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Revision 6

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Waste Sampling and Characterization Facility Quality Assurance Plan

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Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Fluor Hanford
P.O. Box 1000
Richland, Washington

Contractor for the U.S. Department of Energy
Richland Operations Office under Contract DE-AC06-96RL13200

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H. K. Meznarich, Fluor Hanford

June 2003

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Assistant Secretary for Environmental Management

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Revision 6

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LIST OF TERMS

AA	atomic absorption
ADP	Automated Data Processing
APG	Analytical Products Group, Inc.
AS	Analytical Services
ASTM	American Society for Testing Materials
BS	blank spike
CAM	Corrective Action Management
CATS	Corrective Action Tracking System
CCV	continuing calibration verification
CERCLA	*Comprehensive Environmental Response, Compensation and Liability Act*
COC	chain-of-custody
CPO	Characterization Project Operations
CWA	Clean Water Act
DCF	Document Change Form
DEG	deficiency evaluation group
DI	deionized
DLR	decision level count rate
DOE	U.S. Department of Energy
DQO	data quality objectives
DQR	data quality requirements
DS	data system
DTS	Deficiency Tracking System
Ecology	State of Washington Department of Ecology
ECO	Environmental Compliance Officer
EDT	Engineering Data Transmittal
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitative limits
ERA	Environmental Resource Associates
FEAD	Format for Electronic Analytical Data
FEB	Facility Evaluation Board
FC	field changes
FH	Fluor Hanford
GC	gas chromatograph
GEA	gamma energy analysis
GED	general equivalency diploma
HASP	Hanford Analytical Services Program
HASQARD	Hanford Analytical Services Quality Assurance Requirements Documents
HGET	Hanford General Employee Training
HEIS	Hanford Environmental Information System
HEPA	high-efficiency particulate air
HEHF	Hanford Environmental Health Foundation
HMC	Hazardous Materials Control
HNF	Hanford Nuclear Facility
HVAC	heating, ventilation, and air conditioning
IC	ion chromatography
ICP	inductively coupled plasma

ICV	initial calibration verification
ID	identification
IDL	instrument detection limit
IH	industrial hygiene
IRM	Information Resource Management
ITEM	Integrated Training Electronic Matrix
JCS	Job Control System
LA	laboratory analytical procedure
LC	laboratory computer procedure
LCS	laboratory control sample
LE	laboratory essential materials procedure
LEMIS	Liquid-Effluent-Monitoring Information System
LIMS	Laboratory Information Management System
LMS	Laboratory Measurement Systems
LO	laboratory operating procedure
LOI	Letter of Instruction
LR	laboratory reference material procedure
LT	laboratory technology procedure
LQ	laboratory quality control procedure
MAPEP	Mixed Analytes Performance Evaluation Program
MDA	minimum detection activity
MDC	minimum detection concentration
MDL	method detection limit
MS	mass spectrometry
NCR	Nonconformance Report
NIST	National Institute of Standards and Technology
NRIP	NIST Radiochemistry Intercomparison Program
NSL	Nuclear Spectroscopy Laboratory
OJT	on-the-job training
PCS	practical control sample
PE	Performance Evaluation
PHMC	Project Hanford Management Contractors
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Plan
QC	Quality Control
QS	Quality Systems
RCRA	Resource Conservation and Recovery Act
RDL	required detection limit
RF	response factor
RIDS	Records Inventory Disposition Schedule
RL	DOE, Richland Operation Office
RO	reverse osmosis
RPD	relative percent difference
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SD	supporting documents
SOW	statement of work

SPC	statistical process control
S/RIDS	standards/requirements identification document
SST	Sorbent Tube Train
SVOC	semi-volatile organic compounds
TST	Triple Sorbent Trap
VPRQL	Vapor Program Required Quantitation Limits
VOC	volatile organic compounds
WAC	Washington Administrative Code
WAP	Waste Analysis Plan
WFS	Waste Fact Sheets
WIPP	Waste Isolation Project Plan
WMH	Waste Management Federal Services of Hanford, Inc.
WSCF	Waste Sampling and Characterization Facility

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1.0 INTRODUCTION

The objective of this Quality Assurance Plan is to provide quality assurance (QA), implementation of regulatory QA requirements, and quality control (QC) specifications for environmental analytical service. This document implements the requirements of the U.S. Department of Energy (DOE)-issued Hanford Analytical Services Quality Assurance Requirements Documents (HASQARD) (DOE 1998, as amended) and the general requirements of the DOE Order 414.1A, *Quality Assurance.*

This document describes how the laboratory implements QA requirements to meet the federal or state requirements and/or identifies the procedural information that governs how the laboratory operates. The scope of work described in this document is subject to HNF-MP-599, *Quality Assurance Program Description and* PLN-03-QP-001, *Fluor Hanford Site Operations Quality Assurance Program Plan* (QAPP) and adds details specific to the laboratory. A QA Program Index is shown in Table 1-1.

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

A suspension of activities may be ordered by the QA Officer if conditions affecting quality have not been addressed by cognizant management.

Management's commitment to establish an effective quality program shall be implemented through the following:

- All levels of management accept responsibility for their organization's activities and are held accountable for achieving quality.
- Personnel are responsible for the quality of their own work.
- Management provides adequate resources and budget to support effective quality assurance practices that fulfill the customer's program goals and performance objectives.
- Management provides facilities, instruments, support equipment, and materials required to meet current project requirements.
- Laboratory personnel shall have acceptable qualifications and training for their specific job assignments.
- Documentation shall be controlled and maintained in a manner that ensures the laboratories can demonstrate compliance to appropriate requirements.
- Quality is achieved and improved by planned, systematic, self-assessments, and measured actions. This improvement is referred to as Continuous Quality Improvement.

- Corrective action management shall be established to manage, evaluate, correct, and track deficiencies. Corrective actions are identified and are tracked for closure.

- A graded application shall be utilized for all quality affecting activities at the WSCF Laboratory. The graded application process shall determine the appropriate level of analysis, documentation, and actions necessary to comply with requirements through the consideration of prescribed factors, such as the:

  A. Nuclear safety classification or hazard category of the item or activity.
  B. Relative importance to safety, safeguards and security.
  C. Magnitude of any hazard or risk involved.
  D. Adequacy of existing safety documentation.
  E. Impact/consequence of failure on programmatic mission of a facility.
  F. Particular characteristics of a facility or activity.
  G. Life-cycle stage of a facility.
  H. Complexity of items, services, or processes involved.
  I. History of problems at a facility, or within an item, service or process.

- The graded application process shall not be used to circumvent applicable QA, legal or contractual requirements. Rather, grading determines the extent to which controls within the QA criteria are applied. Items, services and processes that are not safety-related and that pose very low risk to projects require the least rigor in applying controls. No controls beyond those inherent in standard commercial practices are required.

Client specific or regulatory driven QA/QC requirements, if requested by the client, that differ from this document shall be described in a Statement of Work (SOW), Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPjP), or Letter of Instruction (LOI) authored by laboratory personnel or by the client with laboratory input. When inconsistencies occur, requirements specified in these documents supersede HASQARD or WSCF QAPP requirements. Modification or deviation from HASQARD is allowed via either data quality objective (DQO) process, negotiation between laboratory and client, and documentation.

Industrial hygiene (IH) analyses are performed at the WSCF Laboratory in accordance with the *Waste Sampling and Characterization Facility Quality Assurance Project Plan for Industrial Hygiene Analyses* (HNF-SD-CP-QAPjP-019). This document is based on the quality requirements of nationally recognized accreditation programs covering IH analyses.

Sampling, analytical, and other activities performed by the WSCF personnel that related to the Waste Isolation Project Plan (WIPP) are excluded from this document.

This document shall be reviewed annually (from the date of issuance/release) and revised as appropriate. The documents and procedures referenced in this QAPP are subject to changes (e.g., revision, document number or title change, cancellation). Such changes are traceable via document control. The revision of this QAPP may not be as frequent as the changes in documents and procedures. Therefore, this QAPP may not reflect the most updated documentation and procedures. However, the most updated documentation and procedures supersede the ones listed in this QAPP document.

Documents issued before December 4, 1996, shall continue to reflect the prior contractor's initials (usually Westinghouse Hanford Company [WHC], Boeing Computer Services, Richland [BCSR], ICF Kaiser Hanford Company [ICF KH], or Kaiser Engineering Hanford [KEH]) in the first field of the document identification number until the next full revision.

All new documents and full revisions of existing documents released on or after December 4, 1996, that currently include contractor initials within the document identification number, shall use Hanford Nuclear Facility acronym "HNF" in place of the contractor initials. For example, a revised document with "WHC-SD-GN-MP-001" as the unique document identification number becomes "HNF-SD-GN-MP-001."

Table 1-1. WSCF Laboratory QA Cross-reference. (4 sheets)

WSCF Laboratory QA Plan HNF-SD-CP-QAPP-017 by Section	HASQARD DOE/RL-96-68	Procedures/Documents
1.0 INTRODUCTION	Volume 1, Section.1.0	HNF-MP-599, Sec. 1; HNF-PRO-261
2.0 ORGANIZATION AND PERSONNEL RESPONSIBILITIES	Volume 1, Section 2.0	ASP-GD-007, -008, -009, -010, and -011.
3.0 PERSONNEL TRAINING AND QUALIFICATION	Volume 1, Section 3.0	HNF-MP-599, Sec. 3 & 5; QAPP-017, Sec. 3.0. ASP-200, Sec. 5.4
4.0 QUALITY ASSURANCE OBJECTIVES 4.1 Data Quality Objectives 4.2 Client Data Quality Requirements	Volume 4, Section 1.0 Volume 4, Section 1.1 Volu me 4, Section 1.2	HNF-MP-599, Sec. 3 & 5; Client's QAPjP, or work authorization document, LOI, or QAPP-017, Sec. 11
5.0 SYSTEMS QUALITY ASSURANCE 5.1 Software Systems 5.2 Administrative Systems 5.3 Physical Facilities Systems	Volume 4, Section 2.0 Volume 1, Section 7.0 Volume 4, Section 2.1 Volume 4, Section 2.2	 HNF-PRO-309; ASP-200, Sec. 1.21 & 1.22; LC-708-001, LC-718-001, ASP-100, -200, and -315

Table 1-1. WSCF Laboratory QA Cross-reference. (4 sheets)

WSCF Laboratory QA Plan HNF-SD-CP-QAPP-017 by Section	HASQARD DOE/RL-96-68	Procedures/Documents
6.0 SAMPLE CUSTODY AND HANDLING	Volume 4, Section 3.0	HNF-MP-599, Sec. 5
6.1 Sample Receipt and Chain of Custody	Volume 4, Section 3.1, and 3.3	LO-090-403
6.2 Samp le Log-in and Tracking Procedure	Volume 4, Section 3.4	LO-090-403; LO-200-400
6.3 Laboratory Internal Chain-of-Custody	Volume 4, Section 3.5	LO-090-400
6.4 Holding Times	Volume 4, Section 3.2	Per RCRA, or method reference
6.5 Sample Disposition	Volume 4, Section 3.6	LO-090-400, LO-090-403
6.6 Sample Collection Devices	*	Refer to specific LA; HNF-5551;
6.7 Waste Disposal	*	ASP-315 Sec. 4.20, 6.1, 6.2, 6.3 & 6.4. LO-100-451, -453, -454, -455-456, -466 and -469, LO-140-417
7.0 CALIBRATION	Volume 4, Section 4.0	Specific Analytical Procedure HNF-MP-599, Sec. 8
7.1 Calibration of Laboratory Measurement Systems	Volume 4, Section 4.4	Specific LA, LO or LQ Procedure
7.2 Specifications of Standards Used In Calibration	Volume 4, Section 4.3	QAPP-017, Sec. 10; HNF-PR0-490 (only f/primary standard weights) As Per Method/Responsible Chemist; HNF-PRO-490 (only f/primary std weights QAPP-017, Sec. 7.6)
7.3 Calibration Records	Volume 4, Section 4.1	
7.4 Calibration of Balances	Volume 4, Section 4.2	
7.5 Calibration of Thermometers	Volume 4, Section 4.2	
7.6 Calibration of Pipettes	Volume 4, Section 4.2	
7.7 Control of Laboratory Measurement Systems		
8.0 LABORATORY PROCEDURES	Volume 1, Section 4.0	HNF-MP-599, Sec.4 & 5; ASP-200 Sec. 1.24; ASP-200, Sec. 1.18, and HNF-PRO-440
8.1 ASP Procedures Group		
8.2 Laboratory Procedures	Volume 1, Sec. 4.1.3	
8.3 Documentation for Analytical Procedure Deviations	Volume 1, Sec. 4.4 & 4.5	
8.4 New Analytical Procedures	Volume 1, Section 4.3	
8.5 Qualification of Analytical Methods	Volume 1, Section 4.6	
8.6 Modification of Required Regulatory Methods	Volume 1, Section 4.7	
8.7 Preparation and Review of Supporting Documents		

Table 1-1. WSCF Laboratory QA Cross-reference. (4 sheets)

WSCF Laboratory QA Plan HNF-SD-CP-QAPP-017 by Section	HASQARD DOE/RL-96-68	Procedures/Documents
9.0 DATA COLLECTION, REDUCTION, AND REPORTING	Volume 4, Section 5.0	HNF-MP-599, Sec. 4 & 5
9.1 Data Collection	Volume 4, Section 5.1	
9.2 Data Reduction	Volume 4, Section 5.2	
9.3 Data Review	Volume 4, Sec. 5.2.3	ASP-315, Sec. 9.3
9.4 Data Reporting	Volume 4, Section 5.3	
10.0 LABORATORY RECORDS	Volume 1, Section 6.0	HNF-MP-599, Sec. 4 HNF-RD-210, HNF-PRO-10588, HNF-PRO-10863, and ASP200, Section 1.04
10.1 Quality Records	Volume 1, Section 6.3	
10.2 Distribution		
10.3 Storage		
10.4 Retrieval		
10.5 Maintenance and Disposal		
11.0 QUALITY CONTROL	Volume 4, Section 6.0	HNF-MP-599, Sec. 5; Customer TSAP, LOI, or Work Authorization Document; QAPP-017, Sec. 11, or specific procedures
11.1 General Laboratory Quality Control	Volume 4, Section 6.1	LO-120-001; LO120-007; LO-150-445; LO-110-400; LO-140-008 & LO-140-410
11.1.6 Control Charts	Volume 4, Sec 7.7	
11.2 Inorganic Analysis	Volume 4, Sec 6.4, 6.5	
11.3 Organic Analysis	Volume 4, Sec 6.6, 6.7	
11.4 Radiochemical Analysis	Volume 4, Sec 6.2, 6.3	LQ-543-401
11.5 Summary of Method Quality Control		
11.6 Instrument Failure		
12.0 PROCEDURES TO ASSESS DATA QUALITY	Volume 4, Section 7.0	Specific LA procedures HNF-MP-599, Sec. 3,4,5
12.1 Precision	Volume 4, Section 7.1	QAPP-017, Sec. 12.1
12.2 Accuracy	Volume 4, Section 7.2	QAPP-017, Sec. 12.2
12.3 Measures of Agreement	Volume 4, Section 7.4	QAPP-017, Sec. 12.3
12.4 Detection Limit considerations	Volume 4, Section 7.5	QAPP-017, Sec. 12.4
12.5 Uncertainty	Volume 4, Section 7.6	QAPP-017, Sec. 12.5

Table 1-1. WSCF Laboratory QA Cross-reference. (4 sheets)

WSCF Laboratory QA Plan HNF-SD-CP-QAPP-017 by Section	HASQARD DOE/RL-96-68	Procedures/Documents
13.0 ASSESSMENTS	Volume 1, Section 10.0	HNF-MP-599, Sec. 9; ASP-200, Sec. 1.2
13.1 Management Assessments	Volume 1, Section 10.1	
13.2 Technical Systems Assessments	Volume 1, Section 10.2	ASP-200, Sec. 1.2
13.3 External Assessments	Volume 1, Section 10.5	HNF-MP-599, Sec. 10
13.4 Data Quality Assessments	Volume 1, Section 10.4	QAPP-017, Sec.13.4
13.5 Performance Evaluation Assessments	Volume 1, Section 10.3	QAPP-017, Sec. 13.5
14.0 PREVENTIVE MAINTENANCE	Volume 1, Section 9.1	HNF-MP-599, Sec.5
15.0 CORRECTIVE ACTION AND QUALITY IMPROVEMENT	Volume 1, Section 5.0	HNF-MP-599, Sec.3; HNF-PRO-052
15.1 Discrepancy & Problem Report	Volume 1, Section 5.1	
15.2 Control of Nonconformances	Volume 1, Section 5.6 and 5.7	HNF-PRO-298
15.3 Trend Analysis	Volume 1, Section 5.4	QAPP-017, Sec. 15.3
15.4 Root Cause Analysis	Volume 1, Section 5.3	HNF-PRO-052
15.5 Continuous Quality Improvement		
16.0 QUALITY ASSURANCE REPORTING	Volume 1, Section 11.0	HNF-MP-599, Sec.9
17.0 PROCUREMENT CONTROLS	Volume 1, Section 8.0	HNF-MP-599 Sec.7; HNF-PRO-123; HNF-PRO-268; HNF-PRO-259; HNF-PRO-129; HNF-PRO-335; HNF-PRO-3144
18.0 REFERENCES	Volume 1, Section 12.0	

Notes:

CM	=	control manual
LA	=	laboratory analytical procedure
LC	=	laboratory computer procedures
LO	=	laboratory operating procedure
LQ	=	laboratory quality control procedure
MOU	=	memorandum of understanding
N/A	=	not applicable
QAPjP	=	Quality Assurance Project Plan
SOW	=	Statement of Work
*	=	not addressed in HASQARD

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2.0 ORGANIZATION AND PERSONNEL RESPONSIBILITIES

2.1 LABORATORY ORGANIZATIONAL STRUCTURE

The WSCF Laboratory is positioned within Analytical Services (AS), Site Services, Fluor Hanford (FH). The organizational/reporting structure for WSCF is shown in Figures 2-1 and 2-2. The charters and responsibilities for each organization are described in ASP-GD-007 to ASP-GD-011. Organization structure is subject to change; any significant changes in the organization or personnel are announced to the WSCF Laboratory through appropriate management.

2.1.1 Management Policy

The policy of FH and WSCF Laboratory management is to direct activities in a manner that ensures the results meet or exceed the customer's expectations and be cost effective in our performance. The achievement of quality will require the total commitment of all laboratory employees to our ethic. WSCF provides key information to our clients who make critical decisions based on this laboratory information. WSCF is totally dependent upon the honesty and integrity of each employee within our organization to provide this information. No acceptable reason exists for any unethical practices within any part of our organization. Our clients for whom we perform services and regulatory agencies can hold the organization liable for unethical behavior.

The quality policy shall be implemented through the following:

- Management provides adequate resources and budget to support effective quality assurance practices that fulfill the customer's program goals and performance objectives. This includes facilities, instruments, support equipment, and materials required to meet the customer's current and projected requirements.

- Personnel are responsible for the quality of their own work and shall check their supplies to ascertain that the items are correct and suitable for use. Laboratory personnel shall have acceptable qualifications and training for their specific job assignments.

- 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, self-assessments and measured actions. Continuous quality improvement will be an ongoing process.

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

2.2 ROLES AND RESPONSIBILITIES

2.2.1 Waste Sampling and Characterization Facility

The WSCF Laboratory organization reports to AS of Site Services, FH. WSCF provides analytical services that include counting room support for stack and room air monitoring and low-level environmental samples, waste characterization including vapor analyses, process control support for liquid effluent treatment systems, permit compliance support for environmental monitoring programs, and analytical support for industrial hygiene monitoring in the workplace. WSCF Laboratory Project consists of Analytical Production, Laboratory Operations and Infrastructure (including Building Operations, Hazardous Material Control, Engineering, Maintenance, Radiological Control, Health and Safety, and Emergency Preparedness), Facility Support, and Environmental Compliance Officer. Project Support, Production Control, Accelerated Projects, and Hanford Analytical Services Program are AS organizations to provide various services.

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.

2.2.1.1 Analytical Production. Analytical Production is responsible for providing analytical support to characterization activities and to routine/non-routine customers. Analytical Production consists of inorganic, organic, radiochemistry, and standards laboratory.

