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Distribution	R. R. Grabbe, 373-7185	Date March 2, 1995
Project Title/Work Order		EDT No. 610060
Quality Assurance Program Plan For the Waste Sampling and Characterization Facility		ECN No. N/A

Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
Central Files (2)	L8-04	X			
O.S.T.I. (2)	L8-07	X			
RRG File	S3-28	X			

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MAR 02 1995

ENGINEERING DATA TRANSMITTAL

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1. EDT 610060

2. To: (Receiving Organization)	3. From: (Originating Organization)	4. Related EDT No.:
Low-Level Laboratories Waste Sampling and Characterization Facility	Low-Level Laboratories Waste Sampling and Characterization Facility	N/A
5. Proj./Prog./Dept./Div.: Project Site and Services/Analtyical Services	6. Cog. Engr.: N/A	7. Purchase Order No.: N/A
8. Originator Remarks:		9. Equip./Component No.: N/A
		10. System/Bldg./Facility: Building 6266/WSCF
11. Receiver Remarks:		12. Major Assm. Dwg. No.: N/A
		13. Permit/Permit Application No.: N/A
		14. Required Response Date:

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	WHC-SD-CP-QAPP-017		0	Quality Assurance Program Plan For the Waste Sampling and Characterization Facility	Q	2		

16. KEY

Approval Designator (F)	Reason for Transmittal (G)	Disposition (H) & (I)
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 4. Review 2. Release 5. Post-Review 3. Information 6. Dist. (Receipt Acknow. Required)	1. Approved 4. Reviewed no/comment 2. Approved w/comment 5. Reviewed w/comment 3. Disapproved w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION (See Approval Designator for required signatures)

(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp.
1	1	Cog.Eng. H. K. Meznarich	H Meznarich		T6-16						
1	1	Cog. Mgr. R. R. Grabbe	Robert R Grabbe	3-2-95	S3-28						
1	1	QA T.L. Tung	Tyn Li Tung	3/2/95	S3-30						
		Safety: N/A									
		Env. N/A									

18.	19.	20.	21. DOE APPROVAL (if required) Ctrl. No.
Signature of EDT Originator / Date	Authorized Representative for Receiving Organization / Date	see bl. #17 Cognizant Manager R.R. Grabbe / Date	[ ] Approved [ ] Approved w/comments [ ] Disapproved w/comments

BD-7400-172-1

RELEASE AUTHORIZATION

Document Number:	WHC-SD-CP-QAPP-017, REV 0
Document Title:	Quality Assurance Program Plan For the Waste Sampling and Characterization Facility
Release Date:	3/2/95

This document was reviewed following the procedures described in WHC-CM-3-4 and is:

APPROVED FOR PUBLIC RELEASE

WHC Information Release Administration Specialist:

Kara M. Broz — March 2, 1995

Kara M. Broz

102 27

SUPPORTING DOCUMENT

1. Total Pages ~~106~~

2. Title	3. Number	4. Rev No.
Quality Assurance Program Plan For the Waste Sampling and Characterization Facility	WHC-SD-CP-QAPP-0017	0

5. Key Words	6. Author
Quality Assurance Program Plan (QAPP) For the Waste Sampling and Characterization Facility (WSCF)	Name: R. R. Grabbe, Manager Signature 3-2-95 Organization/Charge Code 8E600/YL52C

7. Abstract

This document provides the Quality Assurance guidelines for all personnel in the 600 Area Waste Sampling and Characterization Facility.

8. RELEASE STAMP

OFFICIAL RELEASE (2)
BY WHC
DATE MAR 0 2 1995

Quality Assurance Program Plan For the Waste Sampling and Characterization Facility

Prepared by

Waste Sampling and Characterization Facility

Issued by

Waste Sampling and Characterization Facility

Approved for Public Release

QUALITY ASSURANCE PROGRAM PLAN FOR LABORATORY ANALYSES AND PROCESS TESTING

Approvals:

R. R. Grabbe, Manager
Low-Level Laboratories
Waste Sampling and Characterization Facility

3-2-95
Date

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APPENDIX LIST

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LISTING OF ACRONYMS

%D	percent difference
%R	percent recovery
AEQA	Analytical and Environmental Quality Assurance
AS	Analytical Services
ASTM	American Society for Testing Materials
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	U.S. Code of Federal Regulations
CLP	Contract Laboratory Program
DLR	decision level count rate
DOE	U.S. Department of Energy
DQO	data quality objective
ECN	Engineering Change Notice
EDC	Engineering Document Control
EPA/USEPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
GFAA	Graphite Furnace Atomic Absorption Spectroscopy
HASQAP	Hanford Analytical Services Quality Assurance Plan
HEPA	high efficiency particulate air
HVAC	heating, ventilation, and air conditioning system
ICP	inductively coupled plasma spectrometer
ICV	initial calibration verification
IDL	instrument detection limit
LA	Laboratory Analytical Procedures
LIMS	laboratory information measurement system
LO	Laboratory Operating Procedure
LQA	Laboratory Quality Assurance
LR	Laboratory Reference Material Specification
LSC	Liquid Scintillation Counter
MDA	minimum detectable activity
MDC	minimum detectable concentration
MDL	method detection limit
NIST	National Institute of Standards and Technology (formerly the National Bureau of Standards)
OJT	on-the-job training
OQA	Office of Quality Assessment
PRAF	procedure review and approval form
QA	quality assurance
QAMS	Quality Assurance Management Staff
QAPP	quality assurance program plan
QAP_jP	quality assurance project plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RF	response factor
RPD	relative percent difference
RSD	relative standard deviation
SD	Supporting Documents
WHC	Westinghouse Hanford Company
WSCF	Waste Sampling and Characterization Facility

QUALITY ASSURANCE PROGRAM PLAN IDENTIFICATION FORM

DOCUMENT TITLE: Quality Assurance Program Plan for The Waste Sampling and Characterization Facility

DOCUMENT CONTROL NUMBER: WHC-SD-CP-QAPP-0017, Revision 0

ORGANIZATION TITLE: Analytical Services

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PLAN COVERAGE:

Analytical Operation Organizations

Waste Sampling and Characterization Facility

Inorganic Chemistry, Radiochemistry, Organic Chemistry, Environmental Analytical Laboratories

ANALYTICAL CHEMISTRY SERVICES LABORATORIES QUALITY ASSURANCE PLAN

1.0 INTRODUCTION

The objective of this Quality Assurance Plan is to provide quality assurance (QA) guidance, implementation of regulatory QA requirements, and quality control (QC) specifications for analytical service. This document follows the Department of Energy (DOE)-issued Hanford Analytical Services Quality Assurance Plan (HASQAP) and additional federal [10 U.S. Code of Federal Regulations (CFR) 830.120] QA requirements that HASQAP does not cover.

This document describes how the laboratory implements QA requirements to meet the federal or state requirements, provides what are the default QC specifications, and/or identifies the procedural information that governs how the laboratory operates. In addition, this document meets the objectives of the Quality Assurance Program provided in the WHC-CM-4-2, Section 2.1. This document also covers QA elements that are required in the Guidelines and Specifications for Preparing Quality Assurance Program Plans (QAPPs), (QAMS-004), and Interim Guidelines and Specifications for Preparing Quality Assurance Product Plans (QAMS-005) from the Environmental Protection Agency (EPA). A QA Index is provided in the Appendix A.

The personnel of the Waste Sampling and Characterization Facility (WSCF) including managers, analysts, QA/QC staff, auditor, and support staff shall use this document as guidance and instructions for their operational and QA activities. Other organizations that conduct activities described in this document for the WSCF laboratory shall follow this QA/QC document.

Sample analysis under regulatory requirements including , Clean Water Act, National Emission Standards for Hazardous Air Pollutants, National Pollution Discharge Elimination System, and Industrial Hygiene Quality Assurance Requirements shall meet or exceed the QA protocols described in this document.

The WSCF organization provides analytical services which include Counting Room support for stack and room air monitoring and low-level environmental samples; performing analysis of QC split samples for commercial laboratories that are used for the analysis of low-level environmental samples; process control support for new liquid effluent treatment systems; and environmental monitoring programs.

A graded approach is applied on the level of QC, data verification, and data reporting to meet specific needs of the client. Unique QA/QC requirements that differ from this document shall be described in the work statement, analysis plan, or Quality Assurance Project Plan (QAP_jP) for the laboratory to follow.

This document supersedes WHC-SD-CP-QAPP-003 and shall be reviewed annually and revised as appropriate.

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2.0 ORGANIZATION AND RESPONSIBILITY

The charters and responsibilities for each organizations are published in Section 2 of WHC-CM-5-4.

2.1 Management Policy

The policy of the WSCF management is to direct activities in a manner that ensures the results meet or exceed the customer's requirements and provides supporting documentation. This policy shall be implemented through the following:

- Personnel are responsible for the quality of their own work. Personnel shall check items supplied for their work process to ascertain that the items are correct and suitable for use;

  All levels of management accept responsibility for their organization's activities and are held accountable for achieving quality in them;

- Management provides adequate resources and budget to support effective QA practices which fulfill the customer's program goals and performance objectives;

- Management provides facilities, instruments, support equipment, and materials required to meet the customer's current and projected requirements;

- Laboratory personnel have acceptable qualifications and training for their specific job assignment;

- Documentation is controlled and maintained in a manner that ensures that the laboratories can demonstrate compliance to customers' requirements;

- Quality is achieved and improved by planned, systematic and self-assessments, and measured actions; and

- Quality control data documents the accuracy and precision performance of instruments and methods.

2.2 Organization

2.2.1 Structure

WSCF is located within Analytical Services (AS) Division, which is one of the Project & Site Divisions. The Vice President of Projects and Site Division reports directly to the President of Westinghouse Hanford Company (WHC).

The organizational structure for AS and WSCF is Appendix B. Charters for AS and WSCF can be found in WHC-CM-1 and WHC-CM-5-4, respectively.

2.2.2 Functional Responsibilities

The WSCF organization provides analytical services which include Counting Room support for stack and room air monitoring and low-level environmental samples; performing analysis of QC split samples for commercial laboratories that are used for the analysis of low-level environmental samples; and process control support for new liquid effluent treatment systems.

2.2.3 By Organization

2.2.3.1 WSCF Analytical Laboratory Facilities Section

The analytical services provided by WSCF analytical lab include analyses of 200- and 300-Area liquid effluents, environmental monitoring programs, and QA oversight of commercial labs.

The Building Operations Unit provides services to the WSCF that are performed by Plant Operations groups in operating plant facilities, (e.g., landlord services.) This unit also provides stockroom support.

2.2.4 By Position

All laboratory personnel are responsible for implementation of the QA practices described in this document and prescribed in the company manuals applicable to their work assignments.

2.2.4.1 WSCF Manager

The WSCF Manager is responsible for the WSCF, laboratory managers, QA officer, facility operations manager, sample custodian, administrative personnel, budget, all analytical data, and for developing agreement with customers and ensuring that reported data meet customer's data quality objectives (DQOs).

Quality Assurance Officer - The WSCF QA officer is responsible to 1) oversee the QA program in WSCF; 2) review analytical laboratory personnel training and qualifications; 3) periodically monitor analytical instrument performance; 4) evaluate and verify data quality through spot-checking; 5) periodically review and summarize QC reports; 6) perform audits/assessment and recommend correction actions; 7) coordinate external auditing between laboratory personnel and external agencies for overall systemic operations; and 8) coordinate performance evaluation programs.

2.2.4.2 Laboratory Managers

Laboratory managers are responsible for the coordinated operation of the laboratories. This includes financial planning and reporting, adjusting the manpower to meet changing work needs, selection of personnel, integration of manpower with other sections, and assuring that the various training requirements are met. Each manager has the responsibility of implementing the QC activities in his laboratory group.

2.2.4.3 Scientists

Scientists and managers shall complete facility specific training. Laboratory familiarization training is recommended for new scientists that will perform analytical procedures in the laboratory. Scientists are responsible for writing purchase specifications and setting-up and maintaining operability of analytical instrumentation. They develop the analytical methods and write the procedures used in analyzing samples. They provide an overview of method performance and QC. Scientists work directly with the managers and laboratory customers to provide technical support in the evaluation of data and the selection of the best analytical method to use in analyzing samples. They assist in the training of personnel particularly in areas of high technology.

General descriptions delineating qualifications (training and experience) for scientists are maintained by Human Resources.

2.2.4.4 Bargaining Unit Personnel

The senior chemical technologists and the chemical technologists are members of the Bargaining Unit and as such perform work in accordance with the current labor contract.

2.2.4.5 Requesters/Sample Suppliers

Suppliers of samples are responsible for taking samples, identifying samples, designating required analyses, and providing adequate sample information to WSCF. The reliability and traceability of samples from generation to submittal to the laboratory are maintained by the supplier. Upon receipt, WSCF is responsible for maintaining reliability and traceability through proper handling and storage practices. Although responsibility for sampling and sampling plans rests with the requestor, assistance in developing plans may come from WSCF.

2.2.5 By Supporting Organizations

The following services are provided by organizations either within the AS organization or outside the AS organization to achieve QA program for the WSCF Laboratory Operations.

2.2.5.1 Program Management & Integration

The Program Management & Integration (PM&I) organization includes Documentation Administration and Office of Quality Assessment (OQA) that provide services to the WSCF. The PM&I Documentation Administration provides services including records management, administration of laboratory procedures and the WHC-CM-5-4 manual, maintenance of the Laboratory Technical Information Center.

2.2.5.2 Office of Quality Assessment

The OQA organization provides QA oversight through assessments for AS. The OQA is responsible for annual auditing of WSCF.

2.2.5.3 Engineering and Technology Services

The Engineering and Technology Services organization provides engineering services, operational assurance and support, chemical standards services, information and automated data management systems, AS maintenance, and work control.

The Information Systems is responsible for developing and maintaining the automated data management system, e.g., LABCORE. The LABCORE is in the early implementation stage and is scheduled to be implemented by the end of September 1995. The Standard Laboratory is responsible for procuring and preparing chemicals, standards, and reagents. The Laboratory Engineering organization is responsible for calibration of balances and conducting instrument preventive maintenance per request. The Operations Assurance & Support is responsible for the mandatory training program and maintaining the Hanford Action Tracking System (HATS) system.

The AS Maintenance and Work Control is responsible for material procurement and control.

2.2.6 By Other Supporting Organization Outside the Analytical Services

The Analytical and Environmental Quality Assurance (AEQA) Organization provides QA support and services and has QA oversight responsibility to the WSCF.

2.3 Level of Authority

2.3.1 Laboratory Facilities Manager

The laboratory facilities manager has approval authority for procedures and documents prepared in accordance with WHC-CM-5-4. The laboratory manager has approval authority for combined data reports generated by the units reporting to him/her.

2.3.2 Scientist

The scientist has full decision-making authority on the operational readiness of the instrument systems assigned to him/her.

Authority is granted to other personnel, as necessary, to carry out their assigned responsibilities. Personnel have the authority to initiate appropriate actions:

- To prevent reporting results from a measurement system that is out of control, prevent further sample analysis and reporting until corrective action has been completed;
- To identify any laboratory method or procedure that poses quality problems;
- To provide solutions through designated channels, and monitor effectiveness; and

- To initiate a stop-work order where safety, serious quality or health conditions exist.

2.3.2.1 Interfaces

Communication systems used in the various laboratory groups are dependent on the particular sample and customer. A network Laboratory Information Management System(s) (LIMS) is used to communicate sample results to some of the customers. These formal reporting systems are described in the documents listed in Appendix A.

The use of telephone, Don't Say It --- Write It, internal memos, and external letters to communicate laboratory results are used as the conditions warrant. In all cases, original basic laboratory records are maintained to support these communications.

