OAR Box 1712

Prepped by Keeia Richards

Document Number:

21) II-A2-12

Docket Number:

A-98-49

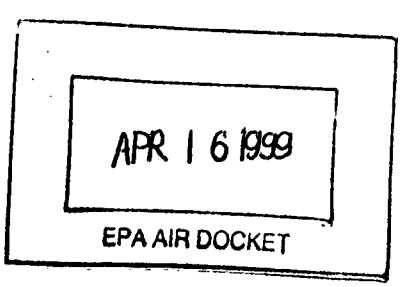
A-98-49
II-A2-12

Document ID: PLN-190
Revision: 3

Lockheed Martin Idaho Technologies Company

IDAHO NATIONAL ENGINEERING & ENVIRONMENTAL LABORATORY

Quality Assurance Project Plan For



The Transuranic Waste Characterization Program

Idaho National Engineering and Environmental Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

PLN-190

Approved By:

T. L. Clements, Jr., Manager
LMITCO TRU Waste Programs Department — Date: 4/5/99

R. E. Arbon
INEEL Site Project Manager — Date: 4/5/99

G. R. Hayes
INEEL Site Quality Assurance Officer — Date: 4/5/99

Manager, CAO Quality Assurance — Date: 4/6/99

Team Leader, National TRU Program — Date: 4/6/99

DOE-ID — Date: 4/5/99

This document has been prepared in accordance with the DOE-CAO Transuranic Waste Characterization Quality Assurance Program Plan CAO-94-1010 Revision 0 dated 4/30/95 with interim changes dated November 1996 and February 1996.

Idaho National Engineering and Environmental Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

PLN-190

Reviewed By:

for T. E. Allen for Sheila Hailey — 4-5-99
Site Data Validation Officer — Date

4/5/99
Analytical Laboratories Department FQAO — Date

4/5/99
Analytical Laboratories Department Manager — Date

4/5/99
RWMC FQAO — Date

C. C. Dwight per telecon — 4/5/99
ANL-W — Date

Idaho National Engineering and Environmental Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

PLN-190

Controlled Distribution List

Controlled distribution shall be done by the Site Project Office.

If you have any questions or need to be on Controlled Distribution, please contact the Site Document Control Officer at (208) 526-2747.

ABSTRACT

This Quality Assurance Project Plan (QAPjP) specifies the quality of data necessary and the characterization techniques employed at the Idaho National Engineering and Environmental Laboratory (INEEL) to meet the requirements of the U.S. Department of Energy Waste Isolation Pilot Plant *Transuranic Waste Characterization Quality Assurance Program Plan* (QAPP) and the associated documents referenced in the QAPP for INEEL contact-handled retrievably stored transuranic waste. This QAPjP is supplemented by five implementation plans that describe the INEEL Transuranic Waste Characterization Program (TWCP) activities applicable to the five participating facilities.

This QAPjP describes the roles and responsibilities of all participants in the TWCP. Data quality objectives and quality assurance objectives are explained. Criteria for selection of waste containers and the parameters that must be characterized are described. Waste container radiography and radioassay procedures, gas and solid/soil sampling procedures, and sample analysis procedures are explained. Associated quality assurance measures are also addressed; these include sample chain-of-custody; data validation, usability and reporting; documentation and records; audits and assessments; field sampling and laboratory quality control samples; and instrument testing, inspection, maintenance, and calibration. Finally, administrative quality control measures, such as document control, control of nonconformances, variances and quality assurance status reporting are described.

QAPjP #PLN-190 EXPLANATION OF CHANGES

Revision 1 dated 4/16/97

This was the initial released version of the QAPjP.

Revision 2 dated 8/31/97

Summary of Changes

Section No.	Description of Change
1.1	Revised Figure 1-1, 1.1.7 & 1.1.13 to clarify independence of the QA functions.
1.6	Added FTIRS personnel to Table 1-6, training and qualification requirements.
6.4	Revised solid/soil sample COC form to more accurately specify requested analyses.
10.3.1	Clarified the requirements pertaining to the RTR independent observation of one scan.
14.2	Revised text and Table 14-1 to include PCB analysis of organic sludges.
15.5	Clarified LCS accuracy requirements (added footnote to Table 15-2).
Numerous	Minor editorial changes.