Analytical Production Manager/Team Lead is responsible for overseeing all laboratory analytical activities.

Analytical Production management (including team lead) is also responsible for coordinating analytical workflow through the laboratory. This includes financial planning and reporting, adjusting the manpower to meet changing work needs, selecting personnel, integrating manpower with other organizations, ensuring that the various training requirements are met, implementation of all QC activities and quality improvement processes, and ensuring that reported data meet customer's DQO.

Chemists/Scientists are responsible for writing purchase specifications and setting-up and maintaining operability of analytical instrumentation, for developing the analytical methods and writing the procedures used in analyzing samples, conducting or over-seeing sample analyses, evaluating method performance and QC samples, reporting data, ensuring the quality of that data, controlling, and maintaining associated data records. Chemists/scientists work directly with the analytical leads/client services representatives and at times with laboratory customers, in conjunction with client services representative, to provide technical support in the evaluation of data and the selection of the best analytical method to use in analyzing samples. As necessary, chemists/scientists assist in the training of personnel as mentors.

Chemical Technologists support the Chemist in the area that they have been trained to perform analytical analyses and maintain associated data records.

- Standards Laboratory team procures, prepares, manages chemical, radiological standards, and reagents, when applicable, at the WSCF Laboratory and 222-S Laboratory.

2.2.1.2 Laboratory Operations and Infrastructure. Laboratory Operations and Infrastructure consists of Engineering, Maintenance, and Operations.

- **Engineering** provides engineering services to WSCF. This includes, but is not limited to, configuration control of facility drawings, procedures, work package resolutions, project support, procurement process, cognizant engineering of facility systems, and coordination of design activities with Fluor Federal Services design services.

- **Maintenance** is responsible for maintaining a trained qualified staff that can perform authorized repairs, calibrations and modifications to equipment, structures, systems, and components. Maintenance is also responsible for material control, instrument preventive maintenance when requested/scheduled, and managing the Job Control System.

- **Operations** is composed of two organizations - Building Operations and Hazardous Material Control.

  Building Operations is responsible for conducting safe and compliant operations of the WSCF Laboratory and facilities. This includes inspections and surveillances, maintaining lock and tag activities, administering the work authorization approval system, establishing works priorities, and facility housekeeping.

  Hazardous Materials Control (HMC) provides technical authority for waste disposal operations for WSCF. HMC conducts inspections of Satellite Accumulation Areas. HMC also acts as the technical authority for laboratory procedures controlling waste operations and generation activities. HMC packages waste for shipment including preparation, pick-up, and segregation. HMC provides guidance and assistance to laboratory personnel in regards to waste generation and handling issues.

2.2.1.3 Health and Safety. The Safety Program organization and matrixed safety staff assist management with analysis of performance against safety goals; development of safety and health related procedures and plans; facility and programmatic surveillance activities; investigation of work related injuries, illnesses, and abnormal events; maintenance of injury records; hazard analysis and exposure monitoring, safety and health orientation and/or training, and support to Zero Accident Councils. The safety and health of employees and implementation of the Occupational Safety and Health Program is a shared responsibility between management and employees.

2.2.1.4 Radiological Control. Radiological Control is responsible for implementing and maintaining radiological safety within the WSCF Laboratory. This includes monitoring sample dose rate, radiation and contamination surveys throughout WSCF, establishing and verification of radiological postings, writing, and issuing Radiological Work Permits. Their duties include maintaining the access control entry systems to the laboratory and collect, track and trend air samples in the laboratory. Provide radiological technical guidance to WSCF management.

2.2.1.5 Environmental Compliance Officer. The environmental compliance officer (ECO) is responsible for ensuring WSCF complies with applicable local, state, and federal environmental regulations. The ECO prepares and/or reviews applicable environmental permits for the facility and provides facility input to site-wide environmental reports. The ECO also oversees handling and management of hazardous and radioactive waste.

2.2.2 Other Organizations

Business and Project Controls provides matrixed services including baseline development and control, work scope prioritization, multi-year planning, site upgrade planning/implementation

The following other AS organizations also provide support to WSCF:

2.2.2.1 Project Support. Project Support coordinates laboratory wide initiatives such as staffing, general resource application, management systems, and improvement projects. Project Support manages and coordinates the support provided from other FH projects such as Waste Management, Plutonium Finishing Plant, CH2M HILL, Waste Vitrification Plant, and other interface areas. Project Support also participates in streamlining operations and organizational structure/functions, strategic planning, as well as laboratory and clients integration. Project Support includes two organizations - Facility Support Program and Training Program.

- **Facility Support Program** provides procedure administration support, including processing, tracking, routing, and maintaining laboratory analytical (LA), laboratory computer (LC), laboratory quality (LQ), laboratory reference material (LR), laboratory technical (LT), administrative and maintenance procedures.

  Facility Support is also the point-of-contact for processes relating to corrective action management, management/general assessments, and required reading.

- **Training Program** is responsible for providing training support for WSCF and for coordinating training for the chemical technicians, chemists/scientists, and management. Project Support Training also supplies a graded/systematic approach to training that enhances development of training programs to the required rigor necessary. This includes developing/delivering initial and continuing training, applying technology as appropriate to maximize outcomes for learners and providing training scheduling and records management. This training organization is also responsible conducting facility specific orientation, dangerous waste plan training, and maintaining training documents and records.

2.2.2.2 Production Control. Production Control includes Analytical Project Management, Client Services, Laboratory Information Management System (LIMS), and Integrated Scheduling. Analytical Project Management group is responsible for project specific quality control, sample receiving and handling, chain-of-custody, preparing final data packages, and coordinating sample disposition with operations or clients for sample return. Client Services manages client interfaces and interactions and coordinates client agreements, SOW, LOI, SAP, etc. LIMS manages current laboratory data management system, LIMS administration, identification and control of hardware and software as related to laboratory instrumentation. LIMS group is also responsible for implementing new LIMS, compliant records management program establishment and maintenance, and integrated analytical data package/reporting

process systems. Integrated Scheduling is responsible for near-term and long-term schedules across laboratories.

2.2.2.3 Accelerated Projects. Accelerated Projects organization provides technical activity expertise related to process development and analysis at Hanford and support to AS functions. The group is accountable for the effective utilization of technical resources for the analytical analysis of client samples. The primary focus is to provide and optimize analytical resources to meet unique client needs directed at the acceleration of Hanford Site cleanup missions including waste and environmental sample analysis, process control support, field and sampling services, and expertise in chemistry and data quality. The Accelerated Projects also provides management systems and administrative support to the 222-S Analytical Chemistry, WSCF Analytical Chemistry, and Technology Project Management organizations.

2.2.2.4 Hanford Analytical Services Program (HASP) performs long-term planning including laboratory capabilities and instrument capital; advises (e.g., regulatory requirements) and technical support; and program management for capital equipment upgrades, facility upgrades, and equipment installation. Provides quality oversight of laboratory data through data reviews, review of documentation (analytical methods, procedures, and QA policies), manage laboratory performance evaluation programs, internal assessments (e.g., technical systems assessments), participation in Deficiency Evaluation process and corrective action management.

Quality Assurance from the Plateau Projects Quality Assurance organization provides facility/project QA support (Engineering QA). The Engineering QA provides QA review for procurement, engineering document and work control.

The QA Officer is matrixed to HASP, reports to the Environmental/Science Assurance organization, and is responsible for the following: developing, implementing, and overseeing the QA Program in WSCF; coordinating quality improvement process; maintaining QA Plan documentation; managing accreditation programs [e.g., State of Washington Department of Ecology (Ecology)]; reviewing documentation and data reports or coordinating review of data reports; oversight/monitoring method performance and quality control; performing assessments and recommending corrective actions; coordinating external audits between laboratory personnel and external agencies; and coordinating/managing performance evaluation programs.

2.2.3 Sample Suppliers

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

2.3 AUTHORITIES

All laboratory personnel have the authority to initiate a stop-work order where safety, serious quality, or health conditions exist. All staff have the authority to initiate actions which:

- Prevent reporting results from a measurement system that is out-of- control
- Identify any laboratory method or procedure that poses quality problems
- Provide solutions through designated channels, and monitor effectiveness of corrective actions
- Stop activities posing serious threat to health, safety or quality.

Figure 2-1

ORGANIZATIONAL STRUCTURE

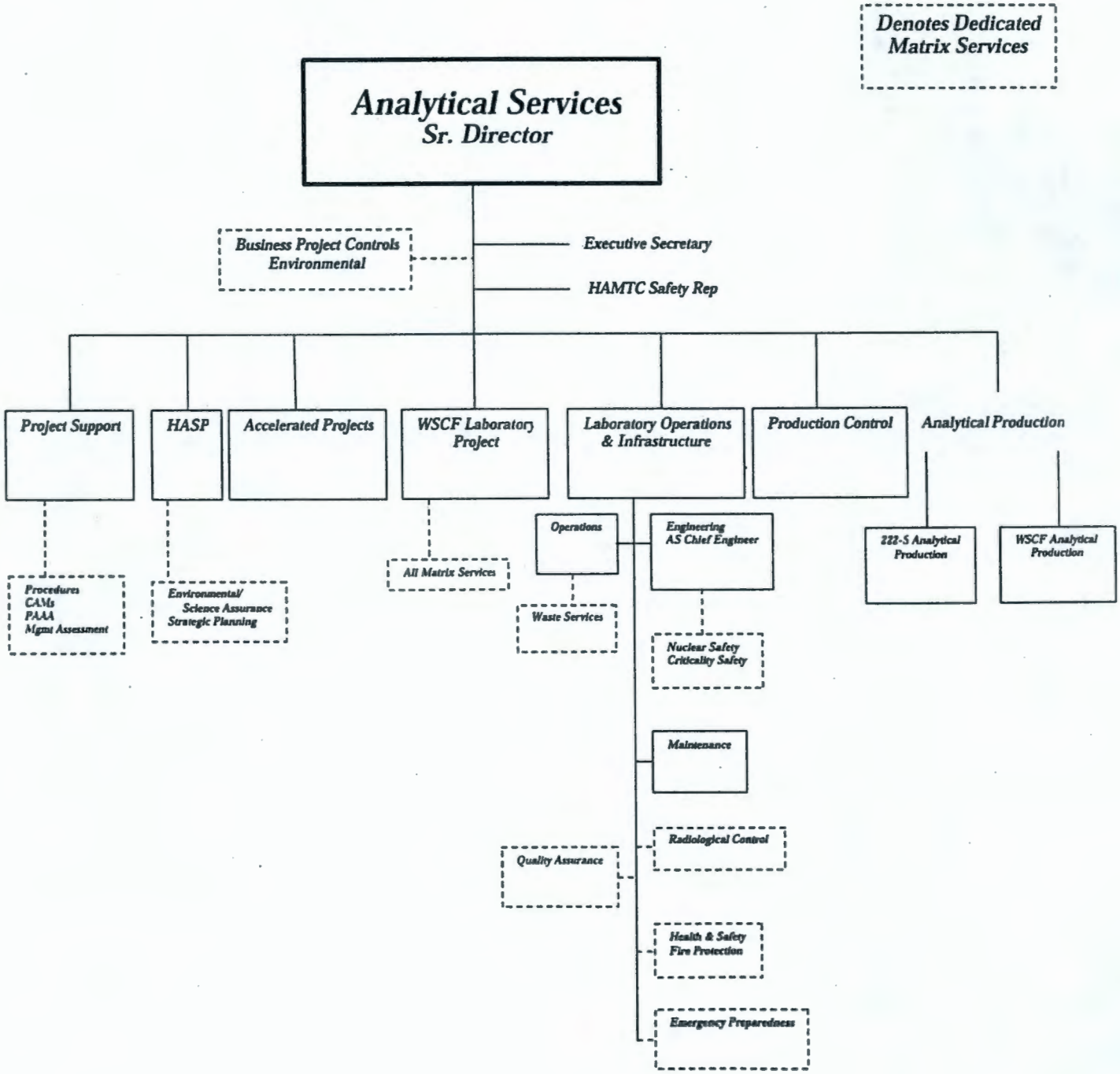
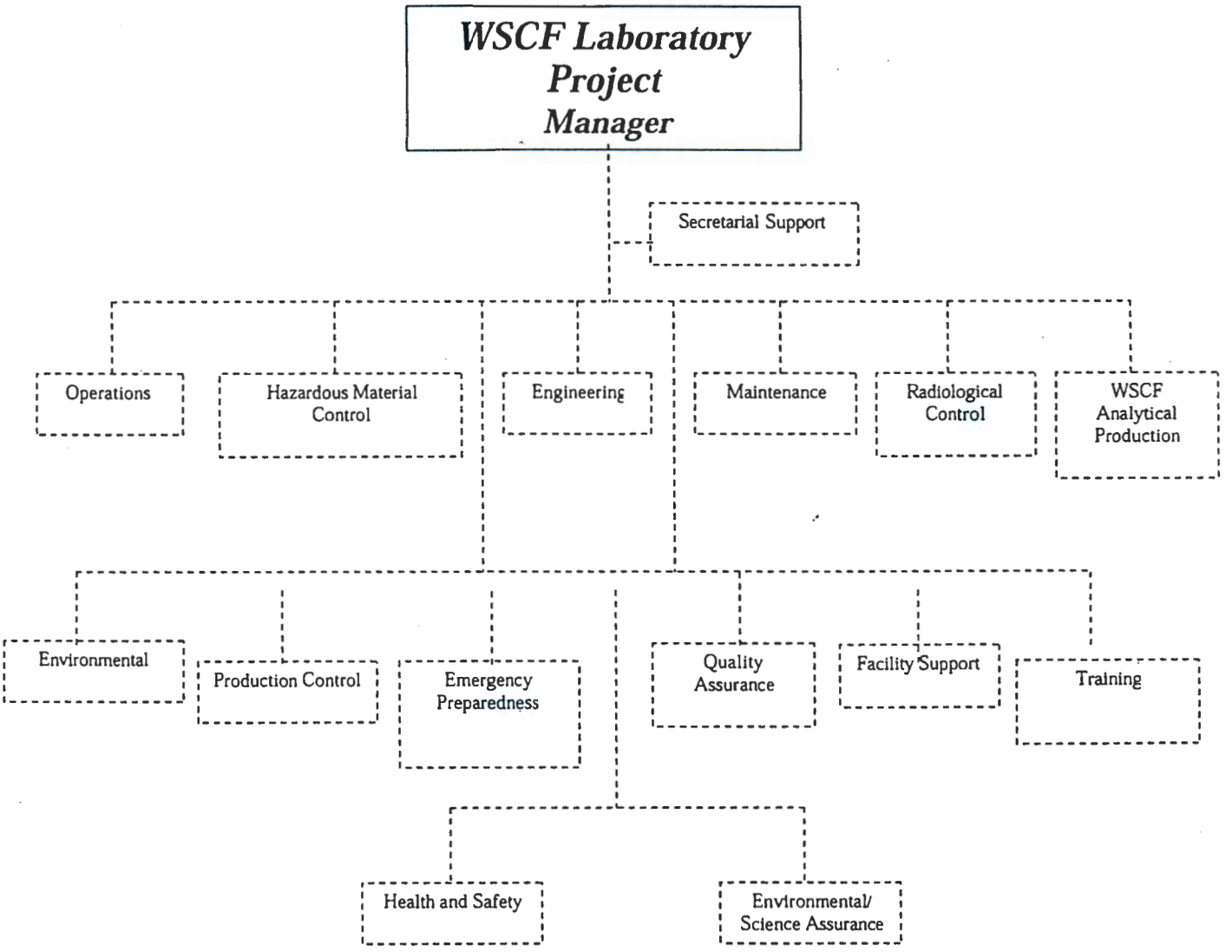


Figure 2-2

ORGANIZATIONAL STRUCTURE



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3.0 PERSONNEL TRAINING AND QUALIFICATION

The qualification and training program includes initial and continuing training and qualification process (e.g., prerequisite qualification and training for qualification).

Employee selection for a job assignment is based upon requirements of the position and the candidate's prerequisite education, training, and previous experience/qualification. Qualification program/process is established based on job requirements and associated tasks and skills. Training plan and qualification process are used to identify general and job specific training courses and/or procedures to meet applicable technical, quality, and project management requirements.

The following sections describe the processes for identifying, designing, performing, and documenting requirements that are related to, but not limited to, technical and quality training.

3.1 GENERAL TRAINING

General training is established for personnel performing various laboratory services to develop and/or enhance their knowledge, skills, and ability to perform in their respective job assignments. General training is divided as follows:

Company-Wide and Facility Classes deal with security and safety topics and are under the control of the groups assigned to monitor and maintain each program. Personnel at WSCF are trained in facility orientation, hazard communication, building emergency procedures, and if required, self-monitoring.

Hanford General Employee Training (HGET) and facility specific courses require retraining at various intervals. These intervals are documented and can be accessed via the Training Information and Record System.

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

Employees who are involved with analytical activities shall receive orientation to the mission and laboratory quality assurance plan. Quality assurance requirements of this document are part of exempt employees required reading and are incorporated into mandatory procedures for bargaining unit employees.

3.2 PROCEDURE TRAINING

Chemical technologists are qualified to perform analyses as they meet the specifications of required training and qualification. Training is tailored to the work and is determined by the manager based on the assignment, education, and experience of the technologist. Qualification is documented per on-the-job training (OJT) outlined in ASP-200, Section 5.4 and the individual's qualification card documentation. As part of the initial training of a method by a qualified

chemist, the trainee should demonstrate acceptable results by analyzing applicable quality control or performance evaluation samples.

Chemists/scientists procedural training includes operational specific procedures for working in the laboratory, applicable QA procedures, and procedure author training. Chemists/scientists may be assigned to different methods based on laboratory workload. Laboratory managers are responsible for selecting appropriate chemists based on individual's experience to perform analytical methods.

Initial qualification for the methods is required for both chemical technologists and chemists/scientists. Initial qualification is defined as "to develop general processing knowledge for the method." Initial qualifications for the method may include, but is not limited to establishing calibration, achieving acceptable standard recovery, completing a Method Detection Limit (MDL) study, or analyzing Performance Evaluation (PE) samples that can demonstrate chemists have general processing knowledge on the method. However, initial qualification on method is not required to be matrix- or analyte-specific. The extent of initial qualification is dependent on chemist/scientist experience and is determined by the manager. Initial qualification for the chemist/scientist is documented on certificate of qualification. The employee's manager holds the certificate of qualification.

3.2.1 Maintaining Position Proficiency

Personnel should work in their positions on a regular basis to maintain proficiency. Unless more specific requirements are identified, an analyst maintains proficiency if they continue to demonstrate acceptable performance on QC samples.

An individual will not be assigned to a position for which they are not proficient. In the event of an extended absence, selected retraining may be required to regain proficiency. The extent of retraining will depend on the duration of the absence or time away from the specific duty area (that is, organic, inorganic, or radiochemistry). General guidelines are as follows:

- Less than one year - No retraining is required.
- Greater than one year - The individual's immediate manager conducts an interview to investigate areas of potential weakness and then decides whether retraining is necessary.
- If there is a problem indicating an inadequate understanding of the analytical methods, the individual's immediate manager shall initiate re-training to maintain proficiency.

3.3 CONTINUING TRAINING

Continuing training may consist of a combination of classroom-type and on-the-job training as it applies to the position. These retraining requirements are typically either annual or biannual. Retraining requirements are called out in the individual's annual training plan, by routine re-qualification tickler from the training department, or by specific training identified

through the continuous improvement program. Continuing training programs are structured with specific position needs and designed to enhance personnel proficiency. Continuing training can also be accomplished through formal classroom presentation, on-the-job training, safety meetings, pre-job briefings, training bulletins, required reading, vendor training, or professional conferences.

3.4 PERSONNEL QUALIFICATION

Education, experience, and training are the components of personnel qualification. Qualification specifications are established based on job function and responsibilities, associated tasks, and skills. Such specifications are used to develop individual training plans and/or qualification requirements, which identify course requirements and other types of technical, quality, and/or applicable project management training needs.