Appropriate meetings between laboratory and customer representatives are held. Formal presentations may be made and written minutes issued. Some meetings are only information exchanges and planning sessions of an informal nature.

References:

ASTM C-1009-83, "Standard Guide for Establishing a QA Program for Analytical Chemistry Laboratories within the Nuclear Industry," Section 5.

WHC-CM-1-2, Company Policies and Charters

WHC-CM-5-4, Laboratory Administration

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3.0 PERSONNEL TRAINING AND QUALIFICATION

3.1 Qualification

Managers are qualified based on their education, training, and experience. Proficiency of performance at that position will be evaluated at least annually through the performance appraisal system.

Qualification of technical staff is based on education and experience in laboratory operations. Scientists, by reason of their education, training, and experience may be qualified as analysts for all methods within their areas of responsibility.

Chemical technologists are qualified to perform analyses as they meet the specifications given in WHC-CM-5-4, Section 4.4. Training is tailored to the work assignment, education, and experience of the technologist as determined by his/her manager. Qualification is documented per WHC-CM-5-4, Section 4.4.

For those tasks that are "performing an analytical measurement," the technologist must demonstrate proficiency by analysis of a method control standard. The number of standards required and the time period for qualification may vary, but the normal criteria is to achieve analytical recovery on three consecutive standards 1) over a period not to exceed three days; and 2) obtain results which are statistically equivalent to the results achieved by those currently trained and performing the work. A score of 70% or better must be achieved on an appropriate performance evaluation sample. Provisions are made on the On-The-Job Training (OJT) Checklist for management to show its approval of each analyst's qualification.

For those tasks not in support of an analytical measurement, the technologist's performance is documented by the trainer based on observation of adherence to the criteria detailed on the OJT Checklist.

Documentation of requalification on modified/revised procedures will be done in accordance with Section 4.4 of WHC-CM-5-4.

3.2 Training Plans

The WSCF employees have an employee-specific training plan, determined by their immediate supervisor using the Training Matrix Program system . The training plan includes job specific required training courses, required OJT, and remedial training to correct performance deficiencies. The training plan also includes optional development within the organization activities including training from those courses offered by AS or WSCF, WHC and others, off-site seminars, symposia and short courses, or other work-related training or professional development activities as appropriate.

The WSCF internal training program is described in WHC-CM-5-4, Section 4.5, "Training Programs". Within this program are specific suites of required course work designed to maintain familiarity with company, facility and specific technical aspects of each job within WSCF. The program specifies required course work, applicability, and retraining requirements.

In addition to meet the formal training requirements in WHC-CM-5-4, Section 4, WSCF and/or AS also provides continuing improvement in the awareness and proficiency of all employees. Where practical, access to other WHC as well as off-site training and professional development opportunities is encouraged.

3.2.1 Training - General

Training of personnel within AS is accomplished through the following means:

Company-Wide Classes - These classes deal with security and safety topics and are under the control of the WHC groups assigned to monitor and maintain each program. WHC policies and WHC-CM-5-4, Section 4.0, specify which classes are required for laboratory personnel.

On-the-Job Training - This training in specific laboratory procedures is provided under the direction and observation of knowledgeable employees. Managers direct the effort, but actual training may be delegated to scientists who have documented qualification in the subject being taught. All OJT instructors must have attended the OJT Instructors Course. All training is accomplished and documented according to Section 4.4 in WHC-CM-5-4.

In general, the training is designed to identify what a trainee should be thinking and what the minimum information they should know when actually performing the procedure in the future. In addition to safety hazards and procedure limitations, reagents and supplies, and good procedural and housekeeping techniques, the following components need to be considered for all training:

Method

- method chemistry
- any QC built into the procedure
- record keeping requirements
- what is normal operation

Instrument

- location and use of maintenance and performance logbooks
- location of spare parts
- mechanism for reporting/addressing physical or operational problems
- facility utility tie-ins
- waste management and safety/health hazards

Data

- proper reporting of analytical results and data validation
- proper way to transfer operation to next shift

Development and Enrichment - There are a number of classes and learning opportunities available to management and technical staff to enhance their performance capability. Many of these courses are taught offsite by non-WHC staff including vendors, consultants, technical experts, and university personnel. Attendance at these classes is contingent on management concurrence.

Scientists - The qualification of a scientist is based upon the education, training, and experience of the individual selected for the position. Additional training requirements are determined by the individuals immediate manager.

Senior Chemical Technologists - Senior chemical technologists shall complete any training specific to the job assignment. Within 90 days candidates they must demonstrate proficiency in the job assignment to the satisfaction of their manager. They shall perform laboratory analyses and/or assignments of diverse and complex nature, requiring the full knowledge of analytical laboratory techniques and procedures, and the ability to operate the analytical laboratory equipment. Senior chemical technologists may direct activities of others and give OJT to less experienced personnel.

Chemical Technologists - Chemical technologists shall complete the initial chemical technologist training, facility specific training, Emergency Procedures/Abnormal Plant Conditions training, and job specific OJT. Initial training includes chemistry fundamentals, calculations, laboratory safety, and laboratory OJT. Facility specific training includes facility orientation, radiological protection/survey training, and hazardous materials/waste handling training. Emergency Procedures/Abnormal Plant Conditions training is a self study with an examination. Job specific OJT is performed in accordance with WHC-CM-5-4 Laboratories Administration Manual, Section 4.1, "On-the-Job Training Program and Qualification of Chemical Technologists". OJT on designated analytical procedures includes three consecutive/concurrent analyses meeting the required standards. Chemical technologists may direct others and give OJT to less experienced personnel.

Hanford General Employee Training and facility specific courses require retraining at various intervals. These intervals are documented in and can be accessed via the Training Information and Record system on soft reporting.

3.3 Continuing Training Requirements

WHC-CM-5-4, Section 4, contains retraining requirements for each course title referenced above. These retraining requirements are typically either annual or biennial. Retraining requirements are called out in the individual annual training plan, by routine requalification tickler from the training department, or by specific training identified through continuous improvement program. Continuing training programs are structured with specific position needs and designed to enhance personnel proficiency.

3.4 Records

Training records for the company and laboratory classes are maintained on a computer data base by the Computer Based Training Department.

OJT Checklists are kept in the employee's field file and go with the employee when he/she transfers. These sheets are kept as long as the employee's qualification is active. A computer based tracking system for employee qualifications is also maintained under the Analytical Laboratory Procedure/Analytical Laboratory Training Records Program Computer Programs.

Copies of employee's annual performance appraisals are maintained in the employee's field file.

References:

ASTM C-1009, Section 7

WHC-CM-5-4, Laboratory Administration

WHC-CM-1-1, Management Policies.

WHC-CM-8-1, Operations Support Services Manual.

WHC-WD-56110-004, 200 Area Analytical Laboratory Training Program for Technologists, Managers, and Scientists.

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4.0 QUALITY ASSURANCE OBJECTIVES

4.1 Data Quality Objectives

Quality assurance objectives provide a set of recognized parameters to monitor performance of an analytical measurement system and to qualify analytical data. Establishment of DQOs criteria can be achieved based on the following applicable factors:

- Regulatory requirements, e.g., Resource Conservation and Recovery Act (RCRA), Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), or Clean Water Act;
- HASQAP; and
- Usage of the data. Typical data uses include regulatory, process control, screening, planning, and development.

The policy of the WSCF is to have a written agreement on client project DQOs before analytical work is initiated. The WSCF either participates in or provides recommendations for establishing data quality criteria.

The agreement between the laboratory and client is established by several mechanisms depending on the size and nature of the project, e.g., through DQOs planning process, test plan, QAP_jP, or using the Request for Special Analysis form.

The QAP_jP is prepared jointly by WSCF and the customer, approved by WSCF management and the customer, and issued by the Engineering Document Control (EDC) organization. The QAP_jP can be changed or modified with review and approvals by the issuing and original reviewing organizations. Each year a review of the QAP_jP will be documented by the issuing organization.

QAP_jPs will be prepared for each project. Procedures for analyses, generation of data, data processing, and QA managements are required to follow the site-specific QAP_jPs. Each procedure has to be followed according to the officially accepted documents. Customer's specifications or project plans may supersede the laboratory QAPP.

In the agreement, the following information shall be provided when appropriate:

- Applicable regulatory requirements;
- Process knowledge, sample source, and sample conditions known to the client that could impact the laboratory worker's safety;
- Handling of radioactive samples in the transport process and in the laboratory;
- Estimated number and matrix of samples;
- Sample handling relative to specific sample or matrix;
- Analysis methods and analyte lists for sample analysis;

- Quality control sample: frequency, type, and acceptance criteria;
- Expected date of sample receipt, sample preservation, delivery methods, storage and container types and volumes, and holding times by method;
- Format and content of sample analysis reports;
- Turnaround time (from date of sample receipt to date of data delivery) in the laboratory;
- Name, address, telephone number of client, and laboratory contacts responsible for the project, and information to establish electronic data transfer; and
- Return of samples and disposition of waste.

4.1.1 A System for Notification of Unique Data Quality Requirements

A communication system in the laboratory for notification of unique data quality requirements after the laboratory - client agreement is set up according to the following:

- Unique data quality requirements are communicated to the appropriate personnel using the group meetings, e.g., staff meetings; and
- Written instructions/minutes shall be prepared and distributed to appropriate persons with lead responsibility.

4.1.2 Client Complaints and Resolution

Resolution for technical issues and complaints shall be coordinated at chemist or laboratory managers level. Corrective action shall be initiated in a reasonable time frame. For example, either stop processing samples until concern is resolved or revision to the original work requests could be used for non-conforming samples upon their submittal to the laboratory.

Issues, complaints, and resolutions from client shall be documented.

4.2 Client Data Quality Requirements

Five parameters are often used by the client to define project data quality requirements. These include precision, accuracy, completeness, comparability, and representativeness. Of these, the precision, accuracy, and representativeness have direct impacts on data quality (see Section 11.0 for limitations associated with precision and accuracy). The client is responsible for ensuring that adequate sample material is available and that appropriate sampling techniques are administered in order to meet their DQOs. The laboratory is responsible for using proper protective sample handling protocols. The laboratory and client share responsibility for selecting appropriate sample preparation and analysis.

The precision and accuracy requirements shall be agreed on by the laboratory and the client and should be based on the error tolerances of the sampling and analytical effort. The laboratory is responsible to provide precision and accuracy values obtained from the standards to the client. If

the client has special requirements for precision and accuracy they must be identified in the agreement. If the client has no special requirement, HASQAP or normal laboratory performance may be specified.

4.2.1 Precision

Precision is defined as an agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses and is expressed as the relative standard deviation (RSD). For very small data sets, relative percent difference (RPD) between duplicate measurements is accepted. Precision is calculated based on the equations listed in Section 12.

Precision of analytical methods is estimated using the laboratory control samples over time. The precision of analytical methods indicates the variability for the analytical method that can be expected on the relatively simple matrices.

Acceptance criteria for precision shall be established for each project and agreed upon by the laboratory and the client. Acceptance criteria for each analyte or analyte method shall be listed in the Project plan, characterization plan, or statement of work. The WSCF provides historical precision values to the client based on the measurements from the standards.

4.2.2 Accuracy

Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Accuracy is calculated based on the equations listed in Section 12.

Accuracy of actual sample is expressed as the percent recoveries of spiked samples. Spiking may not be applicable for analytes present in the samples in relatively high concentration (> 0.1%). In these cases other laboratory control samples can be used to estimate the accuracy of the method. Acceptance criteria for sample accuracy shall be established for each project and agreed upon by the laboratory and the client. Acceptance criteria per each analyte or analyte method shall be listed in the project plan, characterization plan, or statement of work. The WSCF provides historical values to the client based on the measurements from spiked samples or from the standards.

4.2.3 Comparability

Comparability is the confidence with which one data set can be compared to another. For each analyte, comparable precision and accuracy depend on the method and sample matrix. Factors such as analytical method selected, detection limits or uncertainty, precision, accuracy, and matrix effects should be taken into considerations when data is to be compared. A split sample or a known standard shall be used for comparability of different methods.

4.2.4 Completeness

Completeness is a measure of the amount of usable/valid data obtained from a measurement system compared to the amount of data that was expected to be obtained under correct normal conditions. Completeness can be used to evaluate the amount of data produced that meets the client's requirements (e.g., accuracy, precision).

4.2.5 Representativeness

Representativeness is defined as a degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, a process condition, or an environmental condition.

Representativeness of a population or an environmental condition depends on sampling and is outside the control of the laboratory.

Representativeness should be maintained by proper homogenization or appropriate subsampling (if different phases are apparently visible in the sample). Once subsampling occurs, identification of chemical/physical properties of subsamples, proper analytical protocol, and traceability of results to the original subsamples should be in place.

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5.0 SYSTEMS QUALITY ASSURANCE

5.1 Software Systems

Laboratory software systems can be separated by application into two categories: administrative and technical. Administrative software systems are used to manage the work flow or to monitor performance against administrative requirements. Examples of administrative software systems are those that control sample tracking, procedure control, training, and reporting. Technical software systems are those used to control laboratory systems, and accumulate and reduce data. Examples of technical software systems are those that provide instrument interface, calculations, calibration control, and control charts.

5.1.1 Control Requirements

Software control requirements applicable to both commercial and laboratory-developed software shall be developed, documented, and implemented. Software systems shall be protected.

Software systems shall be documented under configuration control. For laboratory-developed software systems, a copy of the original program code shall be maintained, and all changes shall include a description of the change, authorization for the change, and test data that validates the change. Configuration control and acceptance test data shall be maintained for commercial software packages.

The following documents govern laboratory software system configuration control:

- WHC-SD-WM-CM-002 (to be released): "Configuration Management Plan for LABCORE Program"; and
- LC-400-005: "Laboratory Computer Control," and WHC-CM-5-4, Section 8.3, for projects outside the scope of the LABCORE program as well as for any project that does not have a specific configuration management plan in place.

5.1.2 Acceptance Testing

Software systems shall be tested for acceptance when installed, after changes, and periodically during their use. The frequency of the test shall be based on the potential for adverse impact on the laboratory and the ease in which changes can be made to the computer code. Testing may consist of manually performing calculations, or checks against another software system that has been previously tested, comparison of output with previous output, or by analysis of standards.

Test of LABCORE is performed in accordance with WHC-SD-WM-CSWD-058, "LABCORE Software Test Plan".

The following procedures are used for laboratory-developed program/applications outside the scope of the LABCORE program as well as for any project that does not have a specific configuration management plan in place:

- LC-400-001: "FORTRAN Coding and Documentation Guidelines";
- LC-400-002: "Programmable Calculator Documentation and Coding Guidelines";
- LC-400-003: "Basic Coding and Documentation Guidelines";
- LC-400-006: "Spreadsheet Documentation Guidelines"; and
- LC-705-101: "ACE Program - Implementation and Operation of Spreadsheet and Computer Interface".

Documentation of the testing shall include print-outs of the data or results from data generated by the software for comparison, the name of the person performing the test, and the date the test was performed. The version and manufacturer of the software shall be documented.

5.1.3 Backups

Both software and data shall be backed up. The frequency of backup shall be based on the amount of data and the impact of the loss of data or software on the organization.

The following procedures may be followed for software backups:

- LC-718-001: "Laboratory ADP Operations" provides authority to use work sheets for routine operations (i.e., LABCORE or any project outside the scope of LABCORE). A specific work sheet "File System Backup Guide" is followed for LABCORE software and data backup. In addition, specific work sheets can be authorized to support laboratory instrument software and data backup if requested by the laboratory managers; and
- LC-808-101: "Laboratory Computer System Operation" provides authority to use work sheets for routine operations for the Data General Systems in MO-037. Two specific work sheets "MV10000 Full Backup" and "Daily Morning Status" are followed for software and data backup.