Revision 3 dated 2/25/99

Summary of Changes

Section No.	Description of Change
1.1	Revised Figure 1-1, Section 1.1.8 and 1.1.9 to clarify SQAO independence and SDVO and SDCO reporting.
2.1.3	Revised to delete variance and add PCN.
2.1.5	Revised management assessment procedure reference.
2.1.6	Revised to add annual SPO QA audit of facilities.
3	Revised to include TRIPS and added reference to SPO TRIPS procedures.
Table 2-2	Added procedures
10	Added CD ROM as video data media. Revised visual examination process.
15	Deleted GFAA method for selenium analysis and CVAA method for mercury.
16	Updated References.

CONTENTS

FIGURES

TABLES

ACRONYMS

ACL	Analytical Chemistry Laboratory
ALARA	as low as reasonably achievable
ALD	Analytical Laboratories Department
ANL-W	Argonne National Laboratory-West
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BFB	4-bromofluorobenzene%C percent complete
CAO	U.S. Department of Energy-Carlsbad Area Office
CCC	calibration check compound
CH TRU	contact handled transuranic
COC	chain-of-custody
CV	coefficient of variation
CVAF	cold vapor atomic fluorescence spectroscopy
%D	percent difference
DFTPP	decafluorotriphenylphosphine
DOE	U.S. Department of Energy
DOE-ID	U.S. Department of Energy-Idaho Operations Office
DOE-CH	U.S. Department of Energy-Chicago Operations Office
DQO	data quality objective
DVS	drum venting system
EB	equipment blank
ECL	Environmental Chemistry Laboratory

EDF	engineering design file
EICP	Extracted Ion Current Profile
EPA	U.S. Environmental Protection Agency
FB	field blank
FD	field duplicate
FQAO	facility quality assurance officer
FRS	field reference standard
FTIRS	fourier transform infrared spectroscopy
FIP(s)	facility implementation plan(s)
Gas PDP Plan	*Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases for the TRU Waste Characterization Program* (DOE 1995b)
GC/FID	gas chromatography/flame ionization detection
GC/MS	gas chromatography/mass spectrometric detection
GC/TCD	gas chromatography/thermal conductivity detection
HFEF	Hot Fuel Examination Facility
HPLC	high performance liquid chromatography
HSS	headspace sampling system
ICAL	initial calibration
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
IDAPA	Idaho Administration Procedures Act
IDC	item description code
IDL	instrument detection limit
INEEL	Idaho National Engineering and Environmental Laboratory
INEL	Idaho National Engineering Laboratory
LMITCO	Lockheed Martin Idaho Technologies Company

M&O	management and operating
MCP	management control procedure
MDC	minimum detectable concentration
MDL	method detection limit
Methods Manual	*Transuranic Waste Characterization Sampling and Analysis Methods Manual* (DOE 1996c)
MPC	matrix parameter category
NCR	nonconformance report
NDA PDP Plan	*Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program* (DOE 1995c)
NEIC	National Enforcement Investigation Center
NIST	National Institute of Standards and Technology
NQA-1	*Quality Assurance Program Requirements for Nuclear Facilities* (ASME 1989)
NQA-2a	*Quality Assurance Program Requirements for Computer Software for Nuclear Facility Applications* (ASME 1990)
OI	operating instruction
OJT	on-the-job training
OSHA	Occupational Safety and Health Administration
OVA	organic vapor analyzer
PAN	passive/active neutron
PDP	Performance Demonstration Program
PLS	partial least squares
PM	project manager
ppmv*m	parts per million by volume times meters
PRC	project records coordinator (at ANL-W)
PRDL	program required detection limit