Additional analytical instrument operator qualification requirements are listed below according to the Washington State Procedural Manual for Environmental Laboratory Accreditation Program [Washington Administrative Code (WAC) 173-50]. The core chemistry classes for a minor in chemistry degree are considered "equivalent." A bachelor's degree in chemistry or a related field may not be necessary for instrument operators if the immediate supervisor has a bachelor's degree in chemistry or a related field or if the analyst has the number or credit hours in chemistry courses required for a major in chemistry.

Inductively Coupled Plasma (ICP) Spectroscopy

a. Education

Minimum of Bachelor's degree in chemistry or equivalent
Specialized training course in ICP Spectroscopy

b. Experience:

Minimum of six months of applied experience with ICP analysis of environmental samples.

Atomic Absorption (AA) Spectroscopy

a. Education:

Minimum of Bachelor's degree in chemistry or equivalent
Specialized training course in AA instrumentation

b. Experience:

Minimum of six months of applied experience with AA instrumentation for graphite furnace, flame and cold vapor AA.

Inorganic Sample Preparation

a. Education:

Minimum of high school diploma or equivalent

b. Experience

No minimum experience required. After being trained in a method, the trainee should demonstrate acceptable results by analyzing applicable quality control or performance evaluation results.

Classical Laboratory Analysis

a. Education:

Minimum of high school diploma or equivalent

b. Experience:

No minimum experience required. After being trained in a method, the trainee should demonstrate acceptable results by analyzing applicable quality control or performance evaluation results.

Gas Chromatography/Mass Spectroscopy (GC/MS)

a. Education

Minimum of a Bachelor's degree in chemistry or equivalent
Specialized training course in mass spectroscopy

b. Experience

Minimum of six months of applied experience with GC/MS/Data System (DS).

Gas Chromatography (GC)

a. Education

Minimum of a Bachelor's degree in chemistry or equivalent
Specialized training course in GC

b. Experience

Minimum of six months of applied experience in GC.

Organic Sample Preparation

a. Education:

Minimum of high school diploma or equivalent

b. Experience

No minimum experience required. After being trained in a method, the trainee should demonstrate acceptable results by analyzing applicable quality control or performance evaluation results.

Ion Chromatography (IC)

a. Education

Minimum of a Bachelor's degree in chemistry or equivalent
Specialized training course in IC

b. Experience

Minimum of six months applied experience in the operation of IC.

Radiochemistry Sample Preparation/Digestion/Analysis

a. Education

Minimum of high school diploma or equivalent

b. Experience

No minimum experience required. After being trained in a method, the trainee should demonstrate acceptable results by analyzing applicable quality control or performance evaluation results.

Counting Instruments

a. Education

Minimum of a high school diploma or equivalent

b. Experience

No minimum experience required. After being trained in a method, the trainee should demonstrate acceptable results by analyzing applicable quality control or performance evaluation results.

Nuclear Spectroscopy

a. Education

Minimum of a Bachelor's degree in chemistry or equivalent

Specialized training course in Nuclear Spectroscopy

b. Experience

Minimum of six months applied experience in a counting laboratory.

Sample Custodian

a. Education

Minimum of a high school diploma or equivalent

b. Experience

No minimum experience required.

Analyst/Chemical technologist shall have a minimum of a high school diploma or general equivalency diploma (GED) and have completed a training course (or equivalent in-house course) in relevant analyses and have demonstrated ability to produce reliable results through accurate analyses of reference materials, performance evaluation samples, or quality control samples.

3.5 TRAINING RECORDS

Individual training is documented in according to HNF-PRO-249 Training Course Administration, Registration and Records (e.g., completion of individual personnel qualification training, classroom training). Specific job positions have qualification cards listing specific procedures, manuals, or other required reading needed for their jobs.

3.5.1 Integrated Training Electronic Matrix (ITEM) Data Base

Course requirements for personnel are electronically posted and tracked using Integrated Training Electronic Matrix (ITEM). Laboratory managers use the ITEM system as a tool to identify and track the training that employees need. ITEM also serves as the training plan for individuals and positions within each organization. ITEM contains training matrices for positions within AS, which have a significant impact on facility operations and safety.

3.5.2 Field Files

An employee's field file shall contain the following when applicable:

- Records of course content, Certificates of Completion, or attendance sheets
- Records of completion of on-the-job training assignments (non-exempt employees)
- Certificate of Qualification

- Summary of experience (e.g., resume).

3.5.3 Qualification Card Records

Qualification cards document that an individual has completed all essential job specific training for a position. Qualification card records typically identify any position specific courses, required reading, OJT, and task qualification. Qualification cards should be requested by the employee's manager from the applicable AS Training scheduler. When completed, the Qualification Card record serves as a verification of competency to perform activities associated with a specific job position.

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4.0 QUALITY ASSURANCE OBJECTIVES

4.1 DATA QUALITY OBJECTIVES

QA objectives provide a set of recognized parameters to ensure acceptable performance of an analytical measurement system and to qualify analytical data. Establishment of data quality criteria is based on the following applicable factors:

- Regulatory requirements, for example, *Resource Conservation and Recovery Act* (RCRA), *Comprehensive Environmental Response, Compensation and Liability Act* (CERCLA), or *Clean Water Act (CWA)*

- HASQARD

- Data Usage. Typical data uses include regulatory, process control, screening, planning, and development.

The policy of WSCF is to have a written agreement on client project quality requirements before analytical work is initiated. This written agreement can be in the form of a Waste Analysis Plan (WAP), SAP, QAPjP, SOW, LOI or other similar guidance documentation. Documented client specific requirements supersede this QAPP and HASQARD.

When a formalized DQO process is used to construct one of the above listed plans, WSCF either participates in or provides recommendations for establishing data quality criteria to meet the needs of the project. The written agreement is prepared by WSCF or by the customer with WSCF involvement and approved by WSCF management and the customer. The agreement can be changed or modified with review and approvals by the issuing and original reviewing organizations.

Small projects, containing few samples, without the above listed plans will be conditionally accepted by WSCF. In these cases, WSCF will obtain a written agreement from the client stating that this QAPP is adequate to fulfill the customer's data quality requirements. QAPP-017 and/or any of the above listed guidance documents shall provide the following information where applicable:

- Applicable regulatory requirements

- Process knowledge, sample source, and sample conditions (such as radioactivity) known to the client that could impact the laboratory worker's safety

- Sample handling relative to numbers of samples or sample matrix, estimated number and matrix of samples

- Analysis methods and analyte lists for sample analysis - the analyte list should also specify whether or not tentatively identified compounds are required (when applicable)

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

4.1.1 A System for Notification of Unique Data Quality Requirements

Unique data quality requirements shall be communicated to the responsible staff from client service representative to Analytical Production group and if necessary by providing copies to the responsible staff of the client's written agreement.

4.1.2 Client Complaints and Resolution

Resolution for client complaints shall be coordinated through the Client Services representative working with laboratory management. A graded approach is used to document complaints. Complaints that relate to data quality shall be documented per ASP-315, Section 8.2. Documentation will include a description of the issue, the date of complaint, and a description of the resolution. Client complaint on failures to meet client's data quality requirements will also be documented in accordance to Section 15.1.

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 analytical technique.

The precision and accuracy requirements shall be agreed upon by the laboratory and the client and should be based on the error tolerances of the sampling and analytical effort. The

laboratory will provide to the client laboratory-established precision and accuracy values for each method. If the client has special requirements for precision and accuracy, they must be identified in a written agreement. If the client has no special requirements and no corresponding written agreement, then the laboratory default QAPP will specify precision and accuracy determined by the analytical method. Comparability, completeness, and representativeness are parameters normally evaluated by the data validator or the data user. They will not be addressed by the laboratory but are defined below.

4.2.1 Precision

Precision represents a measure of the degree of reproducibility of measurements under prescribed similar conditions. 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 12.0.

Precision of analytical methods is estimated using the laboratory control samples (LCSs) over time. Sample precision is calculated on the basis of laboratory duplicate analyses. Acceptance criteria shall be established for each analyte method and shall be agreed upon by the laboratory and the client.

4.2.2 Accuracy

Accuracy represents the degree to which a measurement agrees with an accepted reference or true value. Accuracy is calculated based on the equations listed in Section 12.0, "Procedures to Assess Data Quality".

Sample accuracy is expressed as the percent recovery of a spiked sample. Spiking may not be applicable for analytes present in the samples in relatively high concentration (> 0.1%). In these cases, other LCSs can be used to estimate the accuracy of the method. Acceptance criteria shall be established for each analyte method and shall be agreed upon by the laboratory and the client.

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 are to be compared between multiple laboratories. 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 and/or valid data obtained from a measurement system compared to the total amount of data requested. Completeness can be used

to evaluate the amount of data produced that meets the client's requirements (e.g., accuracy, precision). A clear definition of completeness should be agreed on between the laboratory and the client.

4.2.5 Representativeness

Representativeness is the degree to which data accurately and precisely represents 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 heavily on sampling and is outside the control of the laboratory. Analytical data should not be taken as the sole indicator of representativeness of the sample process condition. The methods should be assessed after accumulation of sufficient data to represent the same population.

Representativeness should be maintained in the laboratory by proper homogenization or appropriate sub-sampling. If different phases are apparently visible in the sample, the laboratory should consult with the client to determine sub-sampling and homogenization needs.

If sub-sampling is needed, the data quality requirements should be re-evaluated to determine if sub-sampling affects the ability to meet representativeness requirements and if a different methodology is required for preparation and analysis.

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5.0 SYSTEMS QUALITY ASSURANCE

5.1 SOFTWARE SYSTEMS

The laboratory software systems consist of administrative and technical software. Administrative software systems are used to manage the workflow 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, accumulate, and reduce data. Examples of technical software systems are those that provide instrument interface, calculations, calibration control, or 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 from unauthorized or inadvertent changes.

Instrument controlling and data reduction software supplied by commercial sellers are exempt from verification and validation testing provided they have not been modified by any member of the AS; and are an integral part of the measuring instrument test equipment. Successful analysis of a standard [in-house, National Institute of Standards and Technology (NIST) traceable, PE] is considered adequate.

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 validate the change. Configuration control and acceptance test data shall be maintained for commercial software packages.

The following documents govern laboratory software system configuration control:

- HNF-PRO-309, *Computer Software Quality Assurance Requirements*
- ASP 200, 1.21 *Laboratory Configuration Control*
- ASP 200, 1.22 *Documentation of Laboratory Quality-Affecting Software*

5.1.2 Acceptance Testing

Software systems shall be tested for acceptance when installed, after changes, and periodically during their use (ASP 200, Section 1.21). 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, checking against another software system that has been previously tested, comparing output with previous output, or by analyzing standards.

LC-718-001, *Laboratory Automated Data Processing Systems (ADP) Operations,* is used for laboratory-developed programs/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.

Documentation of the testing shall include printouts 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

Quality affecting software will be backed up in accordance with the applicable procedures developed for the use of the software. The backup of data will occur at a frequency determined by the laboratory supervisor depending upon the impact of the loss of the data/software on the organization.

LC-718-001 provides authority to use work sheets for routine operations (that is, 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.

5.1.4 User's Manuals

User instructions for MULTI LIMS are provided in LC-708-001, *MULTI LIMS Use in the Laboratory.*

LC procedures are issued when appropriate for an application. For small or straightforward programs or applications that are outside the scope of the LABCORE system, a controlled manual or special training is not required.

5.1.5 Error Reporting

Software errors found during use shall be reported to the appropriate laboratory management in a reasonable time. When there is a significant impact on the data results or data quality, laboratory personnel shall be assigned to verify errors and document the error notification and corrective actions according to Section 15.1. Error handling shall include users so that previously reported data may be evaluated and corrective actions may be tracked.

5.2 ADMINISTRATIVE SYSTEMS

The PHMC has established an administrative control system based on a hierarchy of controlled procedures/documents (e.g., HNF-PRO procedures or ASP-100, -200 or -315 procedures). These procedures provide documented interpretation of DOE orders and procedures for implementation. In addition to the administrative directions listed in manuals, laboratory procedures are used for specific activities.

5.3 PHYSICAL FACILITIES SYSTEMS

The WSCF complex is located at the 600 Area. 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, Sample Equipment Cleaning Facility, and the Environmental Data Remedial Tracking System Facility. The WSCF Building 6266 consists of administrative area, Nuclear Spectroscopy Laboratory (NSL) (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 deionized (DI) water, piped gases, compressed air, and process vacuum. The electrical (120, 208, or 440, and so forth, 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 standby backup power service for selected ventilation and lighting. Critical computer systems are protected by uninterruptible power supply.

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 within ±3 °F. The NSL has an independent heating, ventilation, and air conditioning (HVAC) system 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.

WSCF maintains essential facility services such as electrical and ventilation through the Job Control System (JCS) as defined in ASP-200, Section 3.1. The schedules for this maintenance are defined by the cognizant engineer responsible for the system and are based on the essential equipment list.

5.3.2 Facility Inspection

The facility manager and/or other managers inspect laboratory facility conditions periodically and document deficiencies for corrective action.

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6.0 SAMPLE CUSTODY AND HANDLING

6.1 SAMPLE RECEIPT AND CHAIN-OF-CUSTODY

The WSCF Laboratory is a secure facility, restricted to authorized personnel only. During the day, visitors need to sign in and out at entry to the laboratory building. Entrance/exit doors to the actual laboratory are locked at all times and require an access code for entry. Visitors are escorted in the laboratory building. Exit/entrance doors to the laboratory building are locked during the night.

Chain-of-custody (COC) begins with sample collection (in some cases preparation of sample bottles before sampling) and is maintained through the time of sampling until destruction by analysis, disposal, or return of unused sample to the client. COC is the responsibility of the client until custody is transferred at the laboratory receiving area.

LO-090-403, *Sample Receiving and Custodianship at the Waste Sampling and Characterization Facility (WSCF)*, describes sample receipt, custody, and handling.

When sample receipt is completed, samples are then logged in for analysis. Upon acceptance of samples, the Sample Custodian shall sign the COC. Internal custody is now the responsibility of the laboratory.

6.2 SAMPLE LOG-IN AND TRACKING PROCEDURE

Laboratory custody begins when samples are received by the custodian and is maintained until sample disposition is completed. The Sample Custodian(s) is responsible for maintaining custody of the samples during the login 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 login and tracking procedures:

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

- LABCORE is used to assign sample numbers. Each sample is given a unique identifier regardless of its re-sample status. Every sample, sample replicate, and sub-sample shall be labeled in a manner that 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 COC form and LABCORE.

- A declaration of radioactivity level is to accompany the samples that are known to be radioactive or a screen for radioactivity level before samples are distributed for analyses shall be performed. LO-200-400, *Radioactive Material Identification and*

Classification Program at WSCF, documents the requirements and protocol for radiological surveys, classification, labeling, and administrative radiological protective measures for handling and analysis of radioactive material at WSCF.

- The LABCORE system is used for tracking sample status. Sample dates as well as the date of receipt are logged into the system. Due dates are normally assigned based on the client's requested turnaround time or the regulatory holding time, whichever is less. It is the responsibility of the chemist to be aware of and to meet the regulatory holding times for the samples and the client's turnaround time requirements.

- Sample receipt anomalies will be documented, segregated nonconformance samples from others, and resolved in one of the following ways:

- The anomalies are recorded on the COC, when necessary, anomalies are entered into the comments section of LIMS during log-in along with the client notification and client response, or

- When applicable, the group that functions as sample management is notified of the anomalies by e-mail and transfers the issue to a Record of Disposition, which documents the issue and client direction. In both cases, the anomalies and the client response or direction are documented.

6.3 LABORATORY INTERNAL CHAIN-OF-CUSTODY

Once a sample is received at the facility, the laboratory is responsible for internal custody. Internal custody refers to maintaining custody as the sample is dispersed to various groups within the laboratory for analysis in the secured facility. Internal custody is maintained until the sample is consumed, returned, or disposed (LO-090-400). Only authorized laboratory personnel have access to samples. Transferring of samples is documented on the sample log sheet. In addition, the following procedures are required for environmental samples.

- Sample preparation date (if applicable) and sample analysis date are recorded in LABCORE. In addition, personnel that handle sample preparation or analysis are also recorded in the appropriate documentation (e.g., in the notebook, sample preparation sheets, or analytical printout).

- The samples requiring refrigeration, when not in analysis, shall be stored in the sample storage area (e.g., room N4, N2). Sample storage refrigerators shall be monitored utilizing LO-150-445, *Monitoring Temperature of Refrigerated Storage Units at the Waste Sampling and Characterization Facility*. All air-filter samples are stored in the NSL.

- Upon request, LABCORE can generate sample tracking history including sample identification (ID), sample receiving date, date of preparation and/or analysis and analyst initials. A bar code system is used to track the location of samples and the

person in control of the samples and is traceable in LABCORE from the time samples are received at the laboratory (log-in) until the analysis is completed.

6.4 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 onset of preparation and/or analysis.

All holding times should be routinely based on method specifications or in special circumstances, as dictated by the client written agreement. If the laboratory is unable to meet prescribed holding times, due to sample activity and such, the client will be notified in writing as soon as possible. Client direction must also be documented in the same manner. It is the responsibility of the client to ensure the timely delivery of samples to enable WSCF to meet holding time requirements.

The sample custodian is responsible for notifying the laboratory personnel (responsible manager and chemists/analysts) upon receiving samples. After sample receipt, laboratory supervisors are responsible to meet holding time requirements.

6.5 SAMPLE DISPOSITION

The sample disposition includes either disposing of or returning original samples to the client. Sample disposal for WSCF is the responsibility of the Waste Handling group under Building Operations. Requirements for sample disposition include the following: a written request (e.g., statement of work or electronic mail) from the client, verification of sample container against client's COC, relinquishing between sample custodian and client or waste handler, and management approval. Sample results or generator's knowledge are evaluated to identify appropriate waste code or to meet transportation regulation before returning or to dispose the un-used portions of samples.

There are various laboratory procedures to meet the following requirements:

- Disposing of or returning the sample to the client
- Records maintenance that identifies the date of disposition
- Meeting all local, state, and federal regulations
- Documenting the status of sample on the COC record - custody records shall document that the sample was returned to the client, consumed in the analyses, or disposed of
- If appropriate, shipping documentation shall be maintained with the sample COC by the sample custodian and shall meet Department of Transportation and applicable carrier requirements for transportation.

6.6 SAMPLE COLLECTION DEVICES

6.6.1 SUMMA™ Passivated Canister Collection Device

SUMMA[1] passivated canisters are stainless steel containers with electro polished interior surfaces. SUMMA™ canisters are cleaned and leak tested using standardized laboratory procedures. Canister cleaning is considered acceptable if any of the Vapor Program target organic analytes do not exceed one half of the Vapor Program Required Quantitation Limits (VPRQL) or as directed by the relevant SAP.

Vapor samples collected in SUMMA™ canisters are analyzed primarily for permanent gases (carbon dioxide, carbon monoxide, hydrogen, methane, and nitrous oxide) and volatile organic compounds (VOC). Refer to the specific LA procedure for aspects of calibration/method performance and/or the customer's SAP.

6.6.2 Sorbent Trap Collection Device

Sorbent trap devices such as Triple Sorbent Traps (TST) are small-bore glass or metal tubes filled with different types of sorbent materials. Carbosieve, Carbotrap and Carbotrap C are typically used. As vapor samples are drawn through the TST, sorbent materials adsorb organic compounds for laboratory analysis later on. In the past, TST have been purchased commercially (glass tubes) or manufactured (stainless steel tubes) by the performing laboratory.

Vapor samples collected with TST are analyzed primarily for volatile compounds, although some semi-volatile compounds can be collected on TST as well. Refer to the specific LA procedure and/or the customer's SAP information on target lists, calibration, and method performance.

6.6.3 Sorbent Tube Train (STT) Collection Device

Standard, off-the-shelf sorbent traps are prepared singly and in series (as sample trains), and provided to the sampling teams for the collection and measurement of inorganic gases/vapors.

Vapor samples collected with STTs are analyzed primarily for ammonia, nitrogen dioxide, nitric oxide and water. Refer to the specific LA procedure for aspects of calibration/method performance and/or the customer's Tank Sample Analysis Plan (TSAP).