5.1.4 User's Manuals

LC-708-001, "MULTI LIMS Use in the Laboratory", provides user instructions on the use of MULTI LIMS. In addition, two training classes are provided: "LABCORE Overview On-the-Job" and "LABCORE Job Specific On-the-Job".

LC procedures are issued when appropriate for an application. For small or straight forward programs or applications that are outside the scope of the LABCORE system, a controlled manual or special training is not required (see WHC-CM-3-10, Software Practices, Section SP-3.4, "Small Job Development").

5.2 Administrative Systems

The WHC has established an administrative control system based on a hierarchy of Controlled Manuals (CM). These manuals provide documented interpretation of DOE orders and procedures for implementation. The WSCF works to the administrative policies and directions published in WHC Controlled Manuals (WHC-CM). In the WHC-CM system, a hierarchy in which Level I manuals provide direction from the President for implementation of key policy and administrative actions, Level II manuals are issued by company organizations for implementation of broadly applicable activities based on specific DOE orders. (i.e. Safety, QA, and Purchasing), and Level III manuals are provided for personnel at the working level, providing additional information not found in higher level documents. In addition to the administration directions listed in manuals, laboratory procedures are used for specific activities.

WHC-CM-5-4 Laboratories Administration, a Level III manual, provides the documentation describing and directing laboratory activities not sufficiently covered in Level II documents.

The following list identifies the documents which provide laboratory personnel with approved directions for various activities:

- Organization charts are published by the Director of AS;
- Procurement controls are in the Level II manual WHC-CM-2-1 Procurement Manual and Procedures;
- WHC-CM-5-4 Laboratories Administration;
- Sample and waste disposal instructions are found in the series of laboratory procedures numbered LO-100-nnn??; and
- Sample receiving and custodianship instructions are found in the series of laboratory procedures numbered LO-090-nnn??.

5.3 Physical Facilities Systems

The WSCF complex is located at the 600 Area. The WSCF includes the Utility Building, the Solid Waste Storage Facility, the Environmental Sample Archive Facility, the Contaminated Liquid Waste Retention Vault, the Mobile Laboratory Storage Facility, and the Environmental Data Remedial Tracking System Facility. The WSCF Building 6266 consists of administrative area, Nuclear Spectroscopy Laboratory (comprised of Counting Room and Air Sample Laboratory), and the Analytical Laboratory North Wing. The Counting Room, Air Sample Laboratory, instrument shop, electrical and machine rooms are located in the basement of Building 6266. Rooms including conference, storage, computer, lunch, locker/change rooms, office areas, and analytical laboratories are located on the main floor. The WSCF complex is designated to handle low-level radioactive samples.

5.3.1 Laboratory Utility Services

Benches and hoods in the laboratories are generally supplied with electrical outlets, sanitary and distilled water, piped gases, compressed air, and process vacuum. The electrical (120, 208, or 440, etc., volts) service within the facility is not regulated. Instruments susceptible to line power fluctuations are protected by stand-alone, in-line power conditioners or by conditioned power through red-orange colored-coded outlets. The facility has emergency power service for selected ventilation and lighting. Critical computer systems are protected by uninterruptable power supplies.

Laboratory work areas are maintained at negative pressure relative to atmospheric pressure using a single-pass ventilation system. The system is designed to release filtered air into the laboratory work areas at a nominal temperature of 72°F $\pm$ 3% and 50% $\pm$ 30% relative humidity. The Nuclear Spectroscopy Laboratory has an independent Heating, Ventilation, and Air Conditioning (HVAC) system without humidity control that has 95% recycled air through high efficiency particulate air (HEPA) filters; exhaust air is HEPA filtered. The remainder of the core Building 6266 has a separate HVAC system that maintains that area at positive pressure with respect to ambient.

5.3.2 Facility Inspection

The WSCF maintains essential facility services such as electrical and ventilation through the Job Control System as defined in WHC-CM-8-8. The schedules for this maintenance are defined by the cognizant engineer responsible for the system and is based on the essential equipment list.

5.3.3 Facility Inspection

The facility manager and/or other managers inspect laboratory facility conditions monthly and document deficiencies for corrective action on safety/housekeeping forms.

Quality Assessment personnel routinely assess on Conduct of Operations, Conduct of Maintenance, Training and Qualification, QA, Occupational Safety, Environmental Management, and Radiation Protection.

Reference:

ASTM C-1009, Section 11

Table 5.1 Physical Facility Systems in the WSCF

FACILITY	WSCF
Sample Receiving Area	Yes
Sample Storage Area	Yes
Standard preparation	Yes
Sample preparation Organic: VOA SMVOA	Yes
GC/MS: VOA, SMVOA GC	Yes
Sample Preparation / Extraction Inorganic: ICP, AA	Yes
Wet Chemistry	Yes
Preparation for Radiochemistry	Yes
Counting Room	Yes
Chemical Storage	Yes
Shipping Area	Yes
Waste Storage	Yes
Flammable Gas Storage	Yes
Non-Flammable Gas Storage	Yes
Lunch Room	Yes
Offices	Yes
Change room	Yes
Equipment	Yes
Equipment Storage	Yes
Quality Assurance Records Storage	Yes
Computer room	Yes

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6.0 SAMPLE CUSTODY AND HANDLING

6.1 Chain-of-Custody

The WSCF laboratory is restricted to authorized personnel only. Admission to the WSCF laboratory area requires special badging and personnel are checked before entering the Hanford sites. The entire WSCF laboratory is considered a secured area. During the day, exit doors are either monitored by the personnel or locked if personnel are not present. Exit/entrance doors are locked during the night.

Chain-of-Custody is maintained as required between the sample collection and the laboratory receiving Area by the client. Custody is transferred at the Laboratory receiving area to the laboratory internal custody. The laboratory receiving area refers to sample receiving custody. Internal custody refers to maintaining custody as the sample is dispersed to various groups within the laboratory for analysis. Internal sample custody is maintained until disposal of the sample from the laboratory.

The following Laboratory Operating (LO) procedures are for Sample Custody and Handling:

- LO-090-402: ABCASH sample receiving and custodianship - 222-S and WSCF Laboratories; and
- LO-090-403: Sample receiving and custodianship at the WSCF.

6.2 Holding Times

The majority of work supporting RCRA and CERCLA requires adherence to holding time requirements. These holding times begin when the sample is collected. Holding time is understood to be the time between sample collection and preparation and/or final analysis.

All holding time agreements should be either based on SW-846 specifications or Contract Laboratory Program (CLP) and shall be established between the laboratory and client before sample analysis. If the laboratory is unable to meet prescribed holding times, due to sample activity and such, the client must agree, in writing, as applicable, to this fact before work commences. The client is responsible for ensuring the timely delivery of samples to the laboratory to enable laboratories to meet holding time requirements.

The sample custodian is responsible for electronically notifying the lab personnel (responsible manager and chemists/analysts) upon receiving samples. After sample receiving, chemist/analysts are responsible to meet the holding times requirements.

6.3 Sample Receiving Procedure

The laboratory Sample Custodian shall perform the following actions according to the LO-090-402 and LO-090-403 procedures:

- Document the common carrier that delivers samples to the laboratory. A copy of the shipping document shall become part of the permanent laboratory record;
- Check the outer-most sample container(s) is not damaged;
- Check the outer-most sample seal(s) is intact;
- Verify that the Chain-of-Custody documentation is accurate, complete, and legible; and includes the following information:
  - project name or number;
  - client name and client sample number;
  - date and time of sampling for each sample, and sampling location;
  - container types, sizes, and number of containers;
  - sample preservation (when used);
  - analyses requested (or reference to);
  - signature for the person receiving and relinquishing;
  - date and time of relinquish and receipt; and
  - descriptions of any deficiencies identified by previous custodians.
- Check and record incoming cooler temperatures where volatiles, acid/based neutral, pesticide, or cyanide analysis are requested; note any deviations from 4°C +/-2°C. Certain conditions, such as sample containers (sample pig and cask), prevent a temperature check. Therefore, verification of the temperature shall be excluded under such condition;
- Verify that client sample numbers on the Chain-of-Custody match those on the sample containers;
- Verify collection date and date of laboratory receipt are within method- or project-specific holding time requirements;
- Notify laboratory staff as soon as possible when the sample holding time is less than 48 hours;
- Notify the client of samples receipt within 24 hours, by telephone, facsimile, or electronic mail. Notification documentation (i.e., copies of the telephone logs, facsimile, or electronic mail) shall be included in the report package, if requested; and

- Notify the client of nonconformance within 24 hours, by telephone, facsimile, or electronic mail. Nonconformance notification and client responses shall be documented by project coordinator and kept on file in the laboratory.

When sample receipt is completed, samples are then accepted for analysis. Upon acceptance of samples, the Sample Custodian shall sign the Chain-of-Custody and shall initiate internal Chain-of-Custody for analytical activities.

6.4 Sample Log-In And Tracking Procedure

Internal Chain-of-Custody is initiated by sample log-in and remains unbroken until sample disposal is completed. The Sample Custodian(s) is responsible for maintaining custody of the samples during the log-in and distribution processes. The Sample Custodian is also responsible for assuring that all records documenting that possession are properly completed and placed in the laboratory record system.

The following activities are part of the sample log-in and tracking procedures:

- The samples are secured in refrigerated storage or storage cabinets as appropriate after sample log-in. Any safety hazards communicated by the client are identified;

- LABCORE is used to assign sample numbers. Subsamples generated at the sample preparation stage. Each sample is given a unique identifier regardless of its re-sample status. Every sample, sample replicate, and subsample shall be labeled in a manner which allows traceability to the parent sample number;

- A cross-reference system is established to correlate client sample number and the laboratory sample number using the Chain-of-Custody form or the LABCORE;

- The LABCORE system is used for tracking sample status and holding times. Regulatory holding times and sample log-in times are recorded in the LABCORE system and can be traceable. In addition, sample retention times that are calculated according the regulatory holding times and date of sample receipt are also tracked in the LABCORE. Retention times and date of sample receipt in the LABCORE are used to track the status of any particular samples by the chemists/analysts. This system allows the laboratory managers to assess whether holding times will be met or exceeded; and

- Sample turnover times (from the time laboratory received samples to delivery of data report to client) are tracked in the same manner as the regulatory holding times.

6.5 Laboratory Internal Chain-of-Custody

Laboratory Operating Procedures, LO-090-402 and LO-090-403, address sample receiving and custodianship for the WSCF. The internal Chain-of-Custody remains unbroken using a sample log-in and log-out system. Once samples are in the laboratory, sample custody is controlled by the Sample Custodian. The location of all samples and the person in control of the samples is traceable from the time samples are received at the laboratory until the analysis is completed and the sample is disposed or returned to the client.

Traceability of samples within the laboratory is established using electronics, based on personnel bar codes, pass words, or other secure techniques.

6.6 Sample Disposal

The sample disposal includes disposing of or returning original samples to the client. Samples are disposed of through waste or material recovery systems. Samples are discarded as they appear on Slurp/Return Lists. The Hazardous Waste organization is responsible for disposing of samples that have relinquished from the laboratory. The laboratory has procedures in place to meet the following requirements:

- Disposing of or returning sample to the client;
- Maintaining records that identify the date of disposal;
- Meeting all local, state, and federal regulations;
- Documenting the status of sample in the Chain-of-Custody record, if samples are returned to client, custody records shall document the return; and
- Shipping documentation shall be maintained with the sample chain-of-custody by the sample custodian and shall meet Department of Transportation and applicable carrier requirements for transportation.

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7.0 CALIBRATION

This section describes calibration practices used by the WSCF. These practices include:

- Calibration of laboratory measurement systems;
- Traceability and documentation of standards used in calibration;
- Record keeping for calibration data; and
- Calibration of balances, thermometers, and pipettes.

The initial and continuing verification of laboratory measurement system calibration is described in Section 11.

7.1 Calibration

7.1.1 Instrument Selection

Table 7.1 lists the major analytical systems used to generate data. Supporting these systems are laboratory instruments such as analytical balances and chemical dispensers.

In their requests for analyses, WSCF customers may request specific WSCF procedures to be followed thereby the types of instruments used for each analysis are prescribed. The WSCF may recommend use of alternate instruments. The WSCF will only recommend alternate instruments acceptable under the governing regulation for the requested analysis.

7.1.2 Calibration of Laboratory Measurement Systems (LMS)

The requirements for calibration of each LMS are included in the Laboratory Procedure(s) that govern its operation. These requirements include, at a minimum:

- Number and range of concentrations or activities to be used in the calibration;
- Frequency of calibration;
- Criteria used to accept calibration; and
- Actions to be taken if calibration fails acceptance criteria.

Table 7.1 Major Analytical Systems	
INSTRUMENT/APPLICATION/TECHNOLOGY	ACRONYM
Atomic Absorption Spectroscopy	AA
Graphite Furnace Atomic Absorption Spectroscopy	GFAA
Inductively Coupled Argon Plasma Spectrophotometer	ICP
Ion Chromatography	IC
Conductivity Meter	...
Spectrophotometer (UV - Visible)	...
Gas Chromatography	GC
Gas Chromatography/Mass Spectrometry	GC/MS
Alpha Energy Analysis	AEA
Gamma Energy Analysis	GEA
Alpha/Beta Counter	A/B
Uranium Fluorimeter	UF
Liquid Scintillation Counter	LSC

Initial instrument calibrations shall be performed according to manufacturer's specifications. General instructions for use in establishing instrument calibration are given in the various Laboratory Analytical (LA) procedures. Specific instructions for use in calibrating a particular piece of equipment are documented. Because each piece of equipment is a component of an analytical measurement system, its performance of each measurement instrument is continually monitored by the use of control charts.

Calibration requirements in Laboratory Procedures used for regulatory-driven analyses must conform with calibration requirements specified in the appropriate regulatory methods. Any variances from the requirements included in LA procedures must be based on an agreement between the laboratory and the client (e.g., via letter of instruction, statement of work, etc.).

Accuracy of initial calibration shall be based on traceable reference standards[1]. If traceable reference standards are not available, appropriate physical and chemical means are used to determine quality. A level of independence shall exist between the materials used for LMS calibration and those used for initial calibration verification (ICV) (described in Section 11).

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[1] NIST, Amersham, or other certified standards are to be used whenever possible

7.2 Specifications of Standards Used in Calibration

Laboratory Reference Material Specifications (LR) indicate the purity of starting materials, concentrations, and tolerances of all components in the standards. Special limitations of storage, use, and age are also detailed. LA procedures give the same information for reagents.

The Standards Laboratory is responsible for the procurement and preparation of materials used for LMS calibration. When appropriate, these materials shall be traceable to a nationally or internationally recognized standard agency source (e.g., the National Institute of Standards and Technology (NIST) [formerly the National Bureau of Standards]), or measurement system. Alternatively, the Standard Laboratory will procure materials of known quality and will document the materials as described below.

The Standards Laboratory maintains records of procured reference materials which include, at a minimum:

- Source vendor;
- Lot number;
- Purity;
- Date of preparation and/or expiration; and
- Certified concentration or activity of the standard material (including uncertainty if available).

In addition, for calibration standards prepared by the Standards Laboratory, the following information, at a minimum, is recorded:

- Name of the preparer;
- Date prepared;
- Unique identification of the standard;
- Dilution or other preparation performed (e.g., digestion or mounting);
- Final concentration or activity; and
- Expiration date or shelf life (standards with indefinite shelf life are so designated).

When these records are maintained, the final standard shall be considered traceable to the original standard reference material.