PRQL	program required quantitation limit
QA	quality assurance
QAO	quality assurance objective
QAPjP	quality assurance project plan
QAPD	*Quality Assurance Program Document* (DOE 1996a)
QAPP	*Transuranic Waste Characterization Quality Assurance Program Plan* (DOE 1996b)
QC	quality control
QPP	quality program plan
r^2	regression coefficient
%R	percent recovery
%RSD	percent relative standard deviation
RA	nondestructive radioassay
RCRA	Resource Conservation and Recovery Act
RFETS	Rocky Flats Environmental Technology Site
RFP	Rocky Flats Plant
RGA	residual gas analysis
RH TRU	remote handled transuranic
RIDS	records inventory and disposition schedule
RPD	relative percent difference
RT	retention time
RRT	relative retention time
RTL	regulatory threshold limit
RTR	real-time radiography
RWMC	Radioactive Waste Management Complex
SDCO	site document control officer

SDVO	site data validation officer
SGRS	high-resolution passive gamma ray spectroscopy
Solid PDP Plan	*Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes* (DOE 1995d)
SOP	standard operating procedure
SPCC	system performance check compound
SPM	site project manager
SPO	site project office
SQAO	site quality assurance officer
SVOC	semivolatile organic compound
SWEPP	Stored Waste Examination Pilot Plant
TC	toxicity characteristic
TIC	tentatively identified compound
TRIPS	Transuranic Reporting and Inventory Processing System
TPR	technical procedure
TRU	transuranic
TRUCON	TRUPACT-II content codes
TRUPACT-II	Transuranic Package Transporter-II
TSA	Transuranic Storage Area
TWBIR	*Waste Isolation Pilot Plant Transuranic Waste Baseline Inventory Report* (DOE 1995e)
TWCP	TRU Waste Characterization Program
UCL_{90}	upper 90% confidence limit
VOC	volatile organic compound
VTSR	validated time of sample receipt
WCA	Waste Characterization Area
WIPP	Waste Isolation Pilot Plant

WIPP WAC	*Waste Acceptance Criteria for the Waste Isolation Pilot Plant* (DOE 1996d)
WSPF	Waste Stream Profile Form
WWIS	WIPP Waste Information System

DEFINITIONS

Accuracy—The degree of agreement between a measured value and an accepted reference or the true value. Accuracy is determined as the percent recovery (%R).

Analyte—The element, ion, or compound an analysis seeks to determine; the element of interest.

Analytical batch—A suite of samples of a similar matrix that is processed as a unit, using the same analytical method, within a specific time period. An analytical batch can be up to 20 samples, (excluding laboratory quality control samples) all of which must be received by the laboratory within 14 days of the validated time of sample receipt of the first sample of the batch.

Audit—A planned and documented independent assessment to determine by investigation, examination, or evaluation of objective evidence, the adequacy of, and compliance with established procedures, instructions, drawings, and other applicable documents, and the effectiveness of implementation. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

Calibration—The establishment of an analytical curve relating instrument response (signal) to analyte amount or concentration.

Chain of Custody (COC)—A set of procedures established to ensure that sample data integrity is maintained.

Comparability—A qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions.

Completeness—The percentage of measurements made that are judged to be valid measurements. The completeness goal is to generate a sufficient amount of valid data based on Program needs.

Data quality objectives (DQOs)—Qualitative and quantitative statements derived from the outputs of the first six steps of the DQO Process (see below). DQOs; 1) clarify the study objective, 2) define the most appropriate type of data to collect, 3) determine the most appropriate conditions from which to collect the data, and 4) specify tolerable limits on decision errors which will be used as the basis for establishing the quantity and quality of data needed to support compliance decisions. DQOs are used to develop a scientific and resource-effective data collection design.

Data reduction—Operations necessary to correct data from the raw form to a final form as required by the customer.

Equipment blanks—Samples of high purity gas or water that are analyzed to determine cleanliness of the sampling equipment. They are collected after the equipment has been cleaned and before sampling. These blanks are useful in documenting adequate cleaning of sampling equipment.

Equipment cleaning batch—A number of sampling equipment items cleaned together at one time using the same cleaning method.

Field blanks—Field blanks are headspace gas background samples that are collected in the field in the immediate vicinity of the sample collection location. They accompany the sample containers through collection, shipment to the analytical laboratory, and storage before analysis, and are used to identify any contamination from field conditions.