6.7 WASTE DISPOSAL

Hazardous waste generated by analytical processes must be managed in a controlled, safe, efficient manner. The WAC that mandates the management of hazardous/dangerous waste generated by WSCF is WAC 173-303. All WSCF personnel are responsible for ensuring that waste generated within their assigned area is managed in accordance with WSCF waste handling

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[1] SUMMA is a trademark of Molectrics, Cleveland, OH.

procedures. All laboratory employees are trained to HNF-5551, *WSCF Laboratory Complex Dangerous Waste Training Plan* and the *WSCF Chemical Hygiene Plan*, ASP-315, Section 4.20.

Waste generated during analytical operations at WSCF is pre-designated to allow routine disposal. Pre-designation is based on knowledge of the types and amounts of chemicals used during sample analysis; and are documented on Waste Fact Sheets (WFSs). If the pre-designation is not used, an inventory is required during collection of the waste, which must be designated prior to being disposed. Final designation is determined using the WFSs, laboratory analyses, listed waste codes carried by samples, and other available information. Refer to ASP-315, Section 6.3, *Waste Fact Sheet Development and Issuance at WSCF (Pre-designation of Waste)*. Requirements for segregation of laboratory hood waste are documented in LO-100-451, *Waste Management in Analytical Process Areas.*

Requirements for managing satellite accumulation areas and 90-day accumulation areas at WSCF are documented in ASP-315, Section 6.1, *Management of Satellite Accumulation Areas-WSCF;* ASP-315, Section 6.2, *Management of 90-Day Accumulation Areas;* and LO-100-466, Manage *Satellite Accumulation Areas and 90-day Accumulation* Areas and other procedures listed in Section 1, Table 1-1.

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7.0 CALIBRATION

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

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

The initial and continuing verification of laboratory measurement system calibration is described in Section 11.0, *Quality Control.*

7.1 CALIBRATION OF LABORATORY MEASUREMENT SYSTEMS

The requirements for calibration of each Laboratory Measurement System (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, when applicable, to be used in the calibration
- Frequency of calibration
- Criteria used to accept calibration
- Actions to be taken if calibration fails acceptance criteria.

General instructions for use in establishing instrument calibration are given in the various LA or LQ procedures. Calibration requirements in laboratory procedures used for regulatory-driven analyses must conform to calibration requirements specified in the appropriate regulatory methods. Specifications listed in the regulatory/consensus method when requested by the client take precedence over HASQARD requirements. If no calibration specifications listed in the regulatory or non-regulatory method is used for analysis, HASQARD calibration requirements (Table 4.1 - 4.9, Volume 4) for specific instruments are followed. Any variances from the QC requirements included in laboratory procedures must be based on an agreement between the laboratory and the client (e.g., via letter of instruction, statement of work).

Accuracy of initial calibration shall be established based on traceable reference standards[2]. If traceable reference standards are not available, appropriate physical and chemical means are used to determine quality (e.g., neat standard). A level of independence shall exist between the materials used for LMS calibration and those used for initial calibration verification (ICV) when such materials are available or feasible.

Accuracy of the calibration shall be confirmed by performing initial calibration verification. When an independent source is not available or standards consists of multiple analytes, or multiple runs are required in the initial calibration (e.g., vapor program), it is

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

acceptable to use an alternate lot of the same material, or use a sub-group of standards that represent or cover the extend spectrum of analytes in the initial calibration analytes. The performance of an instrument measurement system during an analytical run shall be verified by continuing calibration verification. When calibration specifications are not met, corrective actions include investigation and/or re-calibration when necessary. If re-calibration could not re-establish instrument acceptable performance, the instrument shall be posted to prevent further analysis until the problem is corrected.

7.2 SPECIFICATIONS OF STANDARDS USED IN CALIBRATION

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

The Standards Laboratory maintains certificates of procured reference materials, at a minimum:

- Source vendor and material identification
- Lot number
- Purity
- Date of preparation and/or expiration
- 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
- 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. In some instances, the Standards Laboratory will purchase standards, but the chemists will actually prepare the working standards for calibrations. In this case, the Standards

Laboratory will still maintain the reference standards' certifications. The chemist is responsible for maintaining the traceability to the certified standard when preparing working standards.

Calibration standards that have exceeded their expiration date or shelf life shall no longer be used for LMS calibration and clearly marked as unusable for calibration purpose unless were approved and re-certified for other usage. Recertified expired standards may be used as second source check standards as needed.

In radiochemistry, expiration dates, when applicable, are supplied by the vendor or by the Standards Laboratory. An expired radionuclide standard is acceptable for calibration if it has not physically changed, such as through evaporation or precipitation of solids and if the radionuclides have not decayed by more than five half-lives.

Some standards (provided by the Standards Laboratory), such as radioactive materials, are verified by preparing mounts. The mounts are counted by the counting room and compared against the calculated certified value. Standards for ICP Emission Spectroscopy are submitted to the analytical laboratory based on the traceable certified value. The material is then checked against an independent standard by the analytical laboratory for verification. Organic compounds used for calibration standards are purchased by the Standards Laboratory, but are prepared by the chemists before calibration.

Standards that are prepared and used for the first time need to be verified against an existing working standard or against an independent source to ensure accuracy of the standards (e.g., ICVS). When the primary standards that are analyzed for the first time could not meet QC parameters and the problem originated from the manufacturer, a nonconformance report shall be issued. When the secondary standard prepared by the Standard Laboratory could not meet QC parameters, the Standard Laboratory shall be notified.

7.3 CALIBRATION RECORDS

WSCF maintains calibration records for all methods requiring LMS calibration. These records include raw data (that is, instrument output including initial and continuing calibration verifications), the effective date of the calibration, and the analyst's name or initials. Calibration records are maintained as hardcopies in files or as electronic files.

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 more than one calibration per day is possible, then the date and time-of-day information must also be recorded (for both calibration and analyses). Calibration data shall be traceable to the standard(s) used via analysis date and preparation date of calibration standard(s).

7.4 CALIBRATION OF BALANCES

Calibration of analytical balances is performed annually at a minimum. Standard weights used in the calibration of balances are verified once per year. 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. Balances not passing calibration checks are marked and removed from service.

The calibration of balances is verified by measurement of an external check weight using various procedures (e.g., LO-140-008, *Routine Use and Quality Assurance for Analytical Balances at 222S and WSCF*), based on the type of balance being verified. The external check weights shall be S-class when appropriate. The calibration of balances is verified at a minimum before use, or on a daily basis, whichever is less frequent. The results of the check are logged and compared to acceptance criteria in the appropriate notebook, maintained in the same room as the balance. If the check fails after user's calibration described in manufacturer's manual or appropriate laboratory operating procedures (LO), the balance is taken out-of-service. The data from the checks are evaluated by the analyst for performance acceptance. When balance is moved to a different location, the chemist/analyst needs to notify laboratory operations engineering. Balances need to be calibrated after moving to a different location.

In addition, balances are verified once a year by an independent organization. Notification to management is required when the balances fail to meet acceptance criteria. When this condition occurs, potential impact on data quality is evaluated by the management. If there is no impact on data quality, a "no impact statement" will be documented in the balance notebook. If there is impact on the data quality, corrective action will be initiated and documented according to Section 15.1.

7.5 CALIBRATION OF THERMOMETERS

The accuracy of thermometers and thermocouples used for critical temperature measurements (e.g., refrigerator temperature for sample storage in N4, total dissolved solids analysis) shall be verified annually by comparing readings of such devices with the readings of a NIST traceable factory-certified thermometer. The verification of accuracy is performed by the maintenance group. If the verification fails (readings fall outside the stated tolerance) then the thermometer is either taken out of service or a temperature correction factor is determined and is affixed to the thermometer. The user maintains the records.

7.6 CALIBRATION OF PIPETTES

It is recommended and considered good laboratory practice that mechanical pipettes used for "critical" measurements are verified daily or before use, whichever is less frequent, to ensure acceptable performance. "Critical" measurements are defined as those measurements that cannot be or are not second source checked (e.g., making a working standard using a pipette). Calibration curves that are checked using an independent second source will reveal not only a pipette that is not functioning properly but also misuse of the pipette by the analyst. Daily pipette verification is encouraged to avoid costly re-analyses whenever necessary.

7.7 CONTROL OF LABORATORY MEASUREMENT SYSTEMS

Laboratory measurement systems are identified by using either manufacturer's model and serial numbers or a unique tracking number assigned by WSCF laboratory or support personnel.

The measurement systems are operated by the authorized and qualified personnel. The qualification requirements are described in Section 3.0.

Performance of any new instrument(s) shall be verified before placing in service. Operation and use of measurement systems are performed in accordance with manufacturer manuals and/or laboratory procedures. Measurement systems shall be subject to calibration or calibration verification before use for analysis of samples. The continuing calibration verification checks the stability of the original calibration over time. Standard used as the continuing calibration verification may be from the same source as that used for either calibration or initial calibration verification. Acceptance criteria for calibration as well as initial and continuing calibration verification are specified in the analytical methods or in accordance with sections 7.0 and 11.0. When measurement systems fail calibration QC criteria, appropriate corrective actions are required.

Preventive and operational maintenance of measurement systems are performed in accordance to Section 14.0 of this document. Handling of measurement systems repair is described in Section 11.6 of this QAPP.

Records shall be maintained for calibration and control of measurement systems, including type of test, date of test, name of person performing the test, results and their acceptability, corrective actions taken when unacceptable.

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8.0 LABORATORY PROCEDURES

Laboratory activities are directed and controlled by approved procedures, supporting document, or other documentation. When possible, regulatory and consensus methods (e.g., SW-846, American Society for Testing Materials (ASTM), standard methods, Methods for Chemical Analysis of Water and Waste, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, and DOE Methods) are used at WSCF.

8.1 PROCEDURE WRITERS

Procedure writers handle the process to initiate, develop, review, revise, approve, issue, inactivate, reactivate, and cancel the ASP procedures. The LA, LC, LT, LR, LO, and LQ procedures are processed and controlled according to ASP-200, Section 1.24, *Analytical Services Technical Procedure Process*. The maintenance procedures are processed and controlled according to ASP-200, Section 1.18, *Technical Procedure Process.*

The review and approval process for procedural change is documented on a Document Change Form (DCF).

Procedure writers provide goldenrod copies of LA, LC, LT, LR, and LQ procedures. LOs are provided to the laboratory on regular paper. The controlled copy of LO is the electronic procedure that is available on the procedure share area. Electronic documents are referenced to ensure the current version of the hard copy document is being used. A hard copy is provided as a backup copy in case of system failure.

8.2 LABORATORY PROCEDURES

8.2.1 Laboratory Technical Procedures

Procedure writers group processes laboratory technical procedures (LA, LC, LT, LR, LQ and LO), incorporates required changes, prepares the final document, and performs the final administrative review.

Each procedure has a unique identification code based on an approved numbering system (ASP-GD-02, *Laboratory Numbering System*). All procedures have a revision/modification identifier that appears on the bottom of each page, an identification code (document number), release date, official release stamp, author, author's manager, and the title. Traceability of the procedure revision is established via issuance date of the procedure.

Chemists/scientists or other technically qualified personnel prepare procedures for each laboratory analysis. The procedure is then reviewed by technical organizations or individuals to ensure technical correctness. The procedures shall be approved before use in accordance with ASP-200, Section 1.24.

The approved laboratory technical procedures (LA, LT, LR, LC, LQ) are issued as a performance "goldenrod" copy. Procedure distribution is administered such that controlled copies are maintained at controlled user locations and are updated by procedure writers.

Active procedures have no fixed expiration date, but require a review on a periodic basis for accuracy and adequacy by technically qualified personnel. The review is documented on the DCF and maintained by procedure writers.

A procedure can be inactivated or reactivated by the responsible scientist or engineer by using a DCF to notify procedure writers for the status change.

8.2.2 White Copy Procedures

White-copies of procedures are uncontrolled copies and are used for reference only. Use of white-copies is permitted and is available electronically on the Lab Procedure network directory (share area). Before use, white-copies must be verified to be the current version by comparison of the same revision with the electronic procedure.

An LOI can be used when the following situations occur:

- When a regulatory/consensus method requested by the client is not a common method used in the laboratory and is not issued as the laboratory procedure

- When there is no regulatory/consensus method readily available that is suitable for a specific type of matrix or specific types of samples, or unique analysis requested from the client.

LOI includes reference to the regulatory/consensus method when applicable or a brief description of procedural steps to direct the chemist to perform the analysis. An LOI can be prepared by the chemist or technical manager and includes signatures from the manager, client when applicable, and QA signatures or their designee.

Standing orders can be used to direct an individual to perform a procedure under certain conditions (ASP-200, Section 2.15, *Timely Orders*).

8.2.3 Procedural Changes for Issued Laboratory Technical Procedures

The laboratory makes changes to procedures (both regulatory and internally developed procedures) for a variety of reasons (e.g., health and safety issues, environmental and waste disposition considerations). Procedure changes are necessary to ensure procedures reflect current practices, equipment changes, and/or new requirements. When the need for a change to an issued procedure is identified, the change is initiated and processed in accordance with ASP-200, Section 1.24. The review and approval processes for laboratory procedure changes (e.g., LA, LC, etc.) are documented on a DCF.

Category of changes to the issued laboratory technical/analytical procedures includes modifications, temporary changes, or revisions. Three categories of changes are described in the following subsections.

8.2.4 Modifications (based on Document Control aspects only)

Modifications are limited to alterations that do not change the procedure's intent, quality, safety, or process such as changes in format, grammatical, typographical, or spelling errors, pagination, references and organizational names and titles. All modifications are reviewed and approved on a DCF.

8.2.5 Field Changes

Permanent or temporary changes are issued via Field Change (FC) (ASP-200, Section 1.24) to immediately implement a technical change or to permit a temporary departure from an existing procedure.

8.2.6 Revisions

Revisions are any change that does not meet the criteria of the above-described modification. Typical technical changes in which a revision may be needed are changes in technical content, which alter results or affect approval designations. All technical changes made to a procedure are reviewed and approved on a DCF.

8.3 DOCUMENTATION OF TECHNICAL PROCEDURE CHANGES

When a situation occurs such that a change from the analytical procedure is inevitable, documentation in the analyst notebook or preparation sheet is required. The decision to deviate is based on published literature and/or known sample chemistry. Procedural deviations include the following three categories:

- Substitution is an equivalent adjustment to a procedure, which would not have significant effect on the final result. This would be evident in the QC data associated with the final results. A permanent substitution would require a revision to the procedure using the DCF. Substitution is documented in the analyst notebook or preparation sheet.

- Method 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 of the potential impact to the method, such a modification must be demonstrated by application. Modification requires the procedure to be qualified, documented, approved by laboratory management, and agreed upon by the client. Justification of the modification should be evident in the QC data associated with the final results. A modification with long-term applicability should be developed into a new laboratory procedure that is issued with a new title and code. Modification of regulatory methods requires notification to be submitted to the

regulatory agency via DOE-RL (per HASQARD). Refer to Section 8.6.1. for more information regarding notification requirements. A temporary modification is permitted through the use of the FC process.

- Deviation is any divergence from the original procedure that does not adversely impact precision, accuracy, detection limit, and other QC criteria. Documentation of deviations made shall be included in the final report narrative or in the LABCORE comment field. Justification of the deviation should be evident in the acceptable performance associated with the final results. 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. Deviation is documented in the analyst notebook or preparation sheet.

8.4 NEW TECHNICAL PROCEDURES

New analytical procedures shall be qualified before use. New procedures are defined as procedures used for the first time whether based on published, well-understood procedures, or developed in the laboratory. Exception is granted to certain situation(s) that prevent this requirement to be applied such as unique matrix of the samples or unique request of analysis (e.g., roofing material, railroad material, unknown complexed waste sample matrices, particle size determination).

If the procedure is based on a published procedure, then MDL studies (at a minimum in the water matrix), accuracy and precision serve to qualify the procedure. MDLs are filed and maintained in the laboratory.

When developing new analytical procedures, the first stage is to conduct and document the performance using standard materials and to establish the following parameters as appropriate:

- Accuracy/precision
- Detection limits
- Determination of method interferences as appropriate to the method
- Parameter variable studies
- Linear ranges
- Effect of chemical interferences
- Effect of reagent concentrations
- Effect of instrument parameters
- Kinetic effects.

Performance shall be verified using one or a combination of the following parameters as appropriate:

- Complex standards
- Matrix standards, if reasonable
- Spikes or method of standard addition on actual samples

- Independent analytical method
- Sample exchange programs
- Comparison with standard, approved methods on actual samples.

When a regulatory/consensus method requested by the client is not a common method used in the laboratory and is not issued as the laboratory procedure, an LOI shall be established to document the method used for sample analysis. In addition, applicable acceptable QC (e.g., reference materials, standards, or spike) needs to be demonstrated before analyzing the samples. Specific instructions that are not available in the method shall be documented in the analyst notebook for repeatability of the sample analysis.

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, analyze a sufficient number of reference samples and compare the results obtained to known values. A simulated matrix may be the closest performance indicator available; or

- 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.

In all cases, a suitable number of replicate determinations must be made to provide a measure of statistical control. Administrative levels will be set until sufficient data are acquired, typically 30 data sets, to warrant statistical analysis.

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. Method testing includes detection limit determinations, blank evaluation, precision and accuracy determination, counter performance, uncertainty, and determination of matrix interference.

All method qualification data shall be retained on file and traceable to the procedure it supports.

8.6 MODIFICATION OF REQUIRED REGULATORYMETHODS

Modifications to the required regulatory method that is mandatory shall be specifically described by providing a synopsis or direct quotation of the regulatory method requirement and a description of all changes make. The reason(s) why the requirement cannot be met shall be provided.

8.6.1 Regulatory Notification

The notification mechanism available to the laboratory requires DOE, Richland Operations Office (RL) to coordinate with the regulator. The laboratory must obtain documented approval from RL to use the new procedure before starting work. The time frame for acceptance shall be documented and agreed upon. Information regarding regulatory acceptance considerations can be found in references such as WAC 173-303-910(2) and 40 CFR 136.4. Washington State Laboratory Accreditation Program may have other/additional requirements.

8.7 PREPARATION AND REVIEW OF SUPPORTING DOCUMENTS

Supporting documents (SDs) are used to document QAPPs, QAPjPs, basic laboratory practices, technical project plans, or 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/or 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 WSCF organizations. The reviewers document the approval of the SD on the Engineering Data Transmittal (EDT) form (BD-7400-172). See HNF-PRO-440 for detailed information.

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9.0 DATA COLLECTION, REDUCTION, AND REPORTING

9.1 DATA COLLECTION

The WSCF uses a LIMS (e.g., LABCORE) to initiate, track, and report sample analyses. When samples are logged into LIMS, a file is created in the computer database, which initiates sample analyses and begins the tracking process. Each sample is assigned a unique sample number. The date sampled and the analyses requested are included in the record. Sample Availability Reports indicate to laboratory personnel which samples are ready for analysis. The LIMS provides a 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 LIMS.

Raw data is the collection of instrument output and manually generated data associated with a final reportable result. Raw data is primarily generated and maintained electronically, but some manual collection may be necessary depending on the analysis. All raw data output is retained by the individual analyst responsible for the analysis and is filed for record according to Section 10.0. Raw data includes Information on the date of sample collection, sample preparation, analysis performed, sample identification numbers, analyst, and procedure number. Such information is traceable to the raw data output. Procedure revision number is traceable to the procedure issuance date. Critical parameters that are specified in sample preparation procedures shall be recorded in analyst's notebook or spreadsheet.

Where necessary, manual collection of raw data is completed by the analyst and recorded in notebooks or spreadsheets. Entries on paper records and/or notebooks are made in ink and are made in manner such that they can be easily read, understood, and reproducible with standard photocopier. Incorrect entries are initialed, dated, a line drawn through the incorrect entry in such a way that the entry is still readable, and explained as appropriate. All data entries are dated and marked with the initials of the analyst doing the work. Final data from the notebooks or spreadsheets are then entered into LABCORE. At this time, each individual generating data or information should be aware of possible sources of data entry errors, sample identification errors, calculation errors, and correct these errors as needed. Data entries are traceable to the individual who made the entries and the date of the entries. All computer systems have provisions for making corrections. Changes to computerized data records shall be identified such that original and corrected entries are retrievable and the individual initiating the changes can be identified.