This traceability data is available from the Standards Laboratory upon request. Where beneficial, the Standards Laboratory can present this data as a certificate documenting the source of the standard reference material and its subsequent preparation for use as a calibration standard. Some standard materials that have long shelf life may be re-certified using the LO-120-101 procedure.

Calibration standards that have exceeded their expiration date or shelf life shall no longer be used for LMS calibration or clearly marked as unusable for calibration purposes unless re-certified as appropriate.

Some standards, such as radioactive materials, are verified by preparing mounts. The mounts are counted by the counting room and compared against the calculated certified value. Some standards such as these for Inductive Coupled Plasma (ICP) are submitted to the analytical laboratory based on the traceable certified value, material is then checked against on independent standard by the analytical laboratory for verification. The data is documented in the data management system. Organic compounds used for calibration standards are purchased by the Standard Laboratory, but are prepared by the chemists before calibration.

7.3 Calibration Records

The WSCF maintains calibration records for all methods requiring LMS calibration. These records include raw data (i.e., instrument response values necessary to reconstruct the calibration; e.g., peak areas, counts, absorbance values, or emission intensity), corresponding concentration or activity data, the calculated calibration factors (e.g., regression results), the criteria used to accept or reject the calibration (e.g., correlation coefficient), the effective date of the calibration, and the analyst's name or initials.

Calibration records are maintained in logbooks or notebooks as appropriate. When completed, these books are maintained in accordance with Section 10.0.

Results for sample analyses performed at the WSCF shall include an analysis date. This date and the recorded effective calibration date permits traceability of the analysis to the most recent preceding LMS calibration. If ambiguity is possible due to calibration performed after sample analyses on the same date, either time-of-day information must also be recorded (for both calibration and analyses), or the date of the applicable calibration must accompany analysis results.

7.4 Balances, Thermometers, and Pipettes

7.4.1 Balances

Calibration of analytical balances are checked on a quarterly basis using standard weights. The standard weights are verified against the NIST standards once per year by the WHC Physical and Electrical Standards personnel. Balances not passing these checks are removed from service. Stickers are placed on each balance indicating the date of calibration, the expiration date of its calibration, and the initials of the person performing the calibration.

The calibration of balances are verified by the analyst at a minimum, before use or on a daily basis, whichever is less frequent, by measurement of an internal or external check weight (using LO-140-005 procedure). The results of the check are logged in a notebook maintained in the same room as the balance. This notebook also contains acceptance criteria for each balance logged. If the check fails, the balance is taken out of service until it is

recalibrated. The data from the checks are evaluated by the scientist for performance acceptance.

Balances that do not satisfy WSCF calibration requirements are so labelled and are not used for quality-affecting determinations.

7.4.2 Thermometers

Thermometers and thermocouples used for sample storage refrigerators are checked against a nationally recognized standard (e.g., NIST certified thermometers) annually, at a minimum.

If temperature measurements affect the quality of data obtained from specific laboratory measurement systems, the governing Laboratory Procedure shall include the steps required to ensure sufficient temperature accuracy and precision.

7.4.3 Pipettes

Instructions for calibration and use of volumetric pipettes and burets can be found in the Procedure LO-140-410. The tolerances allowed by the calibration are listed in each procedure. At a minimum, each pipet is calibrated annually, checked daily or before use (whichever is less frequent), and documented in the laboratory notebook. When found out of tolerance, the pipet is discarded.

Other WHC documents that include calibration guidelines or policies:

WHC-CM-4-2 Quality Assurance Manual
QR 12.0 Control of Instruments
QI 12.2 Operator Calibrated Measuring and Test Equipment
QI 12.3 Calibration of Plant-Installed Instrumentation
QI 12.4 Calibration Control of Measuring and Test Equipment
QI 12.5 Statistically Controlled Analytical Instruments
QI 12.6 Determinately Controlled Laboratory Instruments
QI 12.7 Assuring Availability of Laboratory Instruments

WHC-CM-5-4 Laboratories Administration
8.2 Laboratory Instrument Calibration Control Program

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8.0 LABORATORY PROCEDURES

Laboratory activities are directed and controlled by approved procedures. Each has a unique identification code based on an approved numbering system.

Procedure types are grouped by general task categories, e.g., LA represents analytical procedure, LC for computer, LO for operating procedure, LQ for quality control, LT for lab technology, LR for laboratory reference material, etc.

8.1 Procedures and Supporting Documents

A standard format is used for each procedure as guided by the Writer's Guided for Technical procedures, DOE-STQ-1029-92 and in WHC-CM-5-4, Section 3.9.

Procedures for each laboratory analysis and/or activity are prepared by chemists or other technically qualified personnel.

The procedure is reviewed by technical organizations or individuals to check technical correctness. Reviewers document their comments using a Procedure Review and Approval Form (PRAF). The procedures shall be approved before use in accordance with WHC-CM-3-5 manual, Sections 12.7 and 4.0.

The approved procedures is issued as a performance "goldenrod" copy with an identification code (document number, revision/modification number), release date, official release stamp, author, author's manager, and the title. Procedure distribution is administered such that controlled copies are maintained up-to-date by the Procedure Administration from the Documentation Administration organization. White-copies of procedure are uncontrolled copies of the goldenrod procedure and used for reference only.

During development of new procedures, proposed procedure can issue in the User Test (Blue User) form to test the feasibility of the new procedure under the direction of the author.

Active procedures have no fixed expiration date, but are required to be reviewed every 24 months for accuracy and adequacy by technically qualified personnel. The review is documented on the PRAF and maintained by the Procedure Administration.

A procedure can be inactivated or removed by the responsible scientist or engineer by using a PRAF or other signed notification with management approval.

8.1.1 Preparation and Review of Supporting Document

Supporting documents (SDs) are used to document QAPPs, QAPjPs, basic laboratory practices, technical project plans, and laboratory test plans. Laboratory SDs do not have a specific format. These documents provide a combination of administrative guidance, technical direction, and quality requirements. They are reviewed internally and externally based on the topic and application. The Supporting Document Release Station assigns identification numbers for all SDs.

The responsible manager (approval authority) for each SD identifies the reviewers within WHC organizations. The approval of the SD is documented on the Engineering Data Transmittal form (BD-7400-127) by the reviewers. An SD can be canceled and recalled by use of an internal letter.

8.2 Change Control

8.2.1 Supporting Document Changes

An SD can be revised by the author using an Engineering Change Notice (ECN). There are two types of ECN changes: direct or supplemental changes. For direct changes, the ECN summarizes the change description and is the authorization for a new revision of the document to be issued. For supplemental changes, the ECN delineates the change details and becomes a part of the current document. Supplemental ECNs are incorporated into the document in the next revision. Both methods are detailed in Procedure EP 2.2 in WHC-CM-6-1. Approval signatures are obtained from the author and the author's manager. Engineering Document Control "release-stamps" the ECN/document, and Information Resource Management Document Control distributes complete document revisions or document with supplemental ECN incorporated to the same (or officially revised) recipients as the original document.

8.2.2 Procedural Changes

The laboratory makes changes to procedures (both regulatory and internally developed procedures) for a variety of reasons. The Procedure Change Authorization, (Form A-6400-242), or PRAF is used for documenting and issuing procedural changes described in the following. Three categories of changes are used based on the HASQAP concept.

8.2.3 Definition of Procedural Changes

Substitution is an adjustment in a procedure that would have no significant effect on final results. This would be clearly evident in the QC data associated with the final results.

Deviation is divergence from the original procedure that does not adversely impact the analyst's ability to meet the precision, accuracy, detection limit, selectivity, and QC criteria of the procedure. Therefore, the decision to deviate shall be based on published literature (e.g., alternate methods) and/or known sample chemistry. For documentation requirements, see Section 8.2.4.

Modification changes the character of a method, and thereby, potentially limits a method's ability to meet the originally stated precision, accuracy, detection limit, selectivity, and QC criteria. Because the impact of such a modification cannot be ascertained before implementation, it must be demonstrated by application. For documentation requirements, see Section 8.2.4.

8.2.4 Control of Procedural Change

Because substitution does not impact the method performed, no documentation of change is required (see Section 8.2.3 under substitute). Only the documentation necessary to allow reproducibility of results is required.

Deviation requires documenting the changes made to a procedure. Documentation of deviations made shall be included in the final report narrative. Justification of the deviation should be evident in the acceptable performance associated with the final results and should also be discussed. Acceptable performance shall be demonstrated by the analyst's ability to meet or exceed the original method's precision, accuracy, detection limit, selectivity, and QC criteria. Whenever possible, the client should be notified of deviations before starting work. When a deviation is used routinely, it shall be incorporated into the procedure.

Modification requires the procedure to be qualified (see Section 8.5), documented, approved by laboratory management, and agreed on with the client before work. Requirements for implementation and personnel training shall apply as necessary to all laboratory procedures. Justification of the modification should be evident in the QC data associated with the final results and should also be discussed. A modification with long-term applicability should be developed into a new laboratory procedure that is issued with a new title and code.

8.3 New Analytical Methods

U.S. EPA, DOE, and consensus methods [e.g., American Society for Testing Materials (ASTM), standard methods] are recommended where the technique is applicable to the sample matrix and the overall objective of the analysis. Analytical methods used by WSCF are listed in Appendix C.

New analytical method procedures shall be qualified before use (see Section 8.5). New methods are defined as methods used for the first time whether based on published, well-understood procedures or developed in the laboratory. The following protocol is followed as applicable for developing a new analytical methods. The first stage is to conduct and document the performance using simple standard materials and to establish the following parameters as appropriate:

A. Accuracy/precision;
B. Detection limits;
C. Individual interference studies;
D. Parameter variable studies;
E. Linearly ranges;
F. Effect of interferences (chemical);
G. Effect of reagent concentrations;
H. Effect of instrument parameters; and
I. Kinetic effects.

The performance shall be verified using the following parameters as appropriate:

A. Complex standards, if available or prepared simulated;
B. Matrix standards, if reasonable;
C. Through the use of spikes or Method Standard Addition on actual samples;
D. By using an independent analytical method;
E. Through sample exchange programs with other labs; and
F. By comparison with "standard" or "accepted" methods on actual samples.

8.4 Modification of Required Regulatory Methods

The following procedures shall be used when modifications to required regulatory methods are made. These procedures shall be followed only when the precision, accuracy, detection limits, and/or QC criteria of approved methods might be impacted (positively or negatively). Method qualification requirements are in Section 8.5.

8.4.1 Justifying Modification

The citation of the original, required regulatory method shall be provided. All modifications to the required regulatory method shall be specifically described by providing a synopsis (or direct quotation) of the regulatory method requirement and a description of all changes made. The reason(s) why the requirement cannot be met and/or the technical, health and safety, environmental, and/or waste disposal merits of the modification(s) shall be provided.

8.4.2 Documenting the Modified Method

In cases where changes are restricted to specific sections of the required regulatory method, the text of the modification shall be provided (e.g., different instrument configuration, different spike or surrogate compounds). A complete copy of the modified method shall be provided when extensive modifications are necessary. The modified method shall be managed as a controlled document, subject to the necessary review and approval.

The impact of the changes on the published precision, accuracy, and/or detection limit of the modified method shall be established by experiment. Any modification to the approved QC procedures for the method shall be described and the acceptance criteria specified (e.g., using special surrogates and/or spikes, detection limit). See Section 8.5 for the approach required for method qualification.

Implementing the final modified method as a production method in the laboratory requires signatures of approval that all requirements have been met. Approval signatures are required from the laboratory QA representative and a representative of laboratory management from the section where the method is to be performed. Modifications to the regulatory required methods also require program and appropriate regulatory agency approval of its use.

All original laboratory test data shall be retained on file to enable retrospective examination of the method should the need arise.

8.4.3 Reporting Results from Modified Regulatory Methods

All modified methods are required to be issued with its unique identification code to notify the data user that the method has been modified. To the extent practical, modified methods shall retain a method reference (identifier) to the original method.

8.4.4 Acceptance Criteria for Modified Methods

Modified methods shall include the acceptance and performance criteria for precision, accuracy, calibration, and detection limit established during the qualification experiments.

8.5 Qualification of Analytical Methods

Qualification is the process of determining the suitability of a measurement system (preparative or analytical) for providing useful analytical data. Performance parameters of the method are compared with the requirements for the analytical data. Several approaches may be used to qualify a method and include the following:

- When suitable reference materials are available to adequately test method performance versus matrix effect, performance is demonstrated quite easily. This test consists of analyzing a sufficient number of reference samples and comparing the results obtained to that quoted for the particular material. A simulated matrix may be the closest performance indicator available; and

- When suitable reference materials are not available, two other approaches are considered reasonable. The first is comparing the new method against a known, well-established (laboratory approved or regulator recognized) method; the second is inter-laboratory comparisons. In limited cases, matrix spikes and/or surrogates may be used; this is the least desirable because of limitations associated with preparing spike and/or surrogate materials. Also, spikes and/or surrogates may behave differently than the actual sample in the process investigated.

Generally accepted standards dictate using a minimum of four replicates for <u>each test case</u>. Whenever possible, seven replicates should be used. This data should then be used to establish statistical control on an advisory basis until sufficient data are acquired, typically considered to be 30 data sets.

A method must also be evaluated for its overall effectiveness in the areas of sensitivity, linear range limitations, matrix or analytical precision, accuracy, and counting statistics (radiochemistry), as applicable to the method and/or analyte. This requires that method testing include:

- method detection level determination and/or minimum detectable; activity (according to Section 13.0);
- method blank evaluation;
- precision and accuracy determination;
- counter performance;
- uncertainty; and

- determination of method interferences as appropriate to the method (i.e., preparative versus determinative).

These studies shall be documented in the procedure or in the SD document that is referenced in the applicable procedure.

References:

ASTM C-1009, Section 8

WHC-CM-5-4, Sections 3.9 and 3.10

WHC-CM-6-1, Standard Engineering Practices.
a. Procedure EP-1.1
b. Procedure EP-1.6
c. Procedure EP-1.7
d. Procedure EP-1.12
e. Procedure EP-2.2

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9.0 DATA COLLECTION, REDUCTION, AND REPORTING

9.1 Data Collection

WSCF uses a LIMS (e.g., LABCORE) for documenting laboratory work (e.g., generating analytical work sheets to initiate the analysis process), tracking samples, and reporting results. Entries on paper records are made in ink. Incorrect entries are initialed, dated, and a line drawn through the incorrect entry in such a way that the entry is still readable. A permanent record of all analysts' names, initials, and signatures must be maintained. All analytical (measurement) data entries are dated and marked with the initials of the analyst doing the work. Data entries in the computer systems are identifiable as to whom made the entries and the dates of entries. The computer systems have provisions for making corrections. Provisions are made in the LIMS for fulfilling the functions of a laboratory record system.

Sample schedules are retained until superseded, or for a year after the project is complete. Sample analysis request forms are kept for a period of one year.

9.2 Data Recording System

9.2.1 Log Book

Various types of log books are used throughout the laboratories. Logbooks shall be paginated before use. Logbooks shall have the dates of use clearly documented on the front of the log. Sample receiving and shipping log books are kept at the WSCF to document the receipt and shipment of samples.

Shift log books are used to transmit administrative information from one shift to another. Such information usually details sample information, analytical measurement system performance and corrective actions required, problems with measurement instruments or systems, special customer instructions, notices of issuance of a procedure change authorization, or a revised issue of a procedure and/or safety items.

Log books have a minimum retention time of five years.

9.2.2 LIMS

When samples are logged into LIMS, a file is created in the computer data base to track the progress of the sample. Each sample is assigned a unique sample number. The date sampled and analyses requested are part of this record. As analyses are completed, the results are entered into the data base. On-line reports indicating the progress of the work are available. When the work requested is complete, the sample is identified on a list for disposal after a specific holding time which varies for different laboratory units. The LABCORE system is in the early stage of implementation, functions such as sample number assignment and tracking are in place. Until the LABCORE is fully implemented, other means of reporting e.g. instrument data printout may be used.