Field duplicates—Two separate, independent samples collected from the same source, as close as possible to the same place and time, stored in separate containers, and analyzed independently. Field duplicates are used to document the precision of the sampling and analysis process.

Field reference standards—Standard headspace gas samples containing known concentrations of target analytes. They are used to identify any bias in the sampling process.

Headspace—For any volume contained by a drum, 55-gallon poly bag, or innermost layer of confinement, the total contained volume minus the volume occupied by the waste material. "Headspace" is also used to refer to the gases contained in this volume.

Independent assessment—A quality assurance program assessment that is conducted by an independent group or organization, having authority and freedom from the line organization, to evaluate the scope, status, adequacy, programmatic compliance, and implementation effectiveness of the quality assurance program.

Management assessment—A determination of managerial effectiveness in establishing and implementing quality assurance program plans that conforms to U.S. Department of Energy (DOE) policy requirements. It is based on an analysis of functional appraisals, internal audits, and other information, and on the application of appropriate criteria. It is a review and evaluation of management performance covering all quality assurance and management responsibilities to ensure proper quality assurance program balance.

Management controls—Methods used to ensure that work is performed compliant with applicable regulations and program requirements. Examples of management controls include, but are not limited to, procedures, training, radiological postings, established time limits, and storage practices.

Matrix parameter category—A collection of descriptive titles, definitions, and associated numerical codes used to classify mixed waste at DOE facilities. Matrix parameter categories are defined in the *DOE Waste Treatability Group Guidance* (DOE 1995a).

Nonconformance—A deficiency in program requirements that renders the quality of an item or sample unacceptable or indeterminate. Nonconforming program data are final reported data that do not meet quality assurance objectives.

On-line batch—The number of headspace gas samples that are collected and analyzed within a 12-hour period using the same on-line integrated sampling/analysis system.

Performance assessment—A determination of the long-term performance of the Waste Isolation Pilot Plant (WIPP) disposal system in accordance with the requirements of the U.S. Environmental Protection Agency Standard 40 CFR Part 191, Subparts B and C.

Precision—A measure of mutual agreement among individual measurements of the same property made under prescribed similar conditions; often expressed as a standard deviation or relative percent difference (RPD).

Quality assurance (QA)—All those planned and systematic actions necessary to provide adequate confidence that a facility, structure, system, or component will perform satisfactorily and safely in service. The goals of QA are to ensure that research, development, demonstration, scientific investigations, and production activities are performed in a controlled manner; that components, systems, and processes are designed, developed, constructed, tested, operated, and maintained according to engineering standards, quality practices, and technical specifications/operational safety requirements; and that resulting technology data are valid, defensible, and retrievable. QA includes quality control, which comprises all those actions necessary to control and verify the features and characteristics of a material, process, product, or service to specified requirements.

Quality assurance objectives (QAOs)—The characteristics of data that are associated with their ability to satisfy a given purpose or objective. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

Quality control (QC)—The routine application of procedures for controlling the monitoring process. QC is the responsibility of all those performing the hands-on operations in the field and in the laboratory.

Radioassay (RA)—Assay methods used to identify and quantify radionuclides in transuranic waste.

Radiography—A nondestructive testing method that uses X-rays to inspect and determine the physical form of a waste.

Recovery—The numerical ratio of the amount of analyte measured by the laboratory method divided by the known amount of analyte added to the matrix (i.e., spiked sample) to be analyzed. It is usually expressed as a percent (%R).

Representativeness—The degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that concerns the proper design of the sampling program.

Review/data review—The process used to ensure that the proper collection and reduction of raw data has been accomplished. Data review requirements for the INEEL TWCP are described in Section 3.1.

Sampling batch—A suite of samples of a similar matrix (gas or solid) collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples (excluding field QC samples), all of which must be collected within 14 days of the first sample in the batch.

SUMMA® canister—A stainless-steel pressure vessel with SUMMA® passivated interior surfaces for the collection and storage of gas samples. The SUMMA® passivation process involves the

formation of chromium-nickel oxide on the interior surface of the canister. This type of canister is used for sample storage stability of many specific organic compounds.