9.2 DATA REDUCTION

Data Reduction is defined as the mathematical operations applied to the raw data to produce a final reportable result according to applicable methods. Data reduction is mainly performed electronically throughout WSCF via spreadsheets or instrument-controlled software (see Section 5.0 of this document for software control requirements). Formula or spreadsheet used for data reduction shall be verified before use to ensure calculation and data manipulation programs perform properly. Analysts or technical personnel need to ensure the following practices are in place: verify that all readings or output are accurate, proper error correction or data changes, use of the appropriate formulas for calculating final results (including applicable backgrounds and/or interference correction), and accurate transcription of data.

9.3 DATA REPORTING

The analytical information reported, at a minimum, includes the named parameters, analytical methods, and the data values (with appropriate qualifiers). Inorganic and organic data are reported as numeric values with appropriate data qualifiers if above the instrument detection limits (IDL)/MDL/VPRQL. When the value is less than IDL and/or MDL, it is reported as undetectable or as IDL/MDL with an appropriate qualifier (e.g., less than value). Radiochemical results are reported or activity corrected for the appropriate blank and supported with estimates of uncertainty.

Analytical results are transmitted to laboratory customers in one or a combination of the following ways: 1) the customer receives data through an established direct link between the LIMS and the customer's database (LEMIS, HEIS, and so forth); 2) a summary data package is generated by LIMS containing all the requested analytical results and/or associated laboratory QC results; 3) a diskette deliverable is provided in a customer-defined format such as Format for Electronic Analytical Data (FEAD); or 4) preliminary data may be sent in the form of a e-mail or telefax. Refer to section 9.3.3 for specific information on data reporting documentation.

The WSCF reports are submitted only to the client contact requesting the work, other recipients specifically identified by the client, or new contractors who have assumed responsibility from the original client. Reports will not be submitted to other parties, with the exception of the DOE without prior written permission of the client.

9.3.1 Significant Figures and Rounding Methods

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.

When a figure is to be rounded to fewer digits than the total number available, the following rules apply:

- When the first digit discarded is less than 5, the last digit retained should not be changed
- When the first digit discarded is greater than 5, the last figure retained is increased by 1
- When the first digit discarded is exactly 5, followed only by zeros, the last digit retained should be rounded upward if it is an odd number, but no adjustment is made for an even number.

Recognizing that vendor-supplied software may not meet the general rules for significant figures, rules used by the vendor are acceptable provided the results are not significantly different from the rules described above.

9.3.2 Data Review Process

Data review is the process of determining whether data conform to specified requirements. Data reports are reviewed before the reports are issued. Laboratory shall perform data review against sample data (e.g., verification of sample identification number, analyst, and date of analysis), laboratory procedures, and against applicable QC criteria. If QC samples did not meet QC criteria, data within the batch shall be evaluated to determine if there were any adverse effects on the data; the sample shall be re-prepared and/or re-run or the data shall be reported with qualification(s), which will be detailed in the LABCORE comment field as appropriate to the condition. The data review process consists of checking for transcription errors and ensuring that applicable QC criteria were met (see ASP-315, Section 9.3, *WSCF Laboratory Data Review*). Errors detected in the review process are referred to the analyst for corrective action and are corrected accordingly.

9.3.3 Reporting Documentation

Laboratory reports shall include the following information:

- Laboratory name
- Sample information including unique laboratory identifier cross-referenced to client identification, sample collection information, date of sample receipt, and date(s) of sample preparation and/or analysis
- Analytical units and results, reported with an appropriate number of significant figures, and identification of subcontracted results if applicable
- Report uncertainty for radiochemical analysis
- Detection limits
- Method reference
- Appropriate QC results (correlation with sample batch shall be traceable and documented) when applicable
- Appropriate data qualifiers or client-specific qualifiers with definitions and a narrative on the quality of the results, if applicable:

  Qualifier "B" is used when the analyte was detected in the associated method blank and exceeded the Estimated Quantitation Limit (EQL), but less than 20X of analyte. When batch QC fails, qualifier "J" (estimated value) is used along with chemist narrative/comments. Qualifier "U" is used when the constituent was analyzed for, but was not detected. Qualifier "N" is used when the analyte is identified based on a mass spectral library search. Qualifier "E" is used for analyte concentration exceeded calibration range. Qualifier "D" is used in the organic results when analyte concentration resulted from a dilution.

- Additional data reporting (e.g., the percent of moisture/solid or correction for equivalent dry weight) may be included if requested by the client.

WSCF offers a variety of analytical data reports or reporting formats depending on the client's needs; (1) LIMS Results Only Reports, which include a LIMS printout of results for each

analyte requested, (2) the Summary Data Package consists of a cover page, Laboratory Report, Analytical Comment Report, Tentatively Identified Peak Report, QC report if requested, (3) the Data Package consists of a coversheet, table of contents, case narrative (including a summary of the nonconformance of samples against COC and corrective actions), results, QC summary, COC, and "end of package" page, and (4) client specific data package (e.g., Vapor Program) directed by the relevant SAP.

The following are examples of Tank Farms Vapor Program Reporting formats (subject to change):

- Format I Reporting (Notification Reports) is utilized for projects that establish notification limits for specific analytes. Analytes that exceed notification limits shall be reported by the Project Manager or delegate by calling Double Shell Tank Farms Operation shift manager as soon as the data are obtained and reviewed by the responsible chemist. This verbal notification must be followed within one hour by electronic notification to the Double Shell Tank Farms Operations shift manager, the Industrial Hygiene and Safety point of contact, the TWRS Process Engineering Data Assessment and Interpretation manager, airlift circulator operations point of contact and the Process Engineering point of contact for vapor sampling.

- Format II Reporting. Information on the sampling shall be reported by the Characterization Project Operations (CPO) sampling team and WSCF as Format II reports and the point of contact in Process Engineering. CPO will provide the sample collection sequence and volumes, start and stop times for the collection of each sample, the Total Organic Carbon reading at the start of the collection of each sample, verification of equipment and field blank use, any anomalous sampling conditions, and WSCF will provide the results of the tritium trap and particulate filter analysis. This report is to accompany, if possible, the shipment of samples. Alternatively, this sampling report may be delivered to WSCF within 48 hours after the samples have been relinquished from the samplers.

- Format VI Reporting. The Format VI report shall consist of one deliverable, the sampling and analytical results and associated QC results. The details of the deliverables are specified in the relevant SAP.

Electronic data deliverable formats will be defined by the client. A direct link from the laboratory's LIMS to the client's database can be established, which will alleviate the need for a diskette deliverable.

9.3.4 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 reports do 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 e-mail or by telefax.

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10.0 LABORATORY RECORDS

10.1 LABORATORY RECORDS

Laboratory RECORDS are defined as "Information contained on hard copy, microfilm, or magnetic tape, photocopy, and electronic systems that is complete in terms of appropriate content and furnishes evidence of the quality of items and/or activities assuring quality. Records may be original documents, or, legible, reproduced copies." HNF-RD-210, HNF-PRO-10588, and ASP-200, Section 1.04 describe record management program and process.

10.1.1 Identification and Generation of Records

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

- COC, disposition report, copy of shipping document
- Training records (attendance records); qualification of personnel and procedures
- Analytical results; instrument output; QC results; calibration records
- Results of reviews
- Instrument run logs and maintenance logs
- Assessments/inspections
- Procurement documents.

Reports and other supporting documentation transmitted to WSCF clients become the clients' records. These are maintained by WSCF only for references, unless the client notifies WSCF otherwise.

10.1.2 Organizational Responsibilities for Records

Record generators are responsible for:

- Storing the in-process records properly
- Filing the Records Inventory Disposition Schedule (RIDS) and indexing the files in each storage area using a RIDS-compatible format
- Complying with laboratory records management program.

Laboratory managers are responsible for ensuring laboratory staff follow the records management program.

Information Resource Management (IRM) is responsible for the records system based on HNF-RD-210 and HNF-PRO-10588, which also governs specification, preparation, review, approval, and maintenance of records.

10.1.3 Record Filing and Record Storage

Based on the nature of records, types of media, stage of record generating process, record storage areas are identified in the following:

10.1.3.1 In-process Record Storage

- Records can be stored in the file cabinets or in the desks with proper identification in the laboratory or in the offices.

- Records shall be labeled properly (e.g., date, type of analyte, project number or sample numbers, analyst signature) and kept chronologically.

- Records stored in the record generating stage shall be kept for a minimum of **two** years (current calendar year and previous two calendar years).

- At the beginning of calendar year, a new recording system should start with a few exceptions (e.g., training records, counting spectroscopy calibration notebooks, instrument maintenance logs, control charts). A new notebook, new magnetic tape, new backup tape should be used in order to facilitate movement to temporary storage. Training records may be kept longer when personnel status remains the same.

- RIDS or indexes that have RIDS compatible format shall be established for the records in the record-generating stage.

10.1.3.2 Temporary Records Storage

- After the end of the two-year period, records should be transferred to file cabinets in a temporary record storage area.

- Records will be held in this area until they are at least three years old. Only authorized personnel have access to those file cabinets. Records stored in the temporary record storage area shall be properly labeled for retrievability.

- Depending on projects needs or the availability of permanent records storage area, the records will be moved to the permanent records storage area in according to HNF-PRO-10588.

10.1.4 Records Media

Records can be in the following media:

- Original hard copy or notebook (HNF-PRO-10863)
- Legible copy of the original
- Electronic media
- Magnetic tapes

- Printed copy of electronic mail message
- Optical Scanner.

Records that are in the electronic media must be retrievable either by the laboratory staff when possible, or by a commercial company specializing in record retrieval. If a client requests record retrieval, any costs of the retrieval will be paid by the requester.

10.2 DISTRIBUTION

Many of the laboratory records are maintained within the laboratory and have no distribution. For those records, that have a distribution, either original records or copies will be maintained in the laboratory upon generation.

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

10.3 STORAGE

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

Access to records system is limited to authorized personnel only.

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

10.4 RETRIEVAL

Data and records are filed in compliance with established RIDS (e.g., customer, laboratory sample number, and/or date). Raw data stored in instrument software must be accessed through the chemist assigned to that instrument and/or laboratory computer.

10.5 MAINTENANCE AND DISPOSAL

Certain laboratory records, such as those for environmental or accountability analyses, are to be maintained indefinitely. The retention times for those laboratory records that can be disposed of, are found in the RIDS, which is prepared by the laboratory and approved by the Records Management group. HNF-PRO-10588 describes the following records management, which are the responsibilities of the Records Management group:

- Maintenance of active records for transmittal, distribution, change and correction, retention, protection, preservation, traceability, archival, disposition, and retrievability.

- Verification that records received are legible and are in agreement with the transmittal document,
- Control for the control and accountability of records removed from the storage location
- Filing of supplemental information and disposition of superseded records
- Replacement, restoration, or substitution of lost or damaged records.

HNF-PRO-10588 describes the processes for receiving, storing, controlling, or disposition of records. Appropriate record retention time is monitored by the RIDS system.

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11.0 QUALITY CONTROL

The following section describes the QC options used for analysis performed at the WSCF. Not all options will be used for each analytical batch. Specifications listed in the regulatory methods requested by the client take precedence over HASQARD requirements. If no specifications of QC listed in the regulatory or non-regulatory method is used for analysis, HASQARD QC requirements (Table 6.1 - 6.8, Volume 4) for appropriate instrument are followed. The degree of QC required for analysis will at minimum meet the requirements of HASQARD and/or the specific QC protocols of the analytical method. Quality Control requirements may be imposed by the client and defined in program or project guidance documents. Unique client requirement shall be documented and agreed upon by the laboratory and the client before work begins. Preparative QC results should be reported to the client as part of routine reports, unless specified by the client. Reported results shall be traceable to the QC performed with those results. The client shall be notified prior to re-preparation of samples due to failure to meet client DQO or data quality requirements (DQR) if additional costs will be incurred.

The following areas of QC are addressed:

- General Laboratory QC
- Inorganic analysis QC (preparative and analytical)
- Organic analysis QC (preparative and analytical)
- Radiochemical analysis QC (preparative and analytical).

11.1 GENERAL LABORATORY QUALITY CONTROL

The use of substandard reagents, standards, materials, and equipment can result in less reliable or unreliable data. Contamination from these sources could jeopardize the accuracy, precision, and sensitivity of reported results. The WSCF controls these sources by consistently monitoring analytical and preparative blanks for contamination.

11.1.1 Reagent Water

Reagent 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. The conductivity is checked periodically after reverse osmosis. The water is then supplied to several laboratory locations where it is further purified using smaller commercial finishing units containing deionizing resins, activated charcoal, filters, or other polishing units as appropriate. These systems are designed to produce equivalent quality to ASTM Type II water.

The quality of water is also monitored through the use of analytical and preparative blanks in each batch. The acceptance of preparative blank is defined in the procedure or documented in the method or in the notebook. Laboratory water quality is maintained by using reagent water as described in this section. The reagent water takes precedence over Type II water if Type II water is referenced in the analytical procedures or other documentation.

11.1.2 Reagents/Chemicals/Standards

All reagents and chemicals used for analyses are ACS reagent grade or better unless this quality is not available or the analytical procedure specifies otherwise. Reagent quality is monitored via preparative and analytical QC performance. LO-120-001 or LO-120-007 provides instructions and guidelines on proper labeling of reagents and standards.

Specification of grade for chemicals and preparation of working standard that are used in the analysis is provided in the analytical procedure/method. See Sections 7.1 and 7.2 for guidance on selection and verification of standard used for calibration. Use of dual-source standards provides the mechanism for verifying the concentration of new standards. When working with neat source materials, independent preparation for calibration and check standards replace the use of dual-source standards.

Prepared working standards are labeled with sufficient information to provide identification, to provide traceability to the appropriate preparation records, and to specify expiration dates when applicable. Laboratory personnel are responsible for labeling working standards and solutions.

The acceptability of such standards and chemicals is monitored via preparative and analytical QC performance. Expired standards may be re-qualified as check standards with new expiration dates after approval by the analytical chemistry management and QA.

Reagents/chemicals/standards are stored according to manufacture's recommendations. Refrigerators/freezers that store reagents, chemicals, and/or standards are classified based on the usage of each refrigerator/freezer (LO-150-445). Temperatures of these refrigerators and/or freezers are monitored periodically. The majority of reagents/chemicals/standards require refrigeration or freezing with no specified temperature range. When no range is specified, WSCF assumes a temperature of less than 15 °C is adequate for refrigeration and a temperature of less than -10 °C is adequate for freezing.

11.1.3 Compressed Gases

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

11.1.4 Labware/Glassware

When performing an analysis, or generating a reagent or standard, labware is chosen such that the tolerance specified in the procedure is met. Volumetric glassware and pipettes purchased from vendors are usually Class A. In some cases, a piece of glassware is purchased, which has a Certificate of Accuracy (or Certificate of Calibration). If tighter tolerances beyond Class A are required, the item is calibrated. Labware is cleaned to be free of contamination based on usage of the labware. Selection of labware is based on the need of testing performed. General requirements and considerations for washing and maintaining labware are provided in LO-110-400, *Labware Cleaning for the WSCF Laboratory*.

11.1.5 Housekeeping

Each employee is responsible to maintain his (her) work area in a neat and orderly manner to avoid contamination. Inspection of housekeeping including inside fume hoods is performed periodically.

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:

- Recognize problems and address them before the system goes out of performance
- Document conditions or corrective actions that "fix" particular performance problems (for future reference).

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 recovery of the LCS. Control limits will be established either (1) set at 3 sigma, (2) based on environmental regulatory requirements, or (3) as determined by the laboratory.

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 shall 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 (for example, generated by the LABCORE or by application software), or hard copy printouts generated periodically as close to real-time as practical.

The technical staff will have an understanding of the maintenance and use of control charts. The technical staff is expected to use them for system monitoring and early trend detection.

11.2 INORGANIC ANALYSIS QUALITY CONTROL

This section describes QC tools, which may be used for batch quality control. Not every QC elements described is required per batch.

11.2.1 Preparative Quality Control

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

(method blank), LCS, duplicate (or matrix spike duplicate), and matrix spike. Preparative QCs are prepared with each batch of samples.

11.2.1.1 Preparative Batch

A preparative batch is a group of samples with similar matrix prepared and/or separated at the same time. A batch does not exceed 20 samples (not including QC samples). More than 20 samples can be prepared at the same time, if the required numbers of QC samples are performed for each batch. If clients do not specify project-specific QC, similar matrix type of samples are combined up to 20 samples for preparation batch with one duplicate (or matrix spike duplicate) and matrix spike. In the case of process testing or unique client requirement, QC elements such as a sample duplicate (or matrix spike duplicate) and matrix spike in the preparation batch can be performed per specified period or for every 20 client-specific samples received. However, the preparation blank and laboratory control sample/blank spike requirement would apply to each batch of samples.

A sample or series of samples, which do not require preparation or separation prior to analysis, would not fall under the requirements in this section. Simple dilution is not considered a sample preparation technique. Additionally, limited sample quantity may limit the laboratory's ability to meet the duplicate and matrix spike requirements. In such cases, alternative approaches should be considered to demonstrate sample precision and accuracy.

11.2.1.2 Preparation Blanks

The sample preparation blank (method blank) is used to monitor contamination resulting from the sample preparation process. The preparation blank is generated by using reagent water or a material similar to sample matrix that contains none of the analytes of interest. The preparation blanks are subjected to the same sample preparation, treatments (e.g., volume or weight, process), 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.

Preparation blank or method blank acceptability is demonstrated by the concentration of the target analytes in the blank meeting one of the following requirements:

1) less than the estimated quantitation limit (EQL) of the associated samples, or, if the blank is equal to or above the EQL,
2) not higher than 5% of the measured concentration present in the sample, or, when a decision level is specified
3) not exceeding 5% of the regulatory decision level for that analyte, unless 2) above applies,
4) client specific data quality requirements.

If the preparation blank (method blank) fails to meet the acceptance criteria, all affected samples in the preparation batch will be re-analyzed, re-prepared and re-analyzed, or results will be reported with an appropriate flag. Sample results do not subtract preparation blank or method blank unless required by the client. In which case, blank subtraction shall be noted in the report.

11.2.1.3 Laboratory Control Sample or Blank Spike

A LCS or blank spike (BS) is used to monitor the effectiveness of the entire analysis process or an analytical system. The LCS or BS shall be prepared with each batch of samples processed at the same time and carried throughout the analysis. The LCS is a material similar in nature to the sample being processed containing the analyte(s) of interest (e.g., standard reference material). The BS is reagent water or other suitable substrate spiked with the analytes of interest. A BS is normally used when an appropriate laboratory control sample is unavailable. Blank spike control is demonstrated by target analytes being within either the preset limit of 80 - 120% or, within statistically determined limits when the $\pm$ 20% criteria cannot be reliably achieved.

LCS control is demonstrated by target analytes being within established control limits. Control limits are established by one of the following:

- As determined by cognizant scientist
- Specified by regulatory requirement
- Tolerance limits or control limits provided by the vendor
- Statistically determined by multiple analysis over time.

When one of the acceptance criteria has not been met for an analyte of interest, samples in the preparation batch or the LCS/BS will be re-analyzed, or re-prepared and re-analyzed for that analyte. In some cases, results can be reported with a flag (e.g., inadequate sample for re-preparation or results meet client DQO or DQR). No adjustment of client sample results based on LCS recovery is made in the laboratory report.

11.2.1.4 Matrix Spike

The matrix spike (MS) is used to monitor method performance in a specific matrix. Recovery of matrix spike is a measure of the accuracy in the measurement of the analyte(s) of interest present in the client sample matrix. The matrix spike is an aliquot of client 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
- 1 to 5 times the EQL.

Otherwise spiking should be performed at a level equivalent to 1 to 2 times that of the sample or at least 25% of that of the sample. When the concentration of the analyte in the original sample is greater than 0.1%, no matrix spike is required unless specified by client.

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
- Laboratory performance over time.

The recommended target level for matrix spike analysis is 75 to 125% recovery. Control limits are not applicable to spikes that are <25% of the analyte concentration in the sample. If the matrix spike recovery fails to meet the criteria, the batch results shall be investigated for sources of error. Re-preparation and/or re-analysis should be conducted as necessary based on the DQO or DQR. Spike recoveries outside established control limits will be flagged and/or explained in the narrative or in a LIMS comment section, 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 is not practical because of solubility concerns. Other methods of evaluating method performance such as serial dilution or post-digestion spike may be used.