The LIMS work sheet identifies the sample number, the material type, analytical method to be used, normal sample or dilution size, the attribute to be measured, and a number of management evaluators (cost code, estimated turn around time, etc.).

9.3 Data Generation

Raw data are generated by manual or electronic means in the WSCF. Most analytical systems in the WSCF are computer controlled and have capability to generate and report both hard copy and electronic data.

Manual collection is conducted by the analyst and recorded in log books. Calculation, if needed, is performed by the instrument controller, associated software, or manually by the analyst, and recorded in log books. Procedures for correcting data entry errors include line drawn through the data, initialing, and dating the change. Final data in log books are then entered into the LIMS. Each individual generating data or information is responsible for identification of data entry errors, sample identification errors, and calculation errors. Peer review of calculations are to be co-signed by the reviewer. Managers are to annually review the logbooks and document the review by entering the pages reviewed, the date, and signing the entry.

Some instruments print the data only on hard copy (no electronic transfer of data). These loose papers may be inserted into the logbook as a permanent record. The means to a fix is to utilize glue or tape. The insert shall be signed across the edge such that the signature is on both the insert and page. Thermal paper may NOT be inserted as a permanent record; a copy of the thermal paper shall be used in its place.

The computer system provides a near-real-time record of the work performed on a sample. As analyses are completed in the laboratory, the results of the analyses are entered into the computer.

The Standards Laboratory maintains documentation for the makeup of all standards produced. The documents detailing Standards' makeup have an unlimited retention time.

9.4 Data Reduction

Data reduction is performed electronically. The automated reduction program reduction factors are included in the reports. All commercial software automated data reduction routines will have vendor-verification. WSCF- or WHC-developed software will be verified in accordance with Section 8.3 of WHC-CM-5-4.

Data reduction procedures on significant numbers, rules for rounding, and reporting rules will be conducted by a procedure to be written based on LO-150-127. All justified digits are carried through all calculations with only the final answer being rounded off to the proper number of significant digits. The justified digits are determined by the responsible scientist.

Raw or processed data are transferred either manually or electronically during record generating processes. Sample identification during sample collection and preparation, and certain raw data records are transferred manually and are checked by the analyst, scientist, and manager.

Final reports are generated electronically by both the instrument controller and/or LIMS.

9.4 Data Reporting

Measured parameters, the details of analysis, and the data values are reported in accordance with the requirements of the end-user as specified in the agreement between the laboratory and the client. The type of information, level of approval, data reporting format, and means of delivery shall be discussed and agreed upon between the laboratory and the client (see 9.4.2 for information required in the reporting documentation).

Radiochemical results shall be reported based on calculated concentration or activity values (whether negative, positive, or zero) using the appropriate blank for each nuclide. The measured activity or concentration should be reported with estimates of the associated counting uncertainty and total propagated uncertainty but without comparison to the estimated *a priori* minimum detectable concentration (MDC). The MDC should not be reported to the client *in lieu* of low-level measurements.

9.4.1 Manual

Analytical results are transmitted to laboratory customers in one of the following ways: 1) The computer prints the results out at the customers computer terminal shortly after laboratory personnel enter the results into the LIMS; 2) When all analyses have been completed on a sample, the computer is used to generate a report containing all the requested analytical results; 3) Report forms developed for specific customers are utilized. These reports have various formats; however, all hard copy reports are signed by laboratory management, and computer reports are approved by pass word-protected authorizations. Some reports are sent with a letter of transmittal; and 4) Memos/letters containing the analytical results are issued and sent to the customer.

The final report is released to the customer after approval by the responsible WSCF Manager.

Retention time for the memos/letters is usually one year; however, results on "record" environmental samples are retained indefinitely.

9.4.2 Data Reporting Documentation

The format of data reporting (e.g. electronic or hard copy) and the level of detailed reporting information shall be agreed between the laboratory and clients. The reporting documentation should include the following information:

- Laboratory name and address;
- Sample information including unique laboratory identifier cross-referenced to client identification, sample collection date and time, date of sample receipt, and date(s) of sample preparation and analysis;

- Analytical units and results, reported with an appropriate number of significant figures;
- Report uncertainty for radiochemical analysis;
- Detection limits;
- Method reference;
- Appropriate QC results (correlation with sample batch shall be traceable and documented);
- Appropriate data qualifiers with definitions and a narrative on the quality of the results, if applicable; and
- Additional data reporting (i.e., the percent of moisture/solid or correction for equivalent dry weight may be included if requested by the client).

9.4.2 Preliminary Reporting

A preliminary data reporting system shall be established between the client and the laboratory to address an emergency situation. The type of information, level of approval, data reporting format, and means of delivery shall be discussed and agreed upon between the laboratory and the client. The emergency situation may include, but is not limited to screening activities for safety issues, critical analytes, or limiting sample amount.

Preliminary report does not go through the routine data review and verification cycle. The immediate supervisor and chemist are responsible to review the data before reporting to the client. The preliminary reporting is delivered through cc:Mail.

Reference:

ASTM C-1009, Section 9

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10.0 RECORDS

10.1 Laboratory Records

10.1.1 Identification

Laboratory records fall into three categories: 1) QA records which must meet the requirements specified in this section; 2) work sheets, which are not QA records, which are used for temporary record storage or transmittal, or for information purposes; and 3) WHC forms which have records management requirements specified in company manuals. Instructions for completion of the WHC forms will not be duplicated here.

Records generated from laboratory activities are identified in different sections of this document, in WHC-CM-5-4, and in various laboratory documents and procedures.

10.1.2 Distribution

Many of the laboratory records are maintained within the laboratory and have no distribution. For those records which have a distribution, either original records or copies are maintained in the laboratory where they are generated. Distribution of records is documented in the Customer Sample Schedules or on the SAR form.

Laboratory management is responsible for the control and distribution of records. Access to data is limited to laboratory personnel and appropriate clients.

10.1.3 Storage

Record storage is the responsibility of the laboratory section managers. Corrective Action Reports (CARs) are maintained by the QA officer. Records of laboratory training are maintained and distributed by the laboratory section managers. The manager of the Standards Laboratory is responsible for standards' makeup documents. The laboratory sections managers are responsible for storage of data tapes.

Records are stored in file cabinets and on shelves in the managers' or scientists' offices and in the laboratory. Some records are maintained at the specific work site. Special tape storage cabinets are used for the magnetic data tapes. Standards' makeup documentation is kept in file cabinets in the applicable standards laboratory.

Laboratory record storage locations are chosen to avoid proximity to water, chemicals, and fumes to minimize damage potential. Fire protection is per building codes. The records in each area are afforded security protection suitable to the classification of the data. Access is limited to laboratory personnel and need to know.

10.1.4 Retrieval

Data and records are stored by customer, laboratory sample number, and/or date. Training records are filed in special folders for each person. Data on computer tapes must be accessed through the chemist assigned to the laboratory computer or by a knowledgeable computer operator.

10.2 Generation of Quality Records

The quality records include, but are not limited to the following:

- Procurement documents;
- Training records;
- Calibration records;
- Maintenance records; and
- Chain-of-Custody forms.

Information Resource Management (IRM) is responsible for the Quality records system based on WHC-CM-3-5, Sections 5 and 9. Specification, preparation, review, approval, and maintenance of quality records are governed by the WHC-CM-3-5, Sections 5 and 9. A computer system is used to maintain and access quality records including electronic media to ensure the records are useable and retrievable.

Documents and data referenced by final reports shall be retrievable from the records system, except for readily available references (e.g., national codes and standards).

10.3 Receipt Control

Program Management and Integration organization is responsible for maintenance of the Laboratory Technical Information Center and for coordinating retrieval of record copy that is maintained in the laboratory. The Information Resource Management (IRM) is responsible for receiving the records and implementing a system of receipt control of records for permanent and temporary storage. Information Resource Management has the capability to do the following:

- Designate the required records;
- Identify records received;
- Receive and inspect incoming records; and
- Submit completed records to the records holding facility without unnecessary delay.

10.4 Records Validation

Records to be considered valid shall be signed, initialed, stamped, or otherwise authenticated and dated by the document's originator (WHC-CM-3-5, Section 9, page 4 of 14). Electronic records shall be validated by signing and dating a paper copy of the first page of the record. These records may be originals or copies. Handwritten signatures are not required if the record is clearly traceable to the person or organization who created the record.

10.5 Correcting Records

Correction to quality records system is described in WHC-CM-3-5, Section 9, paragraph 5.4. The correction of technical or quality related information shall have an appropriate review and approval by an authorized person of the originating organization that was responsible for the approval of the in-process document. The name of the person authorized to issue the correction and the date of correction shall be marked on the quality record.

10.6 Records Identification and Indexing

Records are cataloged and tracked on a database and can be retrieved through a variety of topics including project or activity identification. Record retention times are controlled by the Records Inventory and Disposition Schedule (RIDS) system. Location of the record is indexed on the Record Holding Area - Management Information System (RHA-MIS) .

10.7 Maintenance and Retention of Records

Certain laboratory records, such as those for environmental or accountability analyses, are to be maintained indefinitely. The retention times for those laboratory records which can be disposed of are found in each groups "Records Inventory and Disposition Schedule", which is maintained by management.

WHC-CM-3-5, Sections 5 and 9, describe maintenance of active records for transmittal, distribution, retention, protection, preservation, traceability, accountability, disposition, and retrievability.

WHC-CM-3-5, Section 9, describes the criteria for lifetime record and nonpermanent records. Appropriate quality record retention time is monitored by the RIDS system. Expired records are destroyed after reaching the scheduled retention time.

10.8 Access Control

Access to quality records system is limited only to the authorized personnel (WHC-CM-3-5, Section 5). Access to the electronic media quality records only is granted to hard copies of this information.

10.9 Replacement, Restoration, or Substitution of Records

Lost or damaged records shall be replaced, restored, or substituted when applicable (WHC-CM-3-5, Section 9).

10.10 Records Turnover to Client

Records turnover to the client are copies of the original records. Original records are kept in the WHC and controlled according to the appropriate record requirements defined in WHC-CM-3-5.

References:

ASTM C-1009, Section 10

WHC-CM-3-5, Sections 3.16, 3.16A, 5, 7.2, and 9.

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11.0 QUALITY CONTROL

This section describes the QC measures used for analysis performed at the WSCF. The following areas of QC are addressed:

- Laboratory QC
- Preparation QC
- Analytical QC

The degree of QC required for analysis is determined through negotiations with the client. It is based on the "end-use" of the data. It may not violate, however, the specific QC protocols of EPA analytical methods.

11.1 Laboratory Quality Control

The QC described in this section represents the basic environment surrounding the analytical operation.

11.1.1 Deionized Water

Deionized water is prepared via a Reverse Osmosis (RO) system followed by mixed resin bed deionization located in the mechanical equipment room of the North Wing of the laboratory. It is then supplied to several laboratory locations where the water is further purified using smaller commercial deionized water systems.

The quality of water is measured by:

- A resistivity check of the commercial purified water system (e.g. Q-water system) prior to delivery. The reading must be >1 Mohm.cm or the water is considered suspect and not used; and
- Each system is sampled and tested for pH, metals, anions, and conductivity when appropriate, and monitored by preparation blank. This data is summarized and kept in a binder near each commercial purified water system (e.g., Q-water) station. Criteria and corrective actions are recommended by the cognizant scientist after reviewing the data.

11.1.2 Reagents

Reagent Water Types I and II as defined in ASTM D 1193-77 shall be used for the preparation of reagents and standards. Type I grade of reagent water shall be used where maximum accuracy and precision is indicated, provided dissolved organic matter will not interfere. Type II grade of reagent water shall be used for all other analytical procedures and for procedures requiring water low in organics. Type I water may be used for organic analysis, if a filter for removing organics is a part of the water treatment system. Requirements for the quality of water are identified in the analytical procedure where they differ from those stated above. Water used for all dilutions and chemical analyses follow the same guidelines. Document SD-CP-LB-028, Laboratory Reagents, gives detailed information on the grades of chemicals.

11.1.3 Compressed Gases/Reagents

Percent purity levels necessary for quality analysis are listed in each analytical procedure. The quality of gases or reagents is monitored by the performance of preparation blank.

11.1.4 Labware

When performing an analysis, or generating a reagent or standard, glassware is chosen such that the tolerance specified in the procedure is met. Volumetric glassware and pipettes purchased from vendors are usually Class A and have the standard tolerances of this class. In some cases, a piece of glassware is purchased which has a Certificate of Accuracy (or Certificate of Calibration). Where tighter tolerances than Class A are needed for a piece of glassware, the item is calibrated. Labware will be cleaned according to operating procedure LO-080-116.

11.1.5 Housekeeping

Each employee is responsible to maintain their work area in a neat and orderly manner. Inspection of housekeeping including inside fume hoods is performed periodically. Correction actions are documented.

11.1.6 Control Charts

Control Charts provide a useful tool in assessing trends in system performance. Monitoring these trends by the technical staff will help them:

- Ensure their analytical system is in control;
- Recognize problems and address them before the system goes out of control;
- Document conditions or corrective actions that "fix" particular performance problems (for future reference); and
- Demonstrate continuous quality improvement as they refine their system and their practices.

At a minimum, control charts are maintained for a parameter or parameters that monitor both the preparative process (if any) and the analytical measurement. Normally this requirement can be fulfilled by charting the Laboratory Control Sample (LCS) recovery percent.

Control limits will be set either at 3 sigma or based on environmental regulatory requirements. LABCORE control limits are calculated by the statistical department using classical standard deviation approved by cognizant chemist. The LABCORE database has the option of using either standard deviation or average and moving range. Formulas used for calculation of the control chart are provided by the statistical department for the database systems. See LQ-150-001, Section 7.0, for control chart requirements and tracking systems.

Any data point outside the upper or lower control limit or fails other test rules of trends and patterns shall require chemist investigation.

The cognizant chemist may choose (and is encouraged) to chart other parameters that assist in monitoring instrument or preparative procedures. Such examples include internal check standards, instrument control standards, signal intensities, lamp energies or background levels.

The control charts will be maintained in such a way that they are readily available to the laboratory technologist, cognizant chemist, QA/QC representatives or management. Control charts shall be maintained current for immediate use and may take the form of at-the-bench manually updated charts, on-line electronic displays (e.g., generated by the LIMS or by application software), or hard copy printouts generated periodically as close to real-time as practical.

The technical staff will be trained on the maintenance and use of control charts. The technical staff is expected to use them for system monitoring and early trend detection as described above and to be able to use them to demonstrate that their measurement system is in control.

11.2 Preparative Techniques

Preparative techniques are used to prepare a sample for analysis. This may include sample digestion, dissolution, extraction, and/or leaching.

11.2.1 Batch

A batch is a group of samples with similar matrix processed (at sample preparation step) together. The batch contains all QC samples required for sample analysis.

11.2.2 Preparation Blanks

The sample preparation blank is generated by using deionized water or a material that contains none of the analytes of interest and is similar to the sample. The preparation blanks are subjected to the same sample preparation, treatments, and analysis as the sample in a batch. Blank results may be used to assess the quality of subsequent analysis. Blank results provide the information on contamination of the method analysis. Preparation blanks are prepared as required by the analytical procedures.

11.2.3 Laboratory Control Sample

The laboratory control sample (LCS) (either a LCS prepared from a suitable matrix or a blank spike) is used to monitor the effectiveness of the entire analysis process or a analytical system. The analyte or isotope of interest of known concentration is added to a suitable matrix and carried throughout the analysis.