Surveillance—The act of monitoring, observing or reviewing to verify whether an item, activity, system or process conforms to requirements. A surveillance generally is less formal and focuses on a more specific work scope than an audit.

Testing batch—A suite of waste containers undergoing radioassay (Section 9) or radiography (Section 10) using the same testing equipment. A testing batch can be up to 20 waste containers without regard to waste matrix.

Transuranic (TRU) waste—Laboratory and process waste that contain alpha-emitting radionuclides of an atomic number greater than 92 (e.g., the radioactive isotopes of plutonium), have half-lives longer than 20 years, and are present in concentrations greater than 100 nanocuries per gram of waste.

Validation—An activity that demonstrates or confirms that a process, item, data set, or service satisfies the requirements defined by the user. Data validation requirements for the INEEL TWCP include signature release and are described in Section 3.1.

Validated time of sample receipt—The date and time on which a sample is received at the analytical facility, as recorded on the chain-of-custody record.

Variance—A measure of the dispersion of a series of results around their average. It is the sum of the squares of the individual deviations from the average of the results, divided by the number of results minus one.

Verification—The act of authenticating or formally asserting the truth that a process, item, data set, or service is, in fact, that which is claimed. Data verification is the process used to confirm that all review and validation procedures have been completed. Data verification requirements for the INEEL TWCP are described in Section 3.1.

Waste stream—Waste material generated from a single process or activity that is similar in material, physical form, and hazardous constituents.

QUALITY ASSURANCE PROJECT PLAN FOR THE TRANSURANIC WASTE CHARACTERIZATION PROGRAM

1. PROGRAM MANAGEMENT

The *Transuranic Waste Characterization Quality Assurance Program Plan* (QAPP) (DOE 1996b) requires each U.S. Department of Energy (DOE) facility participating in the Transuranic (TRU) Waste Characterization Program (TWCP) to develop and implement a quality assurance project plan (QAPjP) that addresses all requirements specified in the QAPP. This QAPjP, developed by the Idaho National Engineering and Environmental Laboratory (INEEL) TWCP Site Project Office (SPO) describes the implementation of QAPP requirements by INEEL facilities. The SPO is part of the TRU Waste Programs Department of Lockheed Martin Idaho Technologies Company (LMITCO). This QAPjP complies with the QAPP requirements and quality requirements of the DOE Carlsbad Area Office (CAO) *Quality Assurance Program Document* (QAPD) (DOE 1998a) and is implemented by facility implementation plans (FIPs) developed by INEEL facilities supporting the TWCP.

1.1 Program Organization

Figure 1-1 depicts the INEEL TWCP functional organization chart. At INEEL, SPO personnel are responsible for overall management of the TWCP and project-level data validation and reporting. The SPO consists of the Site Project Manager (SPM), Site Quality Assurance Officer (SQAO), Site Data Validation Officer (SDVO), Site Document Control Officer (SDCO) and professionals assisting with SPO data validation, reconciliation, and QA/QC activities. Section 1.4.3 of this QAPjP describes INEEL waste characterization facilities. The following sections describe the responsibilities of TWCP personnel. Each FIP identifies the facility-specific organization structure related to TWCP activities.

1.1.1 DOE-Carlsbad Area Office

As defined by the WIPP QAPP, the DOE-CAO is responsible for overseeing the specific activities being performed at participating sites and ensuring that program requirements are met with regard to TRU waste testing, sampling, sample handling and custody, and associated data management.

1.1.2 DOE-Idaho Operations Office

The DOE-Idaho Operations Office (DOE-ID) reviews and approves this QAPjP. The DOE-ID program manager ensures all activities are conducted in compliance with DOE orders, the QAPP, the QAPD, this QAPjP, and all applicable U.S. Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA), and state of Idaho regulations. The DOE-ID program manager approves any revisions to this document before the revision is implemented. The DOE-ID program manager provides an interface among the operating contractors at the INEEL, CAO, other DOE operations offices, and DOE-Headquarters to resolve any problems that could affect TWCP quality.