11.2.1.5 Laboratory Sample Duplicate or Matrix Spike Duplicate

Laboratory sample duplicates are used to assess the precision of the preparation and analytical process in a client-specific matrix. Laboratory duplicates are two aliquots of the same sample (intra-laboratory split) that are taken through the entire sample preparation and analytical process. Matrix spike duplicates (MSD) are two spiked aliquots of the same sample that are taken through the entire sample preparation and analytical process. In cases where the sample is not expected to contain reasonable concentrations (greater than 10X the instrument detection limit) of the analyte(s) of interest, matrix spike duplicates are used.

One set of laboratory duplicates (or MS/MSD) is required for each batch of samples. Precision is estimated by calculating the relative percent difference (RPD) of the duplicate analysis.

Inorganic RPD criteria are normally set at 20%; such criteria shall only be applied to samples with analyte concentrations greater than 10 times the methods detection limit. If the RPD fails to meet the criteria, evaluation of the source of error and impact on client DQR shall be performed. If the RPD falls outside the established limits, the results are flagged and explained in the narrative. It does not automatically trigger re-analysis unless directed by client.

11.2.1.6 Practical Control Sample

Practical Control Samples (PCS) are specific samples used to monitor analytical performance at client specified action limit. The PCS is only requested by the client and prepared as a blank spike to be used as an internal check sample. For reporting purpose, the PCS is assigned a sample number and treated as client's sample.

11.2.2 Analytical Quality Control

11.2.2.1 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 (Section 11.2.1). Each analytical sequence has associated with it a prescribed number and type of QC standards that are analyzed in a prescribed order. The analytical sequence is an important aspect of the analytical work performed because it allows the analyst and subsequent data reviewers to determine if there are trends in sample results or QC related to the order in which samples were analyzed. Therefore, the order in which samples are analyzed will be traceable to the analytical sequence.

For most inorganic analyses, the analytical run typically starts with either calibration or confirmation that the calibration is still valid and ends with a continuing calibration standard and blank. More than one batch of samples can be analyzed in an analytical sequence as long as continuing calibration control is maintained.

11.2.2.2 Initial Calibration Verification (ICV)

The ICV is used to confirm the accuracy of the calibration and the standards used for calibration. Standards for ICV are prepared from a source other than that used to prepare the calibration standard. The ICV is required whenever the system is recalibrated. The ICV shall be run following calibration or re-calibration and before analysis of client samples.

Acceptance criteria are stated in the laboratory procedure based on the specific regulatory method, for example, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 (EPA 1994) or in Table 6-3 of HASQARD, Volume 4 (see Section 11.0). Failure of the ICV indicates instrument and/or standard problems that must be evaluated and corrected before any client samples are processed for the analyte(s) of interest.

11.2.2.3 Continuing Calibration Verification (CCV)

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 and need not be nationally or internationally traceable. The ICV standard may also be used as the CCV.

Each inorganic analytical system shall include periodic checks on the stability of the instrument. For an unstable analytical system, these checks will be performed every 10 samples and at the end of the analytical run. The CCV acceptance criteria and frequency are stated in the laboratory procedure or in Table 6-3 of HASQARD, Volume 4 (see Section 11.0). Failure indicates that the analytical system has drifted out-of-control and requires correction for the analytes of interest. If CCV failure occurs, all samples analyzed after the last acceptable CCV shall be re-analyzed. Reanalysis applies to specific analyte failure. In limited cases, isolated

analyte failures may be tolerated if sample results still meet the client DQR. Reporting results in such cases requires justification in the report to the client.

11.2.2.4 Initial and Continuing Calibration Blanks (ICB and CCB)

Initial and continuing calibration blanks monitor effects such as contamination and instrument response drift. The initial/continuing calibration blank is a reagent blank, prepared similarly to the standards but not subjected to sample preparation except for techniques in which the preparation is an integral part of the analysis. In these cases, the preparation blank or method blank can be considered the equivalent of the ICB or CCB.

The ICV shall be followed by an ICB and each subsequent CCV shall be followed by a CCB. The acceptance criteria for these blanks are that they are less than or equal to EQL for each analyte of interest. When an analyte exceeds these criteria, potential impact on data quality shall be investigated and re-analyzed when sample carry-over is present. Samples with concentrations exceeding the blank contamination by a factor of 20 or more can be reported without a B qualifier unless client requirements dictate otherwise.

11.2.2.5 Low-Level Standard

The low-level standard is used to monitor instrument performance in the region at or just above the EQL. In those cases where it is used as part of instrument calibration, a separate low-level standard is not required. A recovery between 75% and 125% is recommended. If the low-level standard fails to meet these criteria, all client samples whose results are less than 10 times the EQL should be evaluated for impact, any limitations will be noted in the laboratory report.

11.2.2.6 Internal Standards

An internal standard (IS) is an analyte that is similar to the analyte(s) of interest in terms of its analytical responses, but which is not normally expected to be found in the sample. When internal standards are used, internal standards are added to every standard, blank, sample, QC samples (e.g., MS, MSD) before analysis. Selecting appropriate internal standards shall be method specific because all results are normalized based on internal standard performance. Internal standards are routinely used in inductively coupled plasma (ICP) - mass spectrometry. Use of internal standard and associated acceptance criteria are described in the specific method or procedures.

11.2.2.7 Interference Check Standards

Interference check standards are typically applied only in ICP Emission Spectroscopy 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 give recoveries 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.

11.2.2.8 Post Digestion Spike (Metals Analyses)

A post digestion spike (PDS) is a spike added to the sample after preliminary preparation, usually just before analysis. If a post digestion spike is used, the sample spiked would be that sample on which a matrix spike was originally performed. The PDS 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. The PDS is typically used for ICP analysis but is appropriate to other analyses as well.

Acceptable recovery is generally 75% to 125% for this spike. Performance of PDS can be used to identify problems caused by sample matrix effects during preparation and/or measurement. In case of unusual matrix effects during the preparation step, the MS will fail but the PDS will pass. If both the MS and PDS fail, the failure is due to matrix effects in both the preparation and measurement steps. If the recovery of PDS meets the acceptance criteria indicating that MS failure occurred during the sample preparation step, all client samples in the batch should be flagged in the laboratory report with comments on sample preparation problem. If the PDS fails to meet the acceptance criteria, this indicates a matrix problem. All results of client samples in the batch should be flagged and the reasons discussed in the laboratory report.

Re-analysis of the samples using different preparation and/or measurement procedures should be considered if alternative procedures are available. Prior to re-analysis, client concurrence must be obtained because the procedural changes may violate mandated existing regulatory or project requirements.

11.2.2.9 Serial Dilution

The serial dilution analysis is typically used when new or unusual matrices are encountered as an indicator of potential matrix-related interferences associated with analysis. 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 in the client sample. A percent difference of 10% or less indicates acceptable performance. The sample dilution is not applicable to analytes whose concentration 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. The client sample results in the batch, which does not meet the serial dilution acceptance criteria, should be noted and the possible matrix effects discussed in the laboratory report.

11.2.2.10 Method of Standard Additions

The method of standard additions consists of a blank and at least three standards to which aliquots of the sample are added. 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. However, the preparative QC is still required (including "auto-zeroing" on the calibration blank). The standards used should be approximate 50%, 100%, and 150% of the expected sample concentration.

11.3 ORGANIC ANALYSIS QUALITY CONTROL

This section describes QC tools, which may be used for batch quality control. Not every QC tool described is required per batch.

11.3.1 Preparative Quality Control

Preparative techniques are used to prepare a sample for analysis. This may include digestion, dissolution, extraction, phase partitioning, and/or leaching of a sample. Separation and/or isolation are also considered preparative if performed before analysis. Preparative QC includes preparation blank (method blank), LCS, duplicate (or matrix spike duplicate), and matrix spike. Preparation QCs are prepared with each batch of samples.

11.3.1.1 Batch

A preparative batch is a group of samples with similar matrix prepared at the same time. A batch does not exceed 20 samples (not including QC samples). More than 20 samples can be prepared at the same time, if the required numbers of QC samples are performed for each batch. If clients do not specify project-specific QC, similar matrix type of samples are combined up to 20 samples for preparative batch with one duplicate (or matrix spike duplicate) and matrix spike. In the case of process testing or unique client requirement, QC elements such as a sample duplicate (or matrix spike duplicate) and matrix spike in the preparation batch can be performed per specified period or for every 20 client-specific samples received. However, the preparation blank and laboratory control sample/blank spike requirement would apply to each batch of samples.

A sample or series of samples, which do not require preparation or separation prior to analysis, would not fall under the requirements in this section. Simple dilution is not considered a sample preparation technique. Additionally, limited sample quantity may limit the laboratory's ability to meet the duplicate and matrix spike requirements. In such cases, alternative approaches should be considered to demonstrate sample precision and accuracy.

11.3.1.2 Preparation Blanks

The sample preparation blank (method blank) is used to monitor contamination resulting from the sample preparation process. The preparation blank is generated by using reagent water

or a material similar to sample matrix that contains none of the analytes of interest. The preparation blanks are subjected to the same sample preparation, treatments (e.g., volume or weight, process), 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.

Preparation blank or method blank acceptability is demonstrated by the concentration of the target analytes in the blank meeting one of the following requirements:

1. less than the estimated quantitation limit (EQL) of the associated samples, or, if the blank is equal to or above the EQL,
2. not higher than 5% of the measured concentration present in the associated sample, or, when a decision level is specified
3. not exceeding 5% of the decision level for that analyte, unless 2) above applies,
4. client specific data quality requirements.

As a special requirement, the concentration of methylene chloride, acetone, 2-butanone and phthalate esters shall be less than 5 times the EQL.

If the preparation blank (method blank) fails to meet the acceptance criteria, all affected samples in the preparation batch will be re-analyzed, re-prepared and re-analyzed, or results will be reported with an appropriate flag. Sample results do not subtract preparation blank or method blank unless required by the client. In which case, blank subtraction shall be noted in the report.

11.3.1.3 Laboratory Control Sample

A LCS or blank spike is used to monitor the effectiveness of the entire analysis process or an analytical system. The LCS or BS shall be prepared with each batch of samples processed at the same time and carried throughout the analysis. The LCS is a material similar in nature to the sample being processed containing the analyte(s) of interest (e.g., standard reference material). The BS is distilled or deionized water or other suitable substrate spiked with the analytes of interest. A BS is normally used when an appropriate laboratory control sample is unavailable. Blank spike control is demonstrated by target analytes being within either the preset limit of 80 - 120% or, within statistically determined limits when the $\pm$ 20% criteria cannot be reliably achieved.

LCS control is demonstrated by target analytes being within established control limits. Control limits are established by one of the following:

- As determined by cognizant scientist
- Specified by regulatory requirement
- Tolerance limits or control limits provided by the vendor
- Statistically determined by multiple analysis over time.

When one of the acceptance criteria has not been met for an analyte of interest, samples in the preparation batch or the LCS/BS will be re-analyzed, or re-prepared and re-analyzed for that analyte. In some cases, results can be reported with a flag (e.g., inadequate sample for re-

preparation or results meet client DQR). No adjustment of client sample results based on LCS recovery is made in the laboratory report.

11.3.1.4 Matrix Spike

The matrix spike is used to monitor method performance in a specific sample matrix. Recovery of matrix spike is a measure of the accuracy in the measurement of the target analyte(s) introduced in the client sample matrix. The matrix spike is an aliquot of client 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
- 1 to 5 times the EQL.

Otherwise, spiking should be performed at a level equivalent to that of the sample or at least 25% of that of the sample. When the concentration of the analyte(s) in the original sample is greater than 0.1%, no matrix spike is required unless specified by client.

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

- Regulatory requirement (e.g., SW-846)
- The client via DQR for a particular project or program
- Laboratory performance over time.

For compounds of interest or matrices not covered by SW-846 or other standard methods, acceptable criteria shall be established based on laboratory performance over time.

The recommended recovery range of matrix spike is 70 to 130%. Control limits are not applicable to spikes that are <25% of the analyte concentration in the sample. In those instances where the sample concentration significantly exceeds the amount of spike added in the prepared samples, the data must be further evaluated to determine if the recovery of the spiked sample is meaningful.

If the matrix spike recovery fails to meet the criteria, the batch results shall be investigated for sources of error. Re-preparation and/or re-analysis should be conducted as necessary based on the DQO or DQR. Spike recoveries outside established control limits will be flagged and/or explained in the narrative or in a LIMS comment section. Minimum spike frequency will be 5%, one per matrix if less than 20 samples are analyzed per matrix or as requested by client.

11.3.1.5 Laboratory Sample Duplicate or Matrix Spike Duplicate

Laboratory sample duplicates are used to assess the precision of the preparation and analytical process in a client-specific matrix. Laboratory duplicates are two aliquots of the same

sample (intra-laboratory split) that are taken through the entire sample preparation and analytical process. Matrix spike duplicates (MSD) are two spiked aliquots of the same sample that are taken through the entire sample preparation and analytical process. In cases where the sample is not expected to contain reasonable concentrations (greater than 10X the instrument detection limit) of the analyte(s) of interest, MSD are used.

One set of laboratory duplicates (or MS/MSD) is required for each batch of samples. Precision is estimated by calculating the relative percent difference (RPD) of the duplicate analysis. Additional replicates may be performed if requested by the client.

Organic duplicate RPD criteria vary widely according to analyte and method. Acceptance criteria are established by a regulatory requirement, client-defined criteria detailed in project or program guidance documents, or lab performance over time for similar matrices and concentration ranges. If the RPD falls outside the established limits, the results are flagged and explained in the narrative or a LIMS comment section. It does not automatically trigger re-analysis.

11.3.1.6 Surrogate

A surrogate is a compound or analyte that is added to all samples (both client samples and QC samples) prior to 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 are generally specific to the method and compounds being detected. Each method that uses surrogates shall specify instructions for surrogate introduction and use. Surrogate control is demonstrated when they are within established control limits. Control limits are established by one of the following: 1) regulatory requirements, 2) laboratory performance over time, or 3) client-defined criteria via DQR for a particular project or program

Surrogate recoveries are normally reported as is measured (that is, no sample recovery corrections are performed based on surrogate recovery). If the surrogate recovery fails to meet criteria, the batch shall be investigated for sources of error. Re-preparation and/or re-analysis should be conducted as necessary based on DQO or DQR.

11.3.1.7 Practical Control Sample

The PCS are specific samples used to monitor analytical performance to client specified action limit. The PCS is only requested by the client and prepared as a blank spike to be used as an internal check sample. For reporting purpose, the PCS is assigned a sample number and treated as client's sample.

11.3.2 Organic Analytical Quality Control

11.3.2.1 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. Each analytical sequence has associated with it a

prescribed number and type of QC standards that are analyzed in a prescribed order. The analytical sequence is an important aspect of the analytical work performed because it allows the analyst and subsequent data reviewers to determine if there are trends in sample results or QC related to the order in which samples were analyzed. Therefore, the order in which samples are analyzed will be traceable to the analytical sequence.

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. The run ends based upon continuing calibration performance for total organic carbon analysis or gas chromatography (GC) or based on analytical clock expiration for gas chromatography mass spectrometry (GC/MS).

11.3.2.2 Initial Calibration Verification (ICV)

The ICV is used to confirm the acceptability of the most recent calibration and the standards used to prepare the calibration. Standards for ICV are prepared from a source other than 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 systems for which calibration applies over an extended period of time (that is, 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).

Acceptance criteria will be stated in the procedure as defined by the specific methods, for example, *Methods for Evaluating Solid Waste: Physical/Chemical Methods* (SW-846). Failure of the initial calibration verification indicates instrument and/or standard problems that shall be evaluated and corrected before any samples are processed for the analytes of interest.

11.3.2.3 Continuing Calibration Verification (CCV)

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. The ICV may be used as the CCV.

CCV and tune are limited to one per analytical sequence for GC/MS. Analytical sequence for GC/MS is limited by a 12-hour period that cannot be exceeded. The analytical sequence starts by a run of tune followed by CCV and samples and ends within this 12-hour period. 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 throughout the runs duration and based

on specific methods (e.g., approximately every 10 samples for GC per SW-846, every 15 samples for total organic carbon analysis).

Failure indicates that the analytical system has drifted out-of-control and requires correction for the analytes of interest. If CCV failure occurs, all samples analyzed after the last acceptable CCV shall be re-analyzed. 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.

11.3.2.4 Continuing Calibration Blanks (CCB)

Continuing calibration blanks (i.e. instrument blanks) monitor effects such as contamination and instrument drift. The CCB is a reagent blank prepared similarly to the standards but not subjected to sample preparation except for techniques in which the preparation is an integral part of the analysis. In these cases, the preparation blank or method blank can be considered the equivalent of CCB. The CCB can be accomplished by an instrument check blank.

Several organic compounds are more readily introduced into blanks. In the case of GC/MS, the blank is typically the equivalent of 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 following each CCV.

The acceptance criteria for these blanks are that they are less than or equal to EQL for each analyte tested. When an analyte exceeds these criteria, potential impact on data quality shall be investigated and re-analyzed when sample carry-over is present. Samples with target analyte concentrations exceeding the blank contamination by a factor of 20 or more can be reported without a B qualifier unless client requirements dictate otherwise.

11.3.2.5 Internal Standards

An internal standard (IS) is an analyte that is similar to the analyte(s) of interest in chemical composition and analytical responses but which is not normally expected to be found in the sample. When internal standards are used, internal standards are added to every standard, blank, sample, QC samples (e.g., MS, MSD) before analysis. Internal standards are used as the basis for quantitation of the analytes of interest.

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 acceptance criteria.

Internal standards are used in organic GC/MS analysis although they may be appropriate to other types of analysis. Generally, area counts falling <50% or >150% of original area counts in the CCV standard are considered unacceptable.

11.3.2.6 Low-Level Standard

The low-level standard is used to monitor instrument performance in the region at or just above the EQL. When the low-level standard concentration is included in the instrument calibration, a separate low-level standard is not required. Low-level standards are not required for total organic carbon analysis.

11.4 RADIOCHEMICAL QUALITY CONTROL

11.4.1 Preparative Quality Control

Preparative techniques are used to prepare a sample for analysis. This may include evaporation, leaching, wet ashing, fusion, digestion, dissolution, and/or extraction of a sample. Separation and/or isolation are also considered preparative if performed before analysis. Preparative QC includes preparation blank (method blank), LCS, duplicate (or matrix spike duplicate), and matrix spike. Preparation QCs are prepared with each batch of samples.

The preparative QC samples described below are not applicable for those radiochemical samples that undergo non-destructive analysis, which cannot be duplicated and do not require any preparation work (dilution or concentration).

11.4.1.1 Batch

A preparative batch is a group of samples with similar matrix, prepared and/or separated at the same time. A batch does not exceed 20 samples (not including QC samples). More than 20 samples can be prepared at the same time, if the required numbers of QC samples are performed for each batch. If clients do not specify project-specific QC, similar matrix type of samples are combined up to 20 samples for preparation batch with one duplicate (or matrix spike duplicate) and matrix spike. In the case of process testing or unique client requirement, QC elements such as a sample duplicate (or matrix spike duplicate) and matrix spike in the preparation batch can be performed per specified period or for every 20 client-specific samples received. However, the preparation blank and laboratory control sample/blank spike requirement would apply to each batch of samples.

A sample or series of samples, which do not require preparation or separation prior to analysis, would not fall under the requirements in this section. Simple dilution is not considered a sample preparation technique. Required QC differs according to the radiochemical analyses requested; therefore, a batch may contain different combinations of the QC samples represented in this section.

Additionally, limited sample quantity may limit the laboratory's ability to meet the duplicate and matrix spike requirements. In such cases, alternative approaches should be considered to demonstrate sample precision and accuracy.

11.4.1.2 Preparation Blanks

The sample preparation blank or method blank is used to monitor contamination resulting from the sample preparation process. The preparation blank is generated by using reagent water or a material that is free of contamination from isotope(s) of interest and is similar to the sample matrix. 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 shall be prepared as required by the analytical procedures and with each batch of samples processed at the same time.

Preparation or method blank acceptability shall be demonstrated by the activity of the target isotope(s) in the blank: 1) less than the estimated minimum detectable activity (MDA) of the associated samples, or, if the blank is equal to or above the MDA, 2) it shall not exceed 5% of the measured activity present in the associated samples, or when a decision level is specified, 3) it shall not exceed 5% of the decision level unless 2) above applies. For low-level activity, method blank shall meet as follows: 1) less than or equal to the estimated MDA of the associated samples, or (2) less than the customer requested detection limit.