Laboratory control sample control is demonstrated by target analytes or isotopes being within established control limits. Control limits are established by one of the following:

- Specifications by vendor;
- Specified by regulatory requirement; and
- Statistically determined by multiple analysis over time.

11.2.4 Matrix Spike

The matrix spike is used to monitor method performance in a specific matrix. The matrix spike is an aliquot of sample spiked with the analyte(s) of interest and processed similar to the original sample.

When the sample concentration is unknown, spiking is typically performed at one of the following levels:

- Equivalent to the regulatory threshold;
- Specified in the method; and
- 1 to 5 times the estimated quantitative limits (EQL).

Otherwise spiking should be performed at a level equivalent to that of the sample or at least 25% to that of sample.

Matrix spike control is demonstrated when target analytes are within established control limits. Control limits are established by one of the following:

- Regulatory requirement;
- The client via data quality requirements for a particular project or program; and
- Laboratory performance.

The recommended target level for matrix spike analysis is 75 - 125% recovery. Control limits are not applicable to spikes that are <25% of the analyte concentration in the sample. Spike recoveries outside this range will be flagged and explained in the narrative, not necessarily generating an automatic reanalysis. Minimum spike frequency will be 5%, one per matrix if less than 20 samples are analyzed per matrix, or as requested by client.

A matrix spike may not be applicable if the analyte concentration in the sample is very large (>0.1%) where addition of large amounts of spikes are not practical because of solubility concerns. Other methods of evaluating method performance such as serial dilution or post-digestion spike may be used.

11.2.4.1 Inorganic Chemistry

The recommended criteria for most inorganic analysis is recovery within 75 - 125%.

11.2.4.2 Organic Chemistry

Acceptance criteria for organic compounds will be stated in the procedure or QAPjP based on method performance or specified by regulatory methods. For compounds of interest not covered by SW-846, the laboratory shall establish spike compound and acceptable level according to Section 8.0.

11.2.4.3 Radiochemistry

In radiochemistry, the matrix spike represents the addition of a known quantity of the isotope of interest to an aliquot of sample. Radiochemical analysis may include either a matrix spike or a post digestion spike (See Section 11.3.8.3); the decision is based on the activity level present in the sample. Spiking additional activity into a sample that already exhibits high activity is <u>not</u> justifiable. In such cases, spiking is generally performed after preliminary sample preparation, but before any additional sample handling except large dilution. However, activity levels in such cases should always meet or exceed the decision or action limit to provide sufficient count rate that the counting error for the spike is significantly lower than the data recovery requirements. Radiochemical techniques typically employ either a tracer, carrier, or matrix spike, <u>or</u> a combination of a matrix spike with a tracer or carrier.

11.2.5 Sample Duplicate or Matrix Spike Duplicate

Duplicates are used to assess the precision of the preparation process. Although more replicates can be requested, typically duplicates are required as a minimum. Duplicates are two aliquots of the same sample that are taken through the entire sample preparation and analytical process. Matrix spike duplicates are two spiked aliquots of the same sample that are taken through the entire sample reparation and analytical process. Precision is estimated by calculating the RPD. To the degree possible, the laboratory and the client should agree upon the use of sample duplicates versus matrix spike duplicates before starting work.

Minimum frequency for sample duplicates will be 5% or one per matrix type, whichever is greater.

11.2.5.1 Inorganic Chemistry

Typically, inorganic preparations include a sample and a sample duplicate because a high probability exists that the analyte(s) of interest will be detected in the sample.

Inorganic duplicate RPD criteria is normally set at 20%; this criteria shall only be applied to samples with analyte concentrations greater than 10 times the methods detection limit or 50 times the instrument detection level.

11.2.5.2 Organic Chemistry

Organic preparations usually rely on a matrix spike or matrix spike duplicate to determine precision, because commonly, a low probability exists that the analyte(s) of interest will be detected. In this case, precision of sample is determined from the analytes spiked into the matrix spike and the matrix spike duplicate.

Organic matrix/spike duplicate RPD criteria control is demonstrated by target analytes being within established control limits. Control limits are established as follows: 1) specified by regulatory requirement; or 2) specified by the client via the DQOs for a particular project or program. For compounds of interest not covered by SW-846 or the CLP, the laboratory shall establish acceptable precision criteria according to Section 8.0.

11.2.5.3 Radiochemistry

Typically, radiochemical preparations include a sample and sample duplicate because a high probability exists that the analyte(s) of interest will be detected in the sample.

A radiochemical duplicate RPD criteria of 20% can also be achieved provided the isotope activity or concentration that has an uncertainty, or counting error, less than 20%. The laboratory should evaluate performance against the RPD.

In all cases (i.e., inorganic, organic, radiochemistry) if the RPD falls outside the established limits the results are flagged and explained in the narrative. It does not automatically trigger re-analysis.

11.2.6 Surrogate

A surrogate is a compound or analyte that is added to all samples during preparation. The surrogate is typically similar in chemical composition to the compound or analyte being determined, yet not normally encountered in most samples. Criteria for selection and recovery of surrogates is generally specific to the method and compounds being detected. Each method that uses surrogates shall specify instructions for surrogate introduction and use. Surrogate recoveries are normally reported as is measured (i.e., no sample recovery corrections are performed based on surrogate recovery).

11.2.6.1 Inorganic Chemistry

Surrogates are typically not applied.

11.2.6.2 Organic Chemistry

Most, if not all, organic techniques employ surrogates; in this case, surrogates are also added to all standards and QC samples. The criteria is based on 1) regulatory requirement; 2) laboratory performance, three standard deviation; and 3) or client via data quality requirements for a particular project or program.

11.2.6.3 Radiochemistry

Surrogates are typically not applied.

11.2.7 Tracer -- Radiochemistry Only

A tracer is used to monitor method performance in a specific matrix. A tracer represents the addition to an aliquot of sample a known quantity of an isotope that is different from that of the isotope of interest but expected to behave similarly. Criteria for selection and recovery of tracers shall be specified in each method as use may be considered unique to the specific isotope being determined. Sample results are normally corrected based on yield recovered on a tracer.

The tracer added to the sample shall be well mixed in the sample in order to reach an equilibrium between the tracer and the isotope of interest. Otherwise, the recovery of tracer does not represent performance of the isotope of interest. Activity of tracer should exceed the decision or action limit to provide sufficient counts. The tracer recovery is calculated and is used to correct sample results based on the tracer recovery.

11.2.8 Carrier -- Radiochemistry Only

A carrier is used to monitor method performance in a specific matrix. A carrier represents the addition to an aliquot of sample of a known quantity of a stable isotope that is expected to behave similarly to the isotope of interest. Criteria for selection and recovery of carriers shall be specified in each method as use may be considered unique to the specific isotope being determined. Sample results are normally corrected based on the yield recovered on a carrier. Radiochemical techniques typically employ a matrix spike (Section 11.2.4), tracer (Section 11.2.7), carrier, or a combination of a matrix spike with a carrier or tracer.

11.3 Analytical Techniques

11.3.1 Analytical Run or Sequence

An analytical run or sequence is defined as a group of samples analyzed together that may include one or more preparation batches (See Section 11.2.1). Analytical QC is used to define the boundary of each analytical run. The analytical run typically starts with either calibration or confirmation that the calibration is still valid.

11.3.1.1 Inorganic Chemistry

For most inorganic analyses, the run ends based upon continuing calibration performance.

11.3.1.2 Organic Chemistry

For organic analyses by gas chromatograph/mass spectrometry (GC/MS), the run ends based on analytical clock expiration. For the gas chromatography (GC) the run ends based upon continuing calibration performance.

11.3.1.3 Radiochemistry

For radiochemistry, the run ends at the last sample or based on time.

11.3.2 Initial Calibration Verification

The ICV is a standard used to confirm acceptability of the most recent calibration. Standards for ICV are prepared from a source other then that used to prepare the calibration standard.

Analytical measurement systems that are calibrated frequently and for which calibration standards are routinely prepared normally follow initial calibration with an ICV.

Analytical measurement system for which calibration applies over an extended period of time (i.e., months for some GC/MS methods to years for many radiochemical methods) normally use the ICV only at the time of initial calibration. Subsequent, routine performance checks are made using the equivalent of a continuing calibration verification (CCV) (See Section 11.3.3).

Acceptance criteria will be stated in the procedure as defined by the specific methods, e.g., "USEPA Methods for evaluating Solids, Physical/Chemical Methods (SW-846)".

11.3.3 Continuing Calibration Verification

The CCV is used to monitor instrument stability over time. Acceptable performance demonstrates the continued appropriateness of the calibration, indicating that the measurement system is still in control. The CCV may be prepared from any reliable source.

11.3.3.1 Inorganic Chemistry

Each inorganic analytical system shall include periodic checks on the stability of the instrument. For unstable analytical system, these checks will be performed every 10 samples and at the end of the analytical run. Failure indicates that the analytical system has drifted out of control and requires corrective action for the analytes of interest. If CCV failure occurs, all samples analyzed after the last acceptable CCV shall be reanalyzed. Reanalysis applies to specific analyte failure. In limited cases, isolated analyte failures may be tolerated if sample results still meet the client data quality requirements. Reporting results in such cases requires justification in the report to the client.

11.3.3.2 Organic Chemistry

Organic analysis by GC/MS is limited by analytical run time. Each run is defined by a 12-hour clock that cannot be exceeded. In this case, periodic CCVs are not required. The analyst shall rely on internal standard and surrogate performance to ensure that the sample run ended in control. Corrective action, such as reanalysis of all samples demonstrating unacceptable internal standard performance, shall be taken. Analytical runs for organic analysis such as GC typically can extend anywhere from several hours to several days. CCV checks are required based on specific methods.

11.3.3.3 Radiochemistry

Many radiochemical analyses are limited by the samples that can be analyzed in one run. In such cases, periodic CCVs are impractical. However, each analytical sequence shall be followed by an acceptable CCV during the next analytical sequence. Failure justifies corrective action and applies to all samples run since the last acceptable check. If no additional standard or spike information is present at the end of the preceding run, all data generated since the last acceptable standard or QC sample shall be considered suspect and investigated. The CCV is commonly referred to as the counter control standard in radiochemistry (see also Section 7.0). For example, daily calibration checks are made on GEA using a pre-prepared check standard.

11.3.4 Initial and Continuing Calibration Blanks

Initial and continuing calibration blanks monitor affects such as contamination and instrument drift. The initial and continuing calibration blank can be accomplished by an instrument check blank.

11.3.4.1 Inorganic Analysis

Blank analysis should always follow standard analysis to identify any carry-over effects.

11.3.4.2 Organic Analysis

Blank analysis is accomplish via the method blank. The method blank is run after the CCV standard. Periodic calibration blanks are not performed. In the case of analysis by GC, periodic blanks are recommended.

11.3.4.3 Radiochemistry

Most radiochemical techniques use instrument background count measurements at least daily. Background counts are a measure of system and/or environment contributions, and a fundamental aspect of the minimum detectable activity (MDA) determination. Background counts are collected when the instrument is not in use for sample analysis. Background counts on alpha/beta counters are subtracted from all subsequent sample counts. These may be either daily or window average (stated windows duration) after determining the daily value is within accepted limits. Gamma spectroscopy uses a fixed background subtract with daily monitoring to determine acceptability. Radiochemical background counts are similar to that of the initial calibration blank.

11.3.5 Internal Standards

Internal Standards are used in the ICP spectrometry analysis although they may be appropriate to other types of analysis, when multiple analytes are being analyzed together.

Internal Standards can be used to correct for analytical problems such as instrument drift, pipetting variation, and injection technique.

Selecting appropriate internal standards shall be method- and compound-list specific because all results are normalized based on internal standard performance. Laboratory procedures shall specify requirements for internal standards and acceptance criteria. Internal standards are added after sample preparation and before analysis.

11.3.6 Low-Level Standard

The low-level standard is used to monitor instrument performance in the region at or just above the EQL (see Section 12.5.1.3). When sample dilution is required, it is preferable to adjust sample size so that results are mid-range on instrument calibration curves.

11.3.7 Interference Check Standards

Interference check standards are typically applied only in ICP spectrometry systems. The interference check normally consists of two standards. The first standard contains known concentrations of the interfering elements that will provide an adequate test of correction factors. The second standard contains both the major interferents and the majority of other analytes tested. The major interferents are spiked into the standards at significant concentrations that are expected to produce an interference effect. All other analytes are spiked at relatively low levels. Data from both standards, when corrected, should recover between 80% and 120% for all analytes tested or inter-element interference is considered inadequate. The first standard, containing only the primary interferents of concern, should produce no analyte concentrations in excess of the EQL. Instruments capable of showing negative results do not require the second standard that contains both interferents and additional analytes tested. If significant interferences are observed the instrument inter-element correction factors should be re-evaluated.

11.3.8 Post Digestion Spike

A post digestion spike is a spike added to the sample after preliminary preparation, usually just before analysis. If a matrix spike is used, the sample spiked would be that sample on which a matrix spike was originally performed. The post digestion spike is used for indication purposes. It provides the analyst with information regarding matrix-related interferences on the analytical system that may or may not still be present in the sample following digestion. Acceptable recovery is generally 75% to 125% for this spike. The analyst should use caution when interpreting recovery failure, such failure could also be the result spectral interferences. This technique is typically used for ICP spectrometry analysis, but is appropriate to other techniques as well.

11.3.9 Serial Dilution -- Inorganic Only

Many inorganic techniques such as ICP spectrometry use a serial dilution. The serial dilution analysis is typically used when new or unusual matrices are encountered. When the sample analyte concentration is less than 50 times the instrument detection limit (IDL), an analytical spike should instead be performed. Although not specially required, serial dilution can be used for other techniques.

Serial dilution is simply a five-fold dilution of a sample followed by analysis. This technique is another indicator of potential matrix-related interferences associated with analysis. Serial dilution is only performed when a sufficient number of analyte concentrations exceed 50 times the IDL. The sample concentration should, therefore, be a minimum of 10 times the EQL before performing the 1 to 5 dilution. A percent difference of 10% or less indicates acceptable performance. The sample dilution is not applicable to analytes whose concentrations is reduced to <10 IDL when diluted.

The serial dilution is not meant to replace a sample dilution necessary to maintain a sample in optimum instrument performance range. The serial dilution is designed to indicate potential problems such as high solids. In these cases, results would begin to vary beyond the 10% criteria because of sample aspiration and the subsequent effect on analyte species detected.

11.3.10 Analytical Spike - Graphite Furnace - AA

An analytical spike is similar to a post digestion spike (See Section 11.3.8) in that a spike that is added to the sample just before analysis. Typically, a very small quantity of spike is added so no significant change occurs in sample volume or matrix results. The concentration spike should equal 50% to 100% of the sample analyte concentration or approximately two times the EQL if no analyte is expected or the concentration is unknown. The analytical spike is applied to every sample. Recoveries outside of 85% to 115% warrant investigation and corrective action. Corrective action may consist of dilution followed by reanalysis, repreparation of the sample followed by reanalysis or in extreme cases the use of standard additions.

11.3.11 Method of Standard Additions

The method of standard additions involves adding known amounts of a blank and standard to aliquots of the sample. The method of standard additions is meant to compensate for a sample effect that enhances or depresses the analyte signal. A method of standard additions can be used in lieu of instrument calibration because each sample essentially has its own calibration. The standards used should be approximate 50%, 100%, and 150% of the expected sample concentration. When the method of standard additions is employed, instrument calibration and periodic calibration verification using standards and blanks is <u>not</u> required.

Method of standard additions or analytical spike is required for all Extraction Procedure Toxicity and Toxicity Characteristic Leaching Procedure sample.

