QAP, that define requirements for the update of data deliverables based on the DQOs should be developed and implemented. The procedures should identify and clearly define specific data deliverables expected from the analytical laboratories supporting its work. These deliverables should be designed to ensure that the information contains the appropriate QC and documentation.

6.1 Laboratory Data Review/Verification

Procedures should be in place defining requirements for data review. The data review should constitute technical verification of raw data information by an individual or individuals other than the original data generator. The laboratory manager has the ultimate responsibility of ensuring that data reported are of known quality and meet technical or contractual requirements. Laboratory supervisors should be responsible for ensuring QC procedures have been followed and for approving all data reported from their section of the facility. Chemists and technicians should have the responsibility for analyzing samples employing designated methodologies, performing all related QC functions, personally reviewing their data and calculations, entering of data into the laboratory's data management system (electronic or hard copy) and, when required, responding to nonconforming data or QC analysis. The laboratory QA function should hold responsibility to oversee the review process and review a percentage of data, based on the data confidence required. Generally accepted laboratory practice is that the following areas are considered when developing data review requirements:

- percentage of data to be reviewed;
- type of data to be reviewed (e.g., final results, raw data, calculations);
- verification that reported results, existing raw data, and related QA/QC information (e.g., calibration, blanks, spikes, duplicated) conform to prescribed sensitivity, accuracy, precision, and any other criteria established to meet the needs of the customer;
- verification that instrument conditions (e.g., calibration curve, response factors) conform to prescribed standards established to meet the needs of the customer;
- level(s) of review (e.g., analytical peer, supervisor, QA function);
- confirmation that results are representative of the sample received;
- confirmation of analytical consistency and completeness; and
- conformation of data package consistency and completeness.

It should be understood that data verified through the laboratory's internal review procedures are not validated data. The purpose of data verification is

for the laboratory organization to internally ensure that: the data meet data validation criteria, and that errors are minimized and clearly identified when detected, and corrected or reconciled prior to delivering the information to the client.

6.2 Data Validation

Data validation is the systematic and independent review of data quality. It requires defined acceptance criteria to provide assurance that the data are adequate for the intended use. Procedures and controls based on the DQOs should define the data validation requirements. The laboratory should be made aware that EMPPM may have established data validation criteria to use for review of analytical information to determine the data useability in relation to the project requirements and objectives. It is in both the DOE's and the laboratory's best interest to be cognizant of project objectives and establish laboratory data review requirements and project data validation requirements and criteria that are consistent and that deliver a product of the quality expected. Since the process of data validation is not a laboratory function and is therefore outside the scope of this document, a technical discussion of data validation issues is not herein presented.

Based on data significance and it's intended use, EMPPM may wish to establish multiple levels of validation requirements. Data of a sensitive nature requiring a high level of confidence may warrant 100 percent validation of reported information, including all raw data and calculations. Data of low sensitivity may require only a percentage of raw data and calculations reviewed for consistency and completeness, and a minimal QA review.

6.3 Data Reporting Criteria

Deliverables may include a diskette. Reporting formats should be compatible with the derived DQOs and contractual requirements. Developing programs to standardize data generation, reporting, transmission, and storage within EM and across agencies are current Interagency activities. These products will ultimately be introduced and implemented through the Field Office and local sample management offices.

The following sections briefly describe generic types of deliverables being requested for environmental projects.

6.3.1 Data Deliverable - Qualitative Results

This type of deliverable may not require a formal, written narrative or inclusion of QC information. It can comprise a list of sample results and concentration units (when applicable) versus customer sample identification. Results should be presented in a clear and logical format. All QC information generated by the laboratory should be held in the laboratory as backup documentation.

Deliverables of this type receive little or no data validation by the project. The project may only review the data for completeness and consistency with other project information.

6.3.2 Data Deliverable - Quantitative Results

This type of deliverable may contain completed sample results plus specific QC sample results as defined by project management. Potential QC sample results to be included may be laboratory blank, duplicate, MS or LCS analyses. Results should be reported in a clear and logical format. Sample data forms should be submitted for approval by both EM project management and analytical laboratory management. Data should be reported with any applicable laboratory review qualifications, and a case narrative should accompany data expressing any pertinent comments by the laboratory regarding data quality. All other information generated by the laboratory, including logbook entries, instrument records, work sheets, calibration data, non-reported QC data, and documentation of communications should be available in the laboratory.

Depending on the degree of information requested in this type of deliverable, validation should basically follow a contract compliance review format. Normally, this deliverable would not contain calibration information, and recalculation of reported data would not be part of the validation process. Therefore, minimal validation of this laboratory data deliverable may be possible.

6.3.3 Standard Quantitative Data Deliverable

This deliverable constitutes a comprehensive report of all laboratory-generated results and QC information. It does not include analytical raw data information such as laboratory notebook pages, analytical instrument output, data work sheets, or documentation of communications. It should comprise a formally formatted data information deliverable that includes: analytical holding time information, specified analytical methods, signed chain-of-custody documentation, laboratory analytical case narrative with problems and corrective actions, LCS analysis with control chart status, sample results, surrogate or tracer recoveries, MS data, method blank data, initial and continuing instrument calibration information, internal standard information, confirmation analysis when required by the method, analytical run sequences, and method specific QC information (i.e., ICP interference check sample data, post digestion spike recovery, method of standard additions information, instrument efficiency checks, method self absorption factors, MS tune, and GC retention time). The form and content of this deliverable should be thoroughly developed, reviewed, and approved by both the responsible analytical laboratory and the EM project prior to initiation of work. All raw data information and records documentation generated by the analytical laboratory should be available at the laboratory.