If the preparation blank (method blank) fails to meet the acceptance criteria, all affected samples in the preparation batch will be re-counted, re-prepared and re-counted, or results will be reported with an appropriate flag. Sample results do not subtract preparation blank or method blank unless required by the client. In which case, blank subtraction shall be noted in the report.

11.4.1.3 Laboratory Control Sample

The LCS (either an LCS prepared from a suitable matrix or a BS) is used to monitor the effectiveness of the sample preparation process. The isotope of interest of known activity is added to a suitable matrix and carried throughout the analysis. The BS is reagent water or other suitable substrate spiked with the isotope(s) of interest.

LCS control is demonstrated by isotopes being within established control or tolerance limits. Control limits are established by one of the following:

- Administrative, client DQR/DQO, or specified by regulatory requirement
- Provided by the vendor. Vendor supplied precision may effect the tolerance limits applied to the isotope
- Statistically determined by multiple analysis over time.

When one of the acceptance criteria has not been met for an isotope of interest, samples in the preparation batch or the LCS/BS shall be re-counted, or re-prepared and re-counted for that isotope. In some cases, results can be reported with a flag (e.g., inadequate sample for re-preparation or results meet client DQR). No adjustment of the client sample results based on LCS recovery is made in the laboratory report.

11.4.1.4 Matrix Spike

The matrix spike is used to monitor method performance in a specific sample matrix. The matrix spike is an aliquot of sample spiked with the isotope of interest and processed through the entire sample preparation and analytical process similarly to the original sample. For radiochemistry, matrix spike may be added to a sample aliquot prior to any sample preparation e.g., fusion, leaching. Alternatively, spiking may be performed after preliminary sample preparation and dilution, but before any radiochemical separation.

When the isotope activity in the sample is unknown, spiking is typically performed at a level that is one of the following: 1) equivalent to the threshold established by the DQO process; 2) specified by the method; or 3) 1 to 5 times the MDA. Otherwise spiking should be performed at a level equivalent to 1 to 5 times that of the sample or at least 25% of that of the sample.

Matrix spike control is demonstrated when isotope(s) are within established control limits. Control limits are established by one of the following:

- Regulatory requirement
- Established by the DQO or DQR process for a particular project or program
- Laboratory performance over time.

The recommended recovery range for matrix spike analysis is 75 to 125%. 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/or explained in the narrative, or in a LIMS comment section, not necessarily generating an automatic reanalysis.. Minimum spike frequency will be 5%, one per matrix if less than 20 samples are analyzed per matrix unless tracer or carrier is used or as requested by client.

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, tracer, carrier, or a combination of a matrix spike with a tracer or carrier; 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.

Due to the screening nature of Gross Alpha and Gross Beta analyses, matrix spikes, matrix spike duplicates, carriers and tracers are not typically performed. Batch QC consists of a blank, LCS, and duplicate.

WSCF Radiochemical techniques typically employ the use of tracers and carriers. Whenever an isotope does not have a tracer, such as tritium, the batch QC requirements are a blank, LCS, matrix spike and laboratory sample duplicate.

11.4.1.5 Laboratory Sample Duplicate or Matrix Spike Duplicate

Laboratory duplicates are used to assess the precision of the preparation and counting process in a client-specific matrix. The degree of agreement between duplicates indicates the reproducibility (precision) of the combined preparation/separation, and measurement process. Laboratory duplicates are two aliquots of the same sample (intralaboratory splits) that are taken through the entire sample preparation and analytical process. Minimum frequency for sample duplicates will be 5% or one per matrix type.

A radiochemical duplicate RPD criteria of 20% can only be applied to an analyte activity which has an uncertainty (1 sigma) less than or equal to 15%. When either the sample or duplicate uncertainty exceeds 15% the data shall be evaluated based on statistical comparability (see HASQARD, Volume 4, Section 7). In cases where the RPD criteria are not met, evaluation of the source of error and impact on client DQRs shall be performed. If the RPD falls outside the established limits, the results are flagged and explained in the narrative or in a LIMS comment section. It does not automatically trigger re-analysis.

11.4.1.6 Tracer

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 or chemically similar element that is different from that of the isotope or element of interest but expected to behave similarly. A tracer is used to calculate radiochemical yield in a specific sample. The amount of tracer recovered through a method reflects effectiveness of the radiochemical separation for the target isotope. Criteria for recovery of tracers shall be specified in each method. Sample results are corrected based on yield recovered on a tracer.

A tracer may be added to an aliquot of prepared (e.g., leached), or diluted sample prior to specific radiochemical manipulations (e.g., separations). The decision on when to spike a sample with tracer is based on the expected isotope activity in the sample. Sufficient tracer must be used to maintain counting statistics to support client DQO needs.

High yields with radiochemical tracers are not always of great importance (e.g., it is common to sacrifice yield to achieve a better separation). Counting statistics for the tracer shall be evaluated before applying a yield correction to make yield correction meaningful.

11.4.1.7 Carrier

A carrier may or may not be 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 or element that is expected to behave similarly to the isotope or element 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. The influence of a carrier on counting of the target isotope (e.g., mass attenuation of beta counting) must be addressed. As with a tracer, the carrier recovery is a measure of the amount of isotope lost in performing the procedure. Sample results are not normally corrected based on the yield recovered on a carrier.

The carrier may be added to an aliquot of prepared (e.g., leached) sample prior to specific radiochemical manipulations (e.g., separations). The decision on when to spike a sample (e.g., before or after digestion) with carrier is based on the expected isotope activity in the sample.

High yields in radiochemistry are not always important (e.g., it is common to sacrifice yield to achieve a better separation). Before applying a yield correction, evaluation of whether or not sufficient measurable mass was achieved to make yield correction meaningful should be made.

11.4.2 Radioanalytical Techniques Quality Control

Counting Sequence - An analytical run or sequence is defined as those samples counted on any specific detector in a period of time between counter control counts. The analytical run starts after the counter control source is counted and ends when the following counter control source is counted. The sequence of samples counted on a detector where the detector face is directly exposed to the sample should be traceable via instrument printout.

11.4.2.1 Verification of Calibration

The calibration verification confirms the acceptability of the calibration. The calibration verification demonstrates that both standards used and the calibration are accurate. Calibration verification shall be performed before commencement of sample analysis. Calibration of counting instrumentation used in support of radiochemical measurements often applies over extended periods of time. One calibration verification is performed daily prior to use, when the detector is in service. Instrument stability and thus calibration stability, is monitored by counter control standards. The concept of calibration verification is accomplished by using one of the four methods: independent standards, use of independent measurements, multiple calibration curves, or data analysis.

11.4.2.2 Counter Control Standard (Continuing Calibration Verification)

The counter control standard is used to monitor instrument stability over time. Acceptable performance demonstrates the continued accuracy of the calibration, indicating that the measurement system is still in control. The counter control standard may be prepared from any reliable source. Acceptable performance is demonstrated when the concentrations/activities measured fall within established control limits.

Each counting sequence shall be followed by an acceptable counter control during the next counting sequence. If a detector system is found to be out of control, corrective action must be applied 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 counter control standard or QC sample shall be considered suspect and investigated.

The counter control standard verification should provide adequate counting statistics over the time period for which the source is to be counted. However, the source shall not be so radioactive as to cause 1) pulse pileups, 2) dead time that is significantly different from that to be expected from routine samples, or 3) gain shift in the case of pulse height analyzer systems.

11.4.2.3 Background Counts

Most radiochemical techniques use instrument background count measurements. 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, alpha spectroscopy, and liquid scintillation 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 and subtracts with daily monitoring to determine acceptability. Radiochemical background counts are similar to that of the initial calibration blank.

11.5 SUMMARY OF METHOD QUALITY CONTROL

Detailed QC requirements have been described in this section including both client QC (e.g., RPD for duplicate of samples, matrix spike recovery) and laboratory analytical QC (e.g., preparation blank, LCS, analytical QC). Information for acceptance limits, frequency, and corrective action are provided in the SOW, LOI (for non-routine or unique situation), regulatory methods, laboratory QA plan, or in the analytical procedures.

Accuracy and precision trends for analytical methods are monitored by control charting of the LCS. Percent recovery of laboratory standards is used as a measure of method accuracy. Standard deviation of percent recovery of laboratory standards is used as a measure of precision of the method.

11.5.1 Control Chart of LCS

The control chart can be prepared by plotting the control limits symmetrically around the true value (if the mean value is not statistically significant from the true value) or around percent recovery of measured value to the true value (Kirchmer 1994).

Control chart will be continually updated as additional results are obtained. The original limits when not enough data have been accumulated (e.g., new method) can be established by administratively, or by regulatory method.

When a minimum of 20 points are accumulated, the control limits can be calculated statistically, where s_d is the standard deviation of standard percent recovery over a period of time.

- Warning limits: $\pm 2\ s_d$
- Control limits: $\pm 3\ s_d$

11.5.2 Control Limits for the Control Chart

Analysts or their delegates are responsible to enter the QC results (i.e., LCS recovery) to the database (e.g., LABCORE or personnel computer). Control limits are established by one of the following methods:

- Statistical control limits calculated by LABCORE, instrument, or personnel computer software, or
- Regulatory limits, or
- Administrative limits.

The regulatory limits can be plotted along with statistically calculated warning and control limits for comparison.

11.5.3 Use of Control Chart

Analyst is responsible to select other QC parameters to construct control chart (e.g., duplicate, spike recovery) when appropriate. Individual outlying QC results can be flagged by the LABCORE when the results are entered to data management system (e.g., LABCORE). Because individual results are monitored by the analyst and/or by the LABCORE, control chart is used for trend analysis to monitor long-term method performance. Use of control chart is recommended in the following based on the analytical control charting (Kirchmer 1994).

When a single value exceeds the "warning" limit but not the "action limit," an immediate action is not necessary. Due to random error, one in 20 values would be expected to exceed the "warning" limits.

If the results fall outside the warning limits too frequently, this may indicate:

- A systematic tendency for results to be higher or lower (provided that only the upper or lower warning limit, respectively, has been crossed); and
- The random error has increased (provided that both warning limits are crossed).

When the LCS falls outside the acceptable limits, the analyst shall investigate to identify any problems. Proper corrective actions and how to report results are outlined below.

When the first run of LCS fails to meet the acceptance limits:

- If the problem can be identified and corrected, the analyst shall re-analyze the LCS and associated batch samples. When the identified problem is associated with systematic error(s) (e.g., dilution error, pipette error, calculation error), the percent of LCS recovery from the first run shall not be used for calculating control limits. In addition, the first run of samples associated with the same batch of the first LCS shall not be reported with some exceptions (see below).

- If the problems are not obvious and could not be identified, the analyst usually re-runs the LCS without running the samples until the acceptable LCS can be achieved. If the second-run of LCS passes the acceptance criteria, both LCS results should be included/tracked in LABCORE or in a notebook and should be used for calculating control limits with appropriate comments. Samples associated with the same batch as the LCS should be re-run.

When the third run of LCS fails to meet the acceptance limits:

- If the third-run of LCS still does not pass the acceptance criteria, the analyst needs to investigate the analytical system e.g., preparation problem, change to a new batch of standards or change to a new batch of reagent. The client's samples shall not be reported until the LCS performance is back to within the acceptable limits.

There are some exception situations listed below that do not need to or cannot follow the above requirements. Results will be reported under evaluation by the chemists and management for these exception situations with the proper documentation (e.g., case narrative, letter of instruction).

- For measuring multiple analytes per measurement, analytes in the LCS that are not requested by the client do not require a re-run if they could not meet the QC acceptable criteria

- Complexity of the sample matrix

- Non-quantitative analysis requested by the client

- Insufficient samples for further re-analysis.

11.6 INSTRUMENT FAILURE

The following steps are followed when the instrument failure (e.g., hardware or software problem) occurs.

- The cognizant chemist is responsible for issuing or declaring the instrument is non-functioning and out of service.

- The cognizant chemist is responsible for recording instrument failure in the instrument notebook e.g., date, name of the instrument, initial, and probable cause of the failure and posts an appropriate sign on the instrument.

- The chemist removes the "sign" from the instrument when the problem is fixed, enters the date that instrument is back in service in the instrument notebook, and provides a summary of the problem and repair.

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12.0 PROCEDURES TO ASSESS DATA QUALITY

This section provides various formulas that are typically used to compute QC parameters that are used to assess data quality. Various quality control parameters are monitored according to Section 11.0.

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 MDL. The precision of test results is expressed as the RSD or 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:

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

where:

SSR = Spiked sample result
SR = Sample result. Assign the value as zero when the result is less than MDL
SA = Spike added or tracer and/or carrier added.

12.2.2 Method Accuracy Based on Standard

The accuracy of an analytical method is expressed as the percent of recovery (%R) of a standard (e.g. LCS, BS). 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.

12.2.3 Yield Recovery (Radiochemistry Only)

Yield percent recovery (%Y) of a tracer or carrier in radiochemical analysis is a measure of the effectiveness of separation methods for some radionuclides. It is expressed as the percent recovery and is generally used to correct the analyte recovery in the sample for radiochemical analysis. Yield percent recovery is calculated according to the following equation:

$$\%Y = \frac{T_m}{T_k} * 100$$

where:

T_m = Measured value of the tracer or carrier
T_k = Known value of the tracer or carrier.

Yield percent recovery should be evaluated per procedure to monitor the effectiveness of the radionuclide separation. If tracer or carrier is not used on every sample, a historic yield percent recovery should be used as the correction factor for the sample analyte.

12.3 MEASURES OF AGREEMENT

12.3.1 Percent Difference

The percent difference (%D) is often used to compare one reference point to another (for example, 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 (for example, spiked sample)
X_u = Miscellaneous contribution (for example, sample contribution)
K = Known value (for example, true spiked value).

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 136, Appendix B) 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
- The region of the standard curve where there is a significant change in sensitivity (that is, 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:

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

where:

X_i = With measurement of the variable X

$\overline{X}$ = 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 (square root of variance) obtained from the MDL replicate measurements.

The MDL is determined when the method is initially qualified and re-determined whenever, in the judgment of the chemist, a change in analytical performance caused by either a change in instrument hardware or operating conditions has occurred. Spiked reagent water will be used when performing MDL studies for aqueous samples. Spiked solids or solid based standards will be used when performing MLD studies for soil or solid samples. Performing MDL in multiple matrices is not required when MDL is matrix independent.

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

- 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 using MDL calculations. 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 concentration of an analyte that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The laboratory uses the following principles to establish EQL. The analyte concentration at the estimated quantitation limit is determined either by (1) approximately 5 to 10 X MDL or IDL (it may be nominally chosen within these guidelines to simplify data reporting), or (2) the lowest non-zero standard in the calibration curve. EQL reported with client data are reflected in all method dilution factors. Results falling between IDL (or MDL) and the EQL should be reported with appropriate qualification (e.g., flag with a "j").

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 according to procedure LA-508-002, *Detection Limits and Uncertainty Calculations for Radioisotopic Counting* (Jones 1995).

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. It must be recognized that the background may and normally is affected by the components of the sample. Therefore, the MDA value is *a priori* estimate for comparative purposes. The MDA is calculated as detection limit defined in the procedure LA-508-002, *Detection Limits and Uncertainty Calculations for Radioisotopic Counting* (Jones 1995) divided by efficiency (e.g., count rate/disintegration rate).

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 variability's as shown in the following equation. The component of uncertainty has to be expressed in the same unit designation (for example, concentration percentage).

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

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 ASSESSMENTS

The effectiveness of the WSCF's QA program is monitored by the use of management system assessments, technical systems assessments, external assessments, data quality assessments, and performance assessments. Deficiencies resulting from these assessments shall be documented. Corrective actions are initiated by laboratory management. These corrective actions will be followed to completion, filed, and maintained by Corrective Action Management (CAM) organization with processing assistance from the DEG. Laboratory managers review the assessments and monitor progress on the corrective actions.

13.1 MANAGEMENT SYSTEMS ASSESSMENTS

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

- Systems for developing technical procedures
- Quality and applicability of current management systems (for example, QA manual, administrative procedures)
- Procedures for the design and conduct of audits/assessments
- Systems for tracking quality adherence (that is, performance indicators)
- The degree of management support as well as current roles and responsibilities.
- Effectiveness of quality records and documentation control.

The WSCF management is responsible for initiating corrective actions in response to the issues identified during the assessment. The deficiency tracking system (DTS) is responsible for ensuring corrective actions are completed and documented. The QA/QC Officer is responsible for CA oversight.

13.2 TECHNICAL SYSTEMS ASSESSMENTS

Technical system assessments consist of inspections, interviews, reviews, and/or evaluations that assess the laboratory's ability to meet client, program, and/or regulatory requirements. Topics in the technical systems assessments include, but are not limited to, adherence to laboratory procedures, performance of analytical procedure, control of standards, calibration, and/or document control. Technical system assessments are performed periodically in accordance to ASP-200, Section 1.2 and should vary, such that over time critical elements are evaluated. Technical system assessments can be performed either by the QA officer or analytical chemistry staff that are independent from the assessed process/procedure. Each system assessment focuses on a particular aspect of analytical processes.

13.3 EXTERNAL ASSESSMENTS

External assessments are performed by agencies or groups that are not under the control of laboratory management. Agency examples are the Department of Ecology performing an audit before granting accreditation or the Department of Health performing an audit of the Radiochemistry lab for analysis supporting radionuclide air emissions monitoring. External groups within PHMC such as the Facility Evaluation Board (FEB), PA, or WSCF clients may also perform assessments of WSCF.

Laboratory management shall be responsible for initiating corrective actions that are required as a result of external assessments. External assessments are controlled and tracked in DTS.

13.4 DATA QUALITY ASSESSMENTS

Data quality assessments are performed either by the QA officer or by representative from PA. Data reports/packages are assessed against client data quality requirements, at a minimum of 5%. Client and/or project can also direct frequency of data quality assessment on their data reports/packages via SOW, LOI, or SAP. Data quality assessment can also be performed via method assessment.

Comments on data reports/packages assessment are documented in the review comment form. Deficiencies and corrective actions are documented in the data review comment form and maintained on file. Some issues that can be used to improve data packages but do not have significant impact on the data quality will be resolved through the continuous quality improvement process.

13.5 PERFORMANCE EVALUATION ASSESSMENTS

Performance assessments consist of laboratory analyses of standard materials that are used to evaluate analyst and method proficiency. Performance evaluation samples are used as an independent means to assess laboratory performance. Performance evaluation samples can also be used to validate analytical methods. If there are no suitable or relevant external PE samples available, then one of the following three options is acceptable: participation in an alternate PE program, performance of internal performance evaluation (e.g., single or double blind samples when economically practical), or repeatability studies.

It is understood that PE samples may not be completely compatible with the sample matrices and analytical procedures used at WSCF. In these cases, the laboratory may perform the analyses according to the instructions provided by the PE program.

Performance evaluation results shall be reported according to specific instructions provided by the PE programs prior to submission of the results to the QA officer. The QA officer or designator coordinates the procurement of PE or blind samples and their submission to the laboratory, reporting of the PE results, distribution to pertinent laboratory staff of the PE reports provided by the PE sponsor, and coordination of the corrective action process for "unacceptable" PE results.

The causes of "unacceptable or outlying" PE results need to be investigated and appropriate corrective actions initiated. If the causes result in negative impacts upon sample data, a notification to client services is necessary to determine a need to inform laboratory clients. Any identified corrective actions will be tracked in DTS and quality improvement actions will be tracked in CATRAX. Corrective actions may include re-analyze the sample if no apparent cause can be identified; analyze another PE sample, correct analytical process, or a combination of these options.

The WSCF participates in the following environmental performance evaluation programs.

- U.S. Environmental Protection Agency (EPA) Environmental Monitoring Systems Laboratory-Cincinnati Semi-Annual Inorganic/Water Pollution Performance Evaluation Studies (no longer supplied by EPA, WSCF purchases this program semi-annually from Environmental Resource Associates [ERA])
- DOE Environmental Measurements Laboratory - New York Semi-Annual Radiological Quality Assessment Program
- DOE Radiological and Environmental Sciences Laboratory - Idaho Falls Mixed Analytes Performance Evaluation Program (MAPEP)
- National Institute of Standards and Technology (NIST) - NIST Radiochemistry Intercomparison Program (NRIP) when appropriate
- Laboratory QA Blind Study [e.g. ERA, Analytical Products Group, Inc. (APG) as determined] as appropriate.