References:

ASTM D 1193-77 (Reapproved 1983), "Standard Specification for Reagent Water"

SD-CP-LB-028, "Laboratory Reagents"

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12.0 PROCEDURES TO ASSESS DATA QUALITY

This section provides various formulas that are typically used to compute QA parameters that are used to assess data quality.

12.1 Precision

Precision is estimated by using duplicate and/or replicate analyses. Samples used to calculate precision should contain the concentrations of analytes above the method detection limit (MDL). The precision of test results is expressed as the RSD or the RPD. The precision of a method in a given matrix is expressed as the RSD or the RPD among matrix spike duplicates.

12.1.1 Relative Standard Deviation

The RSD is used when at least three replicate measurements are performed on a given technique. The RSD is computed using the following equation:

$$RSD = \frac{s}{\bar{x}} * 100$$

where

s = standard deviation with n - 1 degrees of freedom
n = total number of observed values
$\bar{x}$ = mean of observed values.

12.1.2 Relative Percent Difference

The RPD is used when two measurements exist. The RPD is generally used to express the precision of matrix duplicate or matrix spike duplicate samples. The RPD is computed using the following equation:

$$RPD = \frac{|x_1 - x_2|}{\bar{x}} * 100$$

where

$x_{1,2}$ = observed values
$\bar{x}$ = mean of observed values.

12.2 Accuracy

12.2.1 Method Accuracy Based on Sample Spike

Accuracy is expressed as the percent recovery (%R) of a matrix spike (or matrix spike duplicate) sample. The percent recovery is calculated based on the following equation:

where:

SSR = spiked sample result or tracer and/or carrier sample result
SR = sample result
SA = spike added or tracer and/or carrier added.

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

The percent recovery of the tracer and/or carrier in the radiochemical analysis can be used as the yield percent recovery to correct analyte recovery in the sample.

12.2.2 Method Accuracy Based on Standard

The accuracy of an analytical method is expressed as the %R of a standard. The percent recovery of a standard is calculated according to the following equation:

$$\%R = \frac{A_m}{A_k} * 100$$

where

A_m = measured value of the standard analyte
A_k = known value of the standard analyte.

The percent recovery in the radiochemical analysis can be used as the yield percent recovery to correct analyte recovery in the sample.

12.3 Measures of Agreement

12.3.1 Percent Difference

The percent difference (%D) is often used to compare one reference point to another (e.g., average response factor (RF) from initial calibration compared to RF from continuing calibration listed in Section 12.1.2). The %D is calculated using the following equation:

$$\%\ D = \frac{|I - C|}{I} * 100$$

where

I = observed value used as the reference point
C = compared value.

12.3.2 Bias

Bias is often used to measure the deviation of a measured value from a known value or accepted reference value. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample. Thus, the bias caused by the matrix effects based on a matrix spike is calculated using the following equation:

$$B = (X_s - X_u) - K$$

where

X_s = measured value (e.g., spiked sample)
X_u = miscellaneous contribution (e.g., sample contribution)
K = known value (e.g., true spiked value).

The %R could then be determined as follows:

$$\%R = 100\ (X_s - X_u)/K$$

If no miscellaneous contributions exist, X_u would be zero.

12.4 Detection Limit Considerations

12.4.1 Inorganic and Organic Methods

12.4.1.1 Method Detection Limit

The MDL is defined as "the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is greater than zero" (SW-846, consistent with the requirements specified in 40 CFR 40 Appendix B to 40 CFR 136) and is briefly described in the following text.

The concentration of the MDL for the analyte of concern can be estimated by using one of the following:

- An instrument signal-to-noise ratio within the range of 2.5 to 5; and
- The region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

When determining the MDL, a minimum of **three** analyses are required in a matrix spiking with the analyte of interest at a concentration three to five times the estimated MDL. Whenever possible, the matrix should be similar to the sample matrix. All sample processing steps of the analytical method shall be included in the final determination of the MDL.

Variance (S^2) is determined from the replicate measurements, as shown:

$$S^2 = \frac{1}{(n-1)} \left[\sum_{i=1}^{n} (X_i - \bar{X})^2 \right]$$

where

X_i = with measurement of the variable X
$\bar{X}$ = mean of observed variable X.

The MDL should be determined by the following equation:

$$MDL = t\ (n-1,\ \alpha = .99) * (s)$$

where

$t_{(n-1,\ \alpha=.99)}$ = one-sided t-statistical value appropriate for the number of samples used to determine standard deviation

s = standard deviation obtained from the MDL replicate measurements.

The MDL is determined at least quarterly for all analyses. Unless specified by a special analytical project, the laboratory will use the water-based standards for determination of MDL.

12.4.1.2 Instrument Detection Limit

The IDL is determined by spiking reagent water with each analyte of concern. The following considerations apply to the selection of the IDL standard:

- Concentration of the IDL standard should be at least equal to or in the same concentration range as the estimated IDL; and
- Concentration of the IDL standard should be in the region of the standard curve where there is significant change in sensitivity.

A minimum of seven aliquots of the IDL standard are required to determine the IDL. The standards used for IDL determination are run through analytical process only (are not run through preparative steps).

12.4.1.3 Estimated Quantitation Limit

The EQL has been defined by RCRA as the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL should be calculated at 5 to 10 times the IDL or MDL. However, the uncertainty increases as a lower factor is used. In limited applications, using the lowest standard in a calibration curve is more appropriate. This decision shall take into account the data quality requirements from the end-user of the data.

12.4.2 Radiochemistry Methods

12.4.2.1 Decision Level Count Rate

The decision level count rate (DLR) is defined as a 95% confidence limit for a critical decision level. This level is used for making a decision as to whether a sample emits radiation above the appropriate blank background level. The decision should be based solely upon whether the net count rate observed for that sample exceeds this DLR. The DLR is calculated as shown below:

where

R_b = background count rate
T_b = background count time

$$DLR = 1.65 * \sqrt{(\frac{R_b}{T_b}) * (1 + \frac{T_b}{T})}$$

T = sample count time
R_s = sample count rate.

When counting a sample containing no analyte (radionuclide) of interest, R_s is assumed to be equal to R_b. The DLR can be simplified as shown below:

$$DLR = 1.65 * (S_b) * \sqrt{2}$$

where
S_b = standard deviation of background (or appropriate blank) count rate for the counting time (T).

12.4.2.2 Minimum Detectable Activity

The MDA has been defined as a level of activity that is practically achievable by a measurement system. The sample MDA generally is applied as the mean (expected) activity of samples having a 5% probability of escaping detection. The MDA is calculated based on Currie's (1968) formula and is simplified to the following two equations when the counting time in the sample is the same as in the background. It must be recognized that the background may and normally is affected by the components of the sample. Therefore, the MDA value is an (a posteriori) estimate for comparative purposes. The MDA is expressed in μCi.

$$MDA = [(\frac{2.71}{T}) + (2 * DLR)] / K$$

or

$$MDA = [(\frac{2.71}{T}) + (4.65 * S_b)] / K$$

where
T = sample count time
K = detector calibration factor ($min^{-1}/\mu Ci$ or $s^{-1}/\mu Ci$)
S_b = standard deviation of background count rate for the counting time (T).

When T_b is not equal to T_t, MDA is calculated as shown below.

where
R_b = background count rate
T_b = background count time

$$MDA = \frac{2.71 + 3.3\sqrt{(R_b * T_b) * \left[1 + \frac{T_b}{T_t}\right]}}{e * b * LT * k}$$

T_t = sample count time
e = counting efficiency
b = abundance
L_T = elapsed live time (background counting time = T_b)
k = 37,000 disintegrations/second/μCi.

The MDC is defined as the mean concentration of samples having a 5% probability of escaping detection, expressed in μCi/g or μCi/mL.

$$MDC = \frac{MDA}{q * Y * decay}$$

where

q	=	Sample quantity (g or mL)
Y	=	Chemical yield
decay	=	decay factor (correction for radioactive decay to reference date).

Software provided by vendors may have variations of the above formula. A vendor-provided software or data reduction package is adequate for data calculation.

12.5 Uncertainty

Uncertainty is expressed as the range of values in which the true value is estimated to lie. The uncertainty estimate consists of two components, systematic and random variability. Each contributing source of uncertainty is expected to be distributed over its range. Each systematic component can be estimated in terms of the measurement result for the contributing source of uncertainty.

The analytical systematic component can be estimated using standard or spike recovery. The random analytical component can be estimated from replicate measurements of a sample.

The total uncertainty is calculated as the square root of the sum of the squares of random and systematic variabilities as shown in the following equation. The component of uncertainty has to be expressed in the same unit designation (e.g., concentration percentage).

$$\text{Total uncertainty} = \sqrt{(s_x^2) + \sum_{j=1}^{q} \delta_j^2}$$

where

s_x = standard error
q = number of systematic uncertainty component
δ = systematic uncertainties.

Uncertainty is used in the radiochemical analyses to express method and counting error. The total random uncertainty is obtained by propagating the individual variance (s_i^2), and is expressed as the standard error based on multiple determinations of x. However, the typical radiochemical methods used are not sufficient to separate systematic and random uncertainties such that biases can be corrected. Uncertainty will be measured, or uncertainty will be estimated if it cannot be measured.

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13.0 AUDITS

The QA program for the WSCF and Standards Laboratories consists of management system audits, technical systems audits, performance audits, data quality audits, and external audits. The format and distribution of internal assessment results are described in the WHC-CM-5-4 Sections 8.5 and 8.5A.

Results from the external audits are tracked by QA functions. Corrective Actions are initiated by QA/QC staff and distributed to the effective manager and staff. QA/QC officers are responsible for tracking the implementation of corrective actions.

13.1 System Audits

Management system audits are directed by laboratory management. These audits are required to be conducted annually at a minimum. The purpose of these audits is to assess the following:

- Systems for developing technical procedures;
- Quality and applicability of current management systems (e.g., QA manual, administrative procedures);
- Procedures for the design and conduct of audits;
- Systems for tracking quality adherence (i.e., performance indicators); and
- The degree of management support as well as current roles and responsibilities.

Laboratory QA/QC personnel are responsible to coordinate and evaluate technical systems audits. Technical system audits are performed quarterly. Each system audit focuses on a particular aspect of laboratory operation. Aspects of technical system shall be audited once per year. Technical systems audits consist of a review of laboratory operations, procedures, and documentation. Any deficiencies and/or deviations shall be documented and provided to that laboratory management for corrective action initiation.

The laboratory QA representative shall be responsible for documenting that corrective actions have been initiated, follow-up verification of corrective action and closeout of deficiency document, and maintaining all completed responses on file.

13.2 Performance Audits

Laboratory QA function is responsible for coordinating and evaluating performance audits. Performance audits consist of laboratory analyses of standard materials that are used to evaluate analyst and method proficiency. Performance audits may be driven either externally or internally.

13.2.1 Inter-Laboratory Comparison Studies

Inter-laboratory Comparison Studies are used to evaluate the overall performance of the laboratory. The types of inter-laboratory comparison studies are analysis-specific and generally categorized as organic, inorganic, and radiochemical analyses.

Two programs are currently implemented for radiochemical analyses: the EPA Environmental Monitoring Systems Laboratory - Las Vegas, and the DOE Environmental Measurements Laboratory - New York.

Two other programs are currently implemented: the EPA Performance Evaluation - Quarterly Blinds (for inorganic and organic analyses), and the EPA Water Pollution Study (mandatory for NPDES major discharger, DMR-QA Program) for inorganic metals, anions, volatile organic analyses, total organic carbon, biochemical oxygen demand, and pesticides/polychlorinated biphenyls.

A new program, Water Supply, will be implemented as requested for the EPA drinking water program.

The Laboratory QA officer is responsible for delivering comparison samples to the laboratory, coordinating of schedule, delivering results to the program offices, and evaluation of performance results. Corrective action will be issued for the areas of performance failure or as required by program protocol. The QA officer will issue a Nonconformance Report as required by WHC-CM-4-2, Sections QIs 15.1 and 15.2.

All noted deficiencies shall be investigated, resolved, and documented by the appropriate laboratory personnel. All documentation of corrective action shall be maintained in accordance to WHC-CM-1-4, Section 2, and WHC-CM-4-2, Section 16. In addition, OQA is responsible for establishing and maintaining an assessment data base for tracking and trending from the annual assessments (WHC-CM-5-4, Section 8.5).

13.3 Data Quality Audits

Laboratory QA function is responsible to perform data quality audits (also known as data quality reviews). The WSCF performs data quality audits on each data package. The objective is to determine if adequate information and documentation exist within a given data package to support an assessment of its quality. Furthermore, the degree of conformance to client data quality requirements shall be evaluated and documented.

13.4 External Audits

External audits of the laboratory are performed by agencies or groups that are not under the control of laboratory management. As a minimum, Department of Ecology will perform an audit before granting the accreditation. External audits may consist of inspections, interviews, and/or evaluations that focus on the laboratory's ability to meet client, program, and/or regulatory requirements. Laboratory management shall be responsible for initiating, tracking, following-up, and documenting all corrective actions that are required as a result of external audits.

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14.0 PREVENTIVE MAINTENANCE

Most laboratory instruments require some minor maintenance activities or adjustments performed by the analyst as part of its setup or operation. The analytical procedure lists such activities as steps within the method. Laboratory personnel follow the procedure each time they use the instrument. Each instrument shall have a logbook to record maintenance events with date and name of the personnel performing the maintenance. Logbooks are maintained in accordance with WHC-CM-5-4, Section 6.11, and LO-150-401.

Some instrument systems have mechanical components that are included in the WHC preventive maintenance system. The WSCF has a vendor service contract in place, e.g., routine and/or emergency service for the GFAA, GC, LSC, ICP, and GC/MS. Instruments, e.g., Proportional Counter and GEA, for which service contracts are not in place may be contracted to the vendor on as needed basis established by the responsible scientist.

The concept of preventive maintenance is to perform local maintenance on instruments until they will no longer run (run to failure) then use the contract vendor and spare parts to provide timely repair. The 222-S Facilities can be used for backup in the event of a prolonged outage and thus, prevent interruption of customer service.

Spare parts inventories may include day-to-day consumables or other known-to-fail parts maintained at the discretion of the cognizant scientist. Spare parts may be formally maintained in the WHC Spare Parts Inventory, administered at the WSCF by the AS Laboratory Engineering group. The current status of the this inventory can be queried through the Hanford Inventory Program (HIP), available on the local area network (HLAN) through Soft Reporting. Alternatives to spare parts inventories which help ensure minimal loss of analytical capacity include:

Overnight parts availability from suppliers

- Availability of back-up instrumentation (see "Redundant Capacity" below); and
- Vendor service contracts.

WHC documents that include preventive maintenance guidelines or policies:
WHC-CM-4-2 Quality Assurance Manual:

- QR 12.0 Control of Instruments; and
- QI 12.7 Assuring Availability of Laboratory Instruments.

WHC-CM-5-4 Laboratories Administration:

- 10.1 Instrument Preventive Maintenance Program.

15.0 CORRECTIVE ACTION

The corrective action process consists of the identification of an adverse condition or deficiency, root cause analyses, determination of corrective action, and documentation. When fully implemented, this process is the basis for continuous improvement. The laboratory will follow guidelines from the "Corrective Action Management System", Management Requirements and Procedures, WHC-CM-1-1, MRP 5.1, WHC-CM-4-2, QI 16.0 - 16.2, and/or WHC-CM-5-4 "Laboratory Administration", Sections 6 and 8.

The corrective action process shall ensure that laboratory personnel at all levels are responsible for initiating corrective action when conditions may adversely impact laboratory systems (e.g., administrative, analytical, operations).

Examples of conditions where corrective action shall be implemented are:

- Documentation errors;
- Adverse trends in the analysis of standards;
- Failure to comply with approved procedures;
- Failure to follow the preventive maintenance program;
- Failures in the instrument systems;
- Failures in performance evaluation sample analysis;
- Non-compliance issues identified by audits, surveillances and assessments;
- Validation and/or verification issues negatively impacting reported results; and
- Failure to follow client analytical requests and/or DQOs - this condition may be addressed as part of the narrative in the data package report.

15.1 Evaluating Impact

The corrective action processes listed above describe the provisions for 1) determining the significance of the problem; and 2) taking effective corrective action based on the potential impact on the data quality. The process can be initiated by submitting a fact sheet, critique, unusual occurrence report, non-conformance report or letter of observation.

Instrument controllers (computers) provide near-real-time support of management QC decisions. When a QC sample or standard analysis exceeds a control limit, a "corrective action report" is generated.

QC charts are continuously updated to indicate performance and trends.

Whenever QC limits are exceeded, the appropriate corrective action specified in the analytical procedure or QAPjP must be made, and a corrective action statement entered into the analyst's logbook and the "control report" noting the QC deficiency and corrective action taken.

When certain QC results fail to meet the control limits, the responsible chemist or manager is required to override data entry to the LABCORE. Sample results from the same batch associated with this failing QC result shall be entered. In this case, sample results shall be flagged with appropriate qualifiers or samples shall be re-analyzed depending on the types of QC failure.

Implementation of corrective actions shall be verified as appropriate. When corrective actions involve a measurement system, the corrective response will be fulfilled when the flagging is complete and the narrative documents the QC problems.

15.2 Recurring Conditions Adverse to Quality

These processes are established in the procedure utilized to correct the problem. The measures to eliminate or minimize recurrence of quality problems shall be established utilizing the following provisions. These determinations shall include but not be limited to the following:

- Determine the events leading to the adverse condition;
- Understand the technical and work activities associated with the quality problem;
- Ascertain the quality problem's generic implications;
- Determine the extent to which similar quality problems (or precursors to the problem) have been recognized;
- Determine the effectiveness of any corrective actions that were taken;
- Determine the impacts on the completed work;
- Recommend actions that can be taken by the responsible organization to preclude recurrence; and
- Determine if stopping the work associated with the activity is necessary.

15.3 Trend Analysis

Analysis of quality-related information shall include, where possible, identifying common work processes for quality problems, conducting cause-and-effect analysis, and determining effective corrective and preventive actions from internal sources, including other U.S. DOE facilities or sites.

Quality related information to be analyzed shall include, but are not limited to the following, as appropriate:

- Performance data;
- Audit reports;
- Surveillance reports;

- Non-conformance reports;
- Quality-related information from external sources (not limited to one type of work, one facility, or one contractor); and
- Performance Indicators.

15.4 Root Cause Analysis

The extent of the root cause analysis shall be commensurate with the importance or significance of the problem (see WHC-CM-1-4 for reference).

15.5 Continuous Quality Improvement

Quality improvement is a continuous process and is designed to reduce the variability of every process that influences the quality of the product. The concept is used throughout this QA program. Laboratory activities, analytical measurements and results, and QA/QC activities are documented and are traceable for evaluation. For example, management or personnel can analyze the performance indicators, corrective actions, or assessment to identify actions as a means of continuous quality improvement. In addition, information obtained from lessons learned evaluation, safety analysis report, quality environmental safety tracking, peer reviews, or probabilistic risk assessments also are used for management planning and problem prevention as a means of continuous quality improvement.

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16.0 QUALITY ASSURANCE REPORTS

REPORTS	AUTHOR	APPROVED	FREQUENCY	DISTRIBUTION	CORRECTIVE RESPONSIBILITY	TYPE OF DOCUMENT
QA Status	LQA Staff	N/A	Monthly	Lab Management	Lab Management	Internal Letter
Topic Audit Ext. Assess.	OQA Staff	OQA Manager	Annual	Lab Management	Lab Management	Internal Letter
Topic Audit Int. Assess.	OQA Staff	OQA Manager	Quarterly	Lab Management WHC-QA	Lab Management	Internal Letter
QA Data Review	LQA Staff	N/A	Ad hoc	Lab Management Technical Staff	Lab Management	Internal Form
Responses Data Review	Technical Staff & Management	LA Staff	Ad hoc	Lab Management	N/A	Internal Form
Letter of Observation	LQA Staff	N/A	Ad hoc	Lab Management	Lab Management	Internal Letter
Case Narrative	Technical Staff	P.C.	1/data Package	Data Package	N/A	N/A
Case Summary	P.C. Staff	P.S. Manager	1/data Package	QA File	N/A	Internal Forms
Ext. Audit/Assessment	Auditor	N/A	Ad hoc	Lab Management	Lab	Internal/External Letter
External Surveillance	AEQA Staff	AEQA Manager	Ad hoc	Lab Management WHC-QA	Lab	Internal Letter

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17.0 DATA VALIDATION

The project requirements must be defined in the specific QAPP, along with the systematic process to be used in verification or validation of project data. In cases where data must be validated; a specific set of acceptance criteria must be defined which provide assurance that the data generated are adequate for their intended use. The acceptance criteria reflect the requirements generated during the DQO planning process. The validation process will consist of data editing, screening, checking, auditing, verification, flagging, and review. The WSCF will not perform data validation; validation is independent of laboratory data review.

Acceptance criteria for validation purposes may include:

A. Holding times;

B. Preservative methods and container types;

C. Minimum required sample size, for analysis;

D. Calibration criteria and requirements (initial and continuing);

E. Detection limits;

F. Accuracy and precision definitions and requirements;

G. Field blank and preparation blank requirements;

H. Surrogate recoveries for organic compounds;

I. Tracer/carrier recoveries requirements for radiochemical analysis; and/or

J. Integrity of data transfer and result reporting.

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18.0 PROCUREMENT CONTROLS

The WSCF follows the WHC-CM-2-1, "Procurement Control and Manual", and WHC-CM-4-2, QR 4.0, "Procurement Document Control", and HASQAP to ensure procurement process ID documented and controlled. The controls ensure procured items and/or services can:

1. Conform to established specifications;
2. Meet acceptable quality; and
3. Perform as expected.

The subcontractor shall include organizations that provide services and organizations that provide supplies.

When there are indications that subcontractors knowingly supplied items or services of substandard quality, this information shall be forwarded to laboratory management for appropriate action (e.g., subsequent reporting to the U.S. DOE of the Inspector General).

References:

WHC-CM-2-1, Procurement Control

WHC-CM-4-2, Procurement Document Control, QR 4.0

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APPENDIX A

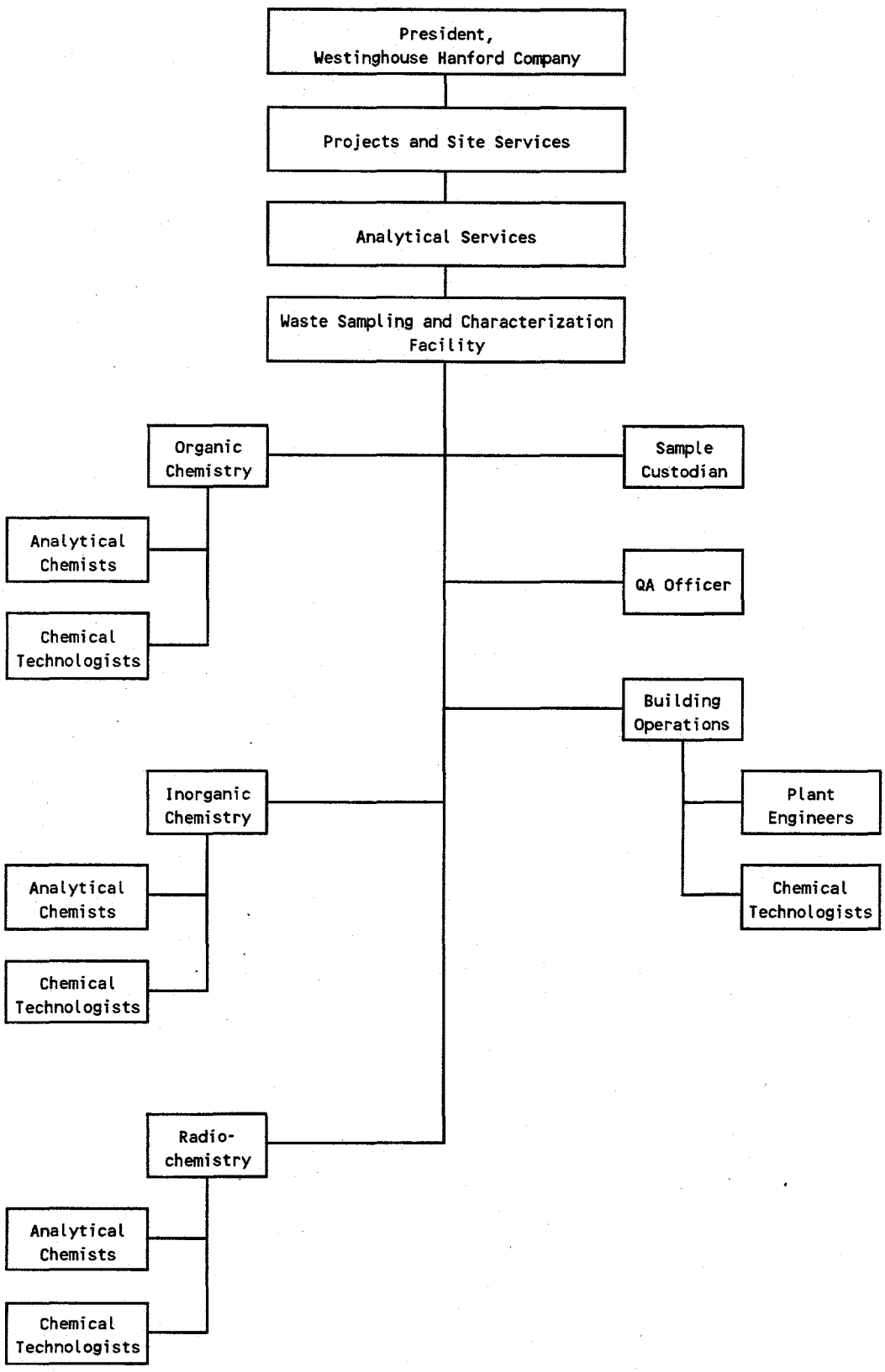
QUALITY ASSURANCE PROGRAM INDEX

QUALITY ASSURANCE REQUIREMENTS	DOCUMENTS - WHC	PROCEDURES - LABORATORY
1. Introduction	NA	NA
2. Organization and Responsibility	WHC-CM-4-2, QI 2.1	
3. Personnel Qualification and Training	WHC-CM-4-2, QI 2.6	WHC-CM-5-4, Sec. 4.0
4. Quality Assurance Objectives a. Data quality objectives b. Client data quality requirements		 Client's QAPjP, State of Work
5. Systems Quality Assurance a. Software systems b. Administrative systems	 WHC-CM-4-2, QR19.0 WHC-CM-5-4	 WHC-SD-WM-CM-002 Specific LC procedures WHC-CM-5-4
6. Sample Custody and Handling a. Chain of custody definition b. Holding times c. Sample receiving procedure d. Sample log-in and tracking procedure e. Laboratory internal chain-of-custody f. Sample disposal		 LO-090-402 or LO-090-403 Specific LO Procedures
7. Calibration	WHC-CM-4-2, QR 12.0, QI12.0, 12.2, 12.3, 12.4, 12.5, 12,6, 12.7	Specific LQ procedures
8. Laboratory Procedures a. Procedures and Supporting Document b. Change Control c. New Analytical Methods d. Modification of Required Regulatory Methods		 WHC-CM-5-4, Sec 3.9 WHC-CM-5-4, Sec 3.10
9. Data Collection, Reduction, and Reporting a. Data collection b. Data reduction c. Data review d. Data reporting		 Specific LA procedures Specific LQ procedures
10. Records	WHC-CM-3-5, Sec 5.0 and 9.0	
11. Quality Control a. Laboratory Quality Control b. Preparative Techniques c. Analytical Techniques		 Specific LO or LQ procedures Specific LA procedures Specific LA procedures
12. Procedures to Assess Data Quality		Specific LA procedures
13. Audits	WHC-CM-4-2, QR18.0	
14. Preventive Maintenance		WHC-CM-5-4, Sec. 10.0
15. Corrective Actions	WHC-CM-4-2, QR16.0, QI16.0 -16.2	WHC-CM-5-4, Sec 6.0 & 8.0
16. Quality Assurance Reporting		
17. Data Validation		
18. Procurement Planning	WHC-CM-2-1 WHC-CM-4-2, QR 4.0	

APPENDIX B

ORGANIZATIONAL STRUCTURE

Project Organization and Responsibility:



APPENDIX C

GLOSSARY

Note: Majority quality related terms are defined throughout the document, therefore, they are not listed in the Glossary.

accuracy	The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X - T, or the difference as a percentage of the reference or true value, 100 (X - T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in a system.
analyst	A person performing a measurement.
analyte	The element, isotope, specie, or characteristic of a measurement.
anomalies	Something different, abnormal, or peculiar, not easily classified.
assessment	The act or instance of assessing (appraisal); the act of reviewing, inspecting, testing, checking, conducting surveillance, auditing, or otherwise determining and documenting whether items processes or services meet specified requirements. The terms assessment and verification as used in DOE Order 5700.6C are synonymous; their use is determined by who is performing the work. Assessments are performed by or for senior management. Verifications are performed by the line organizations. For data, assessment encompasses verification and validation. Data assessment (verification and/or validation) can be performed within the laboratory and/or by an independent review agency at the discretion of the client to the criteria of the project.
client	The person or organization submitting work.
comparability	Expresses the confidence with which one data set can be compared to another.
completeness	A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.
consensus	A procedure, protocol, or guidance document issued by a professional standard organization based on extensive testing and peer review.
continuous	A program or system that monitors performance, evaluates trends, and implements
quality improvement	Changes based on trends.

APPENDIX C

GLOSSARY (Continued)

false negatives	A term that identifies the acceptance of a test or condition as false, when in fact it is true.
false positive	A term that identifies the acceptance of a test or condition as true, when in fact it is false.
matrix	The component or substrate (e..g., surface water, drinking water) that contains the analyte of interest.
nonconformance	A deficiency in characteristic, documentation, or procedure that renders the quality of an item or activity unacceptable or indeterminate.
out-of-control	A system is said to be out-of-control when it fails to meet preselected performance criteria.
precision	A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".
preventive	A program of instrument care based on scheduled activities and spare parts maintenance inventory designed to minimize instrument downtime.
qualify	To qualify laboratory staff or a subcontractor is to provide evidence of meeting a performance standard for fitness by training skill or ability for a designated purpose. To qualify analytical procedures or computer programs is to provide evidence of performance to meet the required standard criteria.
quality assurance	The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.
quality control	Routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.
reagent quality	An analysis or industry accepted grade that denotes purity or applicability for application.
regulatory	Those methods published or promulgated for laboratory use to meet the procedures requirement of a law or government rule.
representativeness	Expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.
traceable	A document trail that identifies the history of a sample, standard, or other material.

APPENDIX C

GLOSSARY (Continued)

valid	Having legal efficacy or force, well grounded or justifiable being at once relevant meaningful logically correct, appropriate to the end in view.
validation	An act, process, or instance of validating. For data, validation is the process by which the data and quality control information is assessed or compared against the client's requirements.
verification	An act or process of verifying. For data, verification is the process of comparing the reported data with the required information.
verifying	To establish the truth, accuracy, or reality.