This deliverable enables the customer to review a comprehensive summary of analytical, calibration, and QC data for the project. However, it does not allow a comprehensive recreation of data from raw data deliverables. Validation, therefore, constitutes more than a contract compliance review, but less than complete independent reconstruction of reported data.

6.3.4 Complete Documentation of Quantitative Data Deliverable

This type of deliverable should be in the form of a comprehensive report of all laboratory-generated results, all QC information, and all raw data

information and records. The report should include all backup raw data produced by the laboratory.

This deliverable should be designed to allow the customer to fully recreate the process that generated each analytical data point reported. Therefore, a complete independent data validation of this information may be performed. This places the comprehensive data information record with the customer for future reference and defense.

6.3.5 Diskettes

Deliverables may include a diskette. Reporting formats should be compatible with the project's system. Standard formats for transmission and database structure requirement should include consistency with interagency standards for collecting, storing, transmitting, and evaluating environmental data.

6.4 References

1. U.S. Department of Energy. March 2, 1981. Order Number 1324.3, Files Management, Office of Administration and Human Resource Management, Washington, DC: Government Printing Office/Document Control.

2. U.S. Environmental Protection Agency. February 1983. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, Interim Final, QAMS 005/80, EPA-600/4-83-004.

3. U.S. Environmental Protection Agency. April 1992. Guidance for Data Useability in Risk Assessment (Part A) Final. Office of Emergency and Remedial Response, Washington, DC. 9285.7-09A.

4. U.S. Environmental Protection Agency. January 1993. Guidance for Data Useability in Site Assessment - Interim Final. Office of Emergency and Remedial Response, Washington, DC. 9345.1-06.

APPENDIX I

DEFINITIONS

conditions adverse to quality. An all-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, and nonconformances.

corrective action. Measures taken to rectify conditions adverse to quality and, where necessary, to preclude repetition.

data validation. A systematic and independent review of data quality. It requires defined acceptance criteria to provide assurance that the data are adequate for the intended use.

data verification. An on-going, routine activity checking to ensure that data have been accurately quantified, recorded and transcribed and that required procedures were followed.

deviation. The departure from specified requirements.

document control. The act of assuring that documents are reviewed for adequacy, approved for release by authorized personnel, and distributed to and used at the location where the prescribed activity is performed.

laboratory duplicate. An initial subsample of a sample which has been homogenized and then further divided into two separate subsamples, and then subjected to the entire analytical procedure after being received by the laboratory.

laboratory matrix spike. (MS) An aliquot of a sample spiked with known quantities of compounds and subjected to the entire analytical procedure after being received by the laboratory.

laboratory matrix spike duplicate. (MSD) A second aliquot of the same sample as the Matrix Spike (MS), with the same known quantities of compounds added as the MS and subjected to the entire analytical procedure after being received by the laboratory.

laboratory splits. Two separate, non-homogenized, subsamples of an individual sample subjected to the entire analytical procedure after being received by the laboratory.

quality assurance program plan. An orderly assemblage of management policies, objectives, principles, and general procedures by which an agency or laboratory outlines how it intends to produce data of known or accepted quality.

quality assurance project plan. An orderly assembly of detailed and specific procedures which delineates how data of known and accepted quality is produced for a specific project. (A given agency or laboratory would have only one quality assurance program plan, but would have a quality assurance project plan for each of its projects.)

APPENDIX II

LIST OF ACRONYMS

ADT	-	Above Decision Threshold
ASD	-	Analytical Services Division
ASP	-	Analytical Services Program
ASTM	-	American Society for Testing and Materials
BDT	-	Below Decision Threshold
DOE	-	Department of Energy
DOT	-	Department of Transportation
DQO	-	Data Quality Objective
DR	-	Deviation Report
EM	-	Environmental Restoration and Waste Management
EMPPM	-	EM Program or Project Management
EPA	-	U.S. Environmental Protection Agency
ESAA	-	Environmental Sampling and Analysis Activities
GC/MS	-	Gas Chromatography/Mass Spectroscopy
ICP	-	Inductively Coupled Plasma
ICS	-	Interference Check Sample
IG	-	Office of the Inspector General
LCS	-	Laboratory Control Sample
MS	-	Matrix Spike
MSD	-	Matrix Spike Duplicate
OSHA	-	Occupational Safety and Health Administration
PE	-	Performance Evaluation
QA	-	Quality Assurance
QAPjP	-	Quality Assurance Project Plan
QA/QC	-	Quality Assurance/Quality Control
QAMS	-	Quality Assurance Management Staff
QAP	-	Quality Assurance Program
QAPP	-	Quality Assurance Program Plan
QARD	-	Quality Assurance Requirements and Description
QC	-	Quality Control
SA	-	Sampling and Analysis
SEN	-	Secretary of Energy Notice
SOP	-	Standard Operating Procedure
TQM	-	Total Quality Management
TP	-	Technical Procedure