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14.0 PREVENTIVE MAINTENANCE

The concept of preventive maintenance is to perform local maintenance on instruments until such time when a contract vendor is needed to provide timely repair. Instrument preventive maintenance consists of routine inspections or instrument maintenance.

Routine inspections are inspections performed on the instrument by the responsible scientist/analyst before instrument operations. The frequency of these inspections is established based upon manufacturer's recommendations.

Instrument maintenance shall be performed by qualified personnel and documented. This action may be provided through an external maintenance contract.

Most laboratory instruments require some minor maintenance activities such as cleaning or other adjustments performed by the analyst as part of its setup or operation. The chemist/scientist can/may consult the instrument manuals for the manufacturer guidelines regarding daily setup and operation.

Schedules and maintenance may be managed through JCS. Instrument maintenance schedules are defined in the JCS system or in the instrument notebook. Schedule activities may be grouped to reduce reporting entries. Completion of the action may be documented with a single statement, or if a table is used a single check mark, indicating the activity is complete.

Each instrument shall have a notebook or other permanent document to record maintenance events with date and name of the personnel performing the maintenance. In addition, instrument failure shall be recorded in the notebook (see subsection 11.6 for instruction).

The WSCF has open requisitions for major instrument vendors. In the event of instrument failure, vendor services are immediately solicited. The vendor brings any necessary equipment and supplies thereby relieving the WSCF of the need of a spare parts inventory. Only daily consumables are stored at the WSCF.

The WSCF maintains redundant analytical capability for most analytical areas. This allows the laboratory to continue to operate while instrumentation is being repaired. Also, contract commercial laboratories and the 222-S Laboratory can provide backup analytical services to meet customer deadlines, if necessary.

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15.0 CORRECTIVE ACTION AND QUALITY IMPROVEMENT

The corrective action process consists of the identification of an adverse condition or deficiency, evaluation of the condition, determination and implementation of corrective action, and documentation of the process. When fully implemented, this process is the basis for continuous quality improvement. Corrective action management process and continuous quality improvement at WSCF are performed in accordance to HNF-PRO-052.

Quality improvement is a continuous process and is designed to reduce the variability of every process that influences the quality of the product. As described throughout this document, laboratory activities, analytical measurements and results, and QA/QC activities are documented and are traceable for evaluation. Processes to identify continuous quality improvement includes evaluation of quality-related information and/or reports generated from various processes (e.g., performance data, lessons learned) or from assessments (e.g., inspections, data quality assessments, technical system assessments).

Laboratory personnel at all levels are responsible for initiating correction/corrective actions when conditions have negative impact on laboratory systems (e.g., administrative, analytical, operations). A graded approach is used. Correction/corrective actions and proper documentation depend on the magnitude or significance of quality problem(s).

The corrective action process includes the following: (1) determining the significance of quality problems, and (2) taking effective corrective action based on the potential impact on the data quality. Preventive action will be initiated, as appropriate, considering the magnitude of potential problems. Preventive action also can be part of the correction actions. Implementation of corrective action shall be verified by the QA officer and/or appropriate management.

Significant conditions adverse to quality are defined as a condition, which, if uncorrected, could have a serious effect on safety, operability, or product quality.

Significant conditions adverse to quality may include:

- Conditions that are not corrected in a timely manner.
- Conditions where prior corrective action has not been effective.
- Recurrent or continuing conditions based on reviews and analyses.
- Conditions which, if not immediately corrected, would result in acceptance of work being withheld; a stop work request; or a major adverse impact on the environment, health and safety, mission, cost, or reputation of WSCF or the WSCF client.

Level of corrective action for an adverse condition is determined based on significance of the adverse conditions. The following considerations, when applicable, shall be used to determine the level of corrective action:

- Determine the events leading to the adverse condition

- Determine 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
- Determine if stopping the work associated with the activity is necessary.

15.1 DISCREPANCY REPORT AND PROBLEM REPORT

There are two types of reports to document corrective actions: discrepancy reports and problem reports (see Appendices A & B). The discrepancy report is used to document an isolated condition that the condition is recognized and corrected during an activity or process before affecting data quality or safety. The problem report is used to document an adverse condition where there is a significantly and negatively impact on reported results on data quality or safety. Problem reports are submitted to Facility Support to process for corrective action in accordance with HNF-PRO-052.

The discrepancy report process is designed to identify discrepancies that are related to analytical laboratory operations. Discrepancy reports (including discrepancy report numbers, corrective actions, and status) are tracked by the QA. The repeated discrepancy that has negative impact on data quality will be elevated to the problem report level.

Examples for conditions where discrepancy reports will be initiated are listed in the following, but not limited to:

- Failure to follow client analytical request and/or DQO
- Misidentification or mishandling of samples
- LCSs remain out of control limits or fail to meet acceptance criteria after the third attempt to correct the problem
- Failure in preparation blank indicating a contamination problem
- Results are inaccurately reported (only apply to isolated cases, or the error is associated with a known reason)

- Re-analysis of samples due to isolated quality problem(s) that have no impact on other data set or samples.

Problem Reports will be completed upon identification of anomalies requiring investigation and corrective actions. All laboratory personnel at all levels are responsible for initiating corrective actions when conditions may adversely impact laboratory systems (administrative or analytical operations). Examples of conditions where corrective action is initiated are:

- Incorrect sample results/incorrect data reporting that are related to a systematic problem and impact multiple clients
- Customer issues or complaints considered to be severe especially regarding reported data
- Noncompliance with policies, procedures, methods, or documented customer requirements that has significant negative impacts on data quality
- Failures in performance evaluation sample analysis, which leads to an unacceptable rating for that program and has a negative impact on the sample results.
- Repeated deficiency is identified during assessment and can lead to significant conditions
- Control charting issues indicating system problems.

The QA officer is responsible for assigning the problem report number. When there is an impact on data quality, all affected sample numbers shall be listed on the problem report. Data users shall be notified as necessary and client contact will be documented on the problem report. Problem reports are tracked by DTS.

15.2 CONTROL OF NONCONFORMANCES

Non-conformance items shall be identified, documented, evaluated, segregated (where practical), disposed in accordance with HNF-PRO-298. Nonconformance Reports (NCRs) will be issued in response to materials, parts, or components that do not conform to procured specifications or requirements in order to prevent their use.

15.3 TREND ANALYSIS

Trend analysis is a tool to be used to identify significant quality trends. Trend analysis is performed periodically using the corrective action database to identify any significant changes. Trends determined to be adverse to quality shall be reported to the organization(s) responsible for corrective action.

15.4 ROOT CAUSE ANALYSIS

During the corrective action management evaluation process, root cause analysis is performed and root cause code is identified in according to HNF-PRO-052. The extent of analysis shall be commensurate with the importance or the significance of the problem (i.e., graded approach).

15.5 CONTINUOUS QUALITY IMPROVEMENT

Quality improvement processes consists of planning to meet client's DQR, and corrective action management processes (HNF-PRO-052). Quality improvement can be achieved through the planning and implementation of client's DQR to prevent problems, reviewing existing performance, detecting and correcting problems, identifying the causes of problems, working to prevent recurrence, documenting corrective actions, and tracking such actions to closure. Quality improvement can also be identified from trending activities.

Quality improvement is an on-going effort performed by personnel at all levels in the laboratory. Problems and deficiencies identified requiring systematic corrective actions shall be brought to the laboratory management or QA officer's attention.

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16.0 QUALITY ASSURANCE REPORTS

The laboratory QA Officer is responsible for reporting to laboratory management the status of the QA Program. The QA officer will provide a QA report (minimum of one per year), which will summarize the following information, when applicable:

- Performance evaluation samples and results
- Regulatory compliance issues
- Technical and management assessments
- External audits, assessments, and surveillance activities
- QA/QC problems and recommended solutions
- Corrective actions and status

Types of report, author, frequency, and distribution of reports generated from WSCF are summarized in Table 16.1.

Table 16.1 Summary of QA Related Reports Generated by WSCF

Reports	Author	Frequency	Distribution	Corrective Responsibility	Type of Document
Discrepancy Report	QA Officer Lab personnel	Ad hoc	QA Officer, Internal	Lab personnel, manager	Internal form
Problem Report	QA Officer Lab personnel	Ad hoc	QA Officer, CAM, DEG	Lab personnel, manager	Internal form
Case Narrative	Technical staff	1/data package	Customer's data package	N/A	Internal form
Management Self-Assessments	Management Staff	Per Assessment Program	ASP Management, Lab Management	Lab Management	Management Assessment Format
Technical Systems Assessments	Auditor, QA staff	Ad hoc	Affected personnel, CAM/DEG	Responsible personnel	e-mail or internal form, general assessment format
QA Reports	QA Officer	Annual	Lab managers	QA Officer, Lab manager	Internal form

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17.0 PROCUREMENT CONTROLS

The WSCF follows the HNF-PRO-123, HNF-PRO-268, HNF-PRO-259, HNF-PRO-186, HNF-PRO-335, HNF-PRO-129, and HNF-PRO-3144 to ensure the procurement process is documented and controlled. The controls ensure procured items and/or services can:

- Conform to established specifications
- Meet acceptable quality
- Perform as expected.

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 (for example, subsequent reporting to the U.S. DOE Office of the Inspector General).

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18.0 REFERENCES

40 CFR 136, Appendix B, 1994, *Definition and Procedure for the Determination of the Method Detection Limit, Rev. 1.11.* U. S. Code of Federal Regulations, Title 40, U.S. Environmental Protection Agency, Washington, D.C.

American Public Health Association, American Water Works Association, and Water Pollution Control Federal, 1989, 1992 *Standard Methods for the Examination of Water and Wastewater (Standard Methods),* American Public Health Association, Washington, D.C.

American Society for Testing Materials (ASTM) methods

Clean Air Act of 1977, 42 USC 7401 et seq.

Clean Water Act of 1977, 33 USC 1251 et seq.

Resource Conservation and Recovery Act of 1976, 42 USC 6901 et seq.

Comprehensive Environmental Response, Compensation, and Liability Act of 1980, 42 USC 9601 et seq.

DOE, 1994, Methods for Evaluating Environmental and Waste Management Samples, DOE/EM-0089T, U.S. Department of Energy Richland Field Office, Richland, Washington

DOE, 1998, *Hanford Analytical Services Quality Assurance Requirements Documents,* DOE/RL-96-68, U.S. Department of Energy Richland Field Office, Richland, Washington.

DOE, 1999, *Quality Assurance,* DOE Order 414.1A, U.S. Department of Energy, Washington, D.C.

EPA, 1980, *Prescribed Procedures for Measurement of Radioactivity in Drinking Water,* Environmental Protection Agency, Cincinnati, OH.

EPA, 1983, *Methods for Chemical Analysis of Water and Wastes,* Environmental Protection Agency, Cincinnati, OH.

EPA, 1999, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846, Third Edition, Updates I, II, IIA, IIB, III, and IIIA, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

Kirchmer, C.J., 1994, *Analytical Control Charting*, Procedural Manual for the Environmental Laboratory Accreditation Program, Washington State Department of Ecology, January 1994, Manchester, WA.

WAC, 1994, Washington Administrative Code 173-50, *Procedural Manual for the Environmental Laboratory Accreditation Program*, Washington Department of Ecology, Manchester, Washington

Other Hanford Documents

HNF-SD-CP-QAPP-019, *Waste Sampling and Characterization Facility Quality Assurance Project Plan for Industrial Hygiene Analyses*

LA-508-002, *Detection Limits and Uncertainty Calculations for Radioisotopic Counting*

LC-708-001, *MULTI LIMS Use in the Laboratory.*

LC-718-001, *Laboratory ADP Operations*

LO-090-400, *Internal Chain of custody at WSCF*

LO-090-403, *Sample Receiving and Custodianship At the Waste Sampling and Characterization Facility*

LO-100-451, *Waste Management in Analytical Process Areas*

LO-100-453, *Package Low-Level and Mixed Solid Laboratory Waste in a 55-Gallon Drum - Waste Sampling and Characterization Facility*

LO-100-454, *Collect, Package, and Ship Low-Level Compactable Waste*

LO-100-455, *Collect and Package Liquid Organic Wastes - WSCF*

LO-100-456, *Package Nonradioactive Hazardous Waste*

LO-100-466, *Manage Satellite Accumulation Areas and 90-Day Accumulation Areas*

LO-100-469, Bulk Waste Scintillation Vials and Fluids

LO-110-400, *Labware Cleaning for the WSCF Laboratory*

LO-120-001, *Labeling of Standards and Reagents by Standards Laboratory Personnel*

LO-120-007, *Proper Labeling of Secondary Containers at 222-S Complex and WSCF*

LO-140-008, *Routine Use and Quality Assurance for Analytical Balances at 222-S and WSCF*

LO-140-410, *Calibration Check of Pipets (under revision)*

LO-140-417, *Development of Instrumentation, Methods, Performance of Process Testing, and Chemical Compatibility Reviews*

LO-150-445, *Monitoring Temperature of Sample Storage Units of the Waste Sampling and Characterization Facility (WSCF)*

LO-200-400, *Radioactive Material Identification and Classification Program at WSCF*

LQ-543-401, *Quality Control Criteria for Sample Preparation Processes for Radiochemical Analysis*

ASP-GD-007, *Senior Director's Office and Functional Support Roles and Responsibilities*
ASP-GD-008, *Waste Sampling and Characterization Facility Roles and Responsibilities*
ASP-GD-009, *222-S Laboratory Roles and Responsibilities*
ASP-GD-010, *Production Control Roles and Responsibilities*
ASP-GD-011, *Analytical Chemistry Roles and Responsibilities*

ASP-200, Fluor Hanford Analytical Services Procedures
- Section 1.2, *Assessment Program*
- Section 1.4, Analytical Services Laboratory Records System
- Section 1.18, *Technical Procedure Process*
- Section 1.21, *Laboratory Configuration Control*
- Section 1.22, *Documentation of Laboratory Quality-Affecting Software*
- Section 1.24, *Analytical Services Technical Procedure Process*
- Section 2.15, *Timely Orders*
- Section 3.1, *Work Management*
- Section 5.4, *Analytical Services On-the-Job Training*

ASP-315, WSCF Administrative Procedures
- Section 1.9, *WSCF Laboratory Issues Management*
- Section 4.20, *WSCF Laboratory Complex Chemical Hygiene Plan*
- Section 6.1, *Management of Satellite Accumulation Areas-WSCF*
- Section 6.2, *Management of 90-Day Accumulation Areas*
- Section 6.3 *Waste Fact Sheet Development and Issuance at WSCF*
- Section 6.4 *WSCF Laboratory Waste Management Program*
- Section 8.2 *WSCF Sample Management*
- Section 9.3, *WSCF Laboratory Data Review*

ASP-GD-02, *Laboratory Numbering System*

HNF-5551, *WSCF Laboratory Complex Dangerous Waste Training Plan*
HNF-MP-599, *Quality Assurance Program Description*

PLN-03-QP-001, *Fluor Hanford Site Operations Quality Assurance Program Plan*

HNF PROs

HNF-PRO-052, *Corrective Action Management*
HNF-PRO-123, *The Material Request/Purchase Requisition/Contract Requisition Process*
HNF-PRO-129, *Controlling Spare Parts Inventory*
HNF-PRO-186, *Preparing A Statement of Work for Services*
HNF-PRO-249, *Training Course Administration, Registration and Records*
HNF-PRO-259, *Graded Approach*
HNF-PRO-268, *Control of Purchased/Acquired Items and Services*
HNF-PRO-298, *Nonconforming Item Reporting and Control*
HNF-PRO-309, *Computer Software Quality Assurance Requirements*
HNF-PRO-335, *Use and Control of Purchasing Card (P-Card)*
HNF-PRO-440, *Document Change Control Not Associated with Construction*

HNF-PRO-490, *Calibration Management Program*
HNP-PRO-3144, *Supplier Quality Assurance Program Evaluation*
HNF-PRO-10588, *Records Management Process*
HNF-PRO-10863, *Notebooks and Logbooks*
HNF-RD-210, *Records Management Program*
HNF-RD-8310, *Document Control Program*

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APPENDIX A SAMPLE OF A WSCF DISCREPANCY REPORT

WSCF DISCREPANCY REPORT	
INITIATOR/DATE	DISCREPANCY REPORT NUMBER
DISCREPANCY (INCLUDING VIOLATED REQUIREMENTS) [ ] Failure to follow client analytical request and/or DQO Type of Instrument:_____ Model Number:_ [ ] Failure in performance evaluation sample analysis [ ] Misidentification or mishandling of samples [ ] Laboratory control samples were out of control limits after the third re-calibration [ ] Failure of Laboratory Preparation Blank indicating a contamination problem [ ] Inaccurate results: only apply for samples within a batch with a known error not systematic error [ ] Re-analysis of sample due to quality problem before reporting the data [ ] Others	
PROBABLE CAUSE	
SAMPLES AFFECTED	CLIENT NOTIFICATION REQUIRED? NO
PROBLEM REPORT (PR) NEEDED? Yes/No	IF PROBLEM REPORT NEEDED, DATE THAT THIS DR IS DISCONTINUED: NO OF THE PR REPLACING?
CORRECTION	
AFFECTED RECORDS CORRECTED? (notebook, worksheet, etc.) YES_ N/A	CORRECTED REPORT _ YES _ NO ISSUED?
Closure Date:	QA Officer Signature:

APPENDIX B SAMPLE OF A WSCF PROBLEM REPORT

PROBLEM REPORT	
INITIATOR/DATE	PROBLEM REPORT NUMBER
PROBLEM (INCLUDING VIOLATED REQUIREMENTS)	
PROBABLE CAUSE	
SAMPLES AFFECTED	CLIENT NOTIFICATION REQUIRED? YES/NO
CORRECTIVE ACTION PLAN (INCLUDING SCHEDULE FOR COMPLETION)	
AFFECTED RECORDS CORRECTED? (notebook, worksheet, etc.) YES_ N/A	CORRECTED REPORT _ YES _ NO ISSUED?
MANAGER OR TEAM LEADER CONCURS (SIGNATURE/DATE)	QA COORDINATOR CONCURS (SIGNATURE/DATE)
PROGRESS REPORT attach additional pages if necessary	
COMPLETION (MANAGER OR TEAM LEADER SIGNATURE/DATE)	PR CLOSURE (QA COORDINATOR SIGNATURE/DATE)

GLOSSARY

Note: Majority of quality related terms are defined throughout the document; therefore, they are not listed in the Glossary. See HASQARD, Volume 1, Appendix A for reference.

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

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.

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

Blank Spike. The blank spike is reagent water or other suitable substrate spiked with the isotope(s) or analyte(s) of interest. A blank spike can be used when an appropriate laboratory control sample is unavailable.

Client. The person or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.

Comparability. Measure of 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 document. 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.

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

Data Quality Objectives (DQO). A strategic systematic process for planning scientific data collection efforts. The DQO process helps investigators answer the following basic questions: Why do we need data? What must the data represent? How will we use the data? and How much uncertainty is tolerable? By using the DQO Process, investigators ensure that the data collected for decision-making are the right type, quantity, and quality.

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

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.

May. Denotes permission but not a requirement.

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; nonfulfillment of a specified requirement.

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 maintenance. 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 activities for planning, implementing, assessing, reporting, and quality improvement efforts to meet user requirements.

Quality Control. The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality.

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

Traceability. A document trail that identifies the history of a sample, standard, or other material.

Valid. Having legal efficacy or force, well grounded or justifiable being at once relevant meaningful logically correct, appropriate to the end in view.

Validation. Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. In design and development, validation concerns the process of examining a product or results to determine conformance to user needs. For data, validation is the process by which the data and quality control information is assessed or compared against the client's requirements.

Verification. Confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, verification concerns the process of examining a result of a given activity to determine conformance to the stated requirements for that activity. For data, verification is the process of comparing the reported data with the required information.

Verifying. To establish the truth, accuracy, or reality.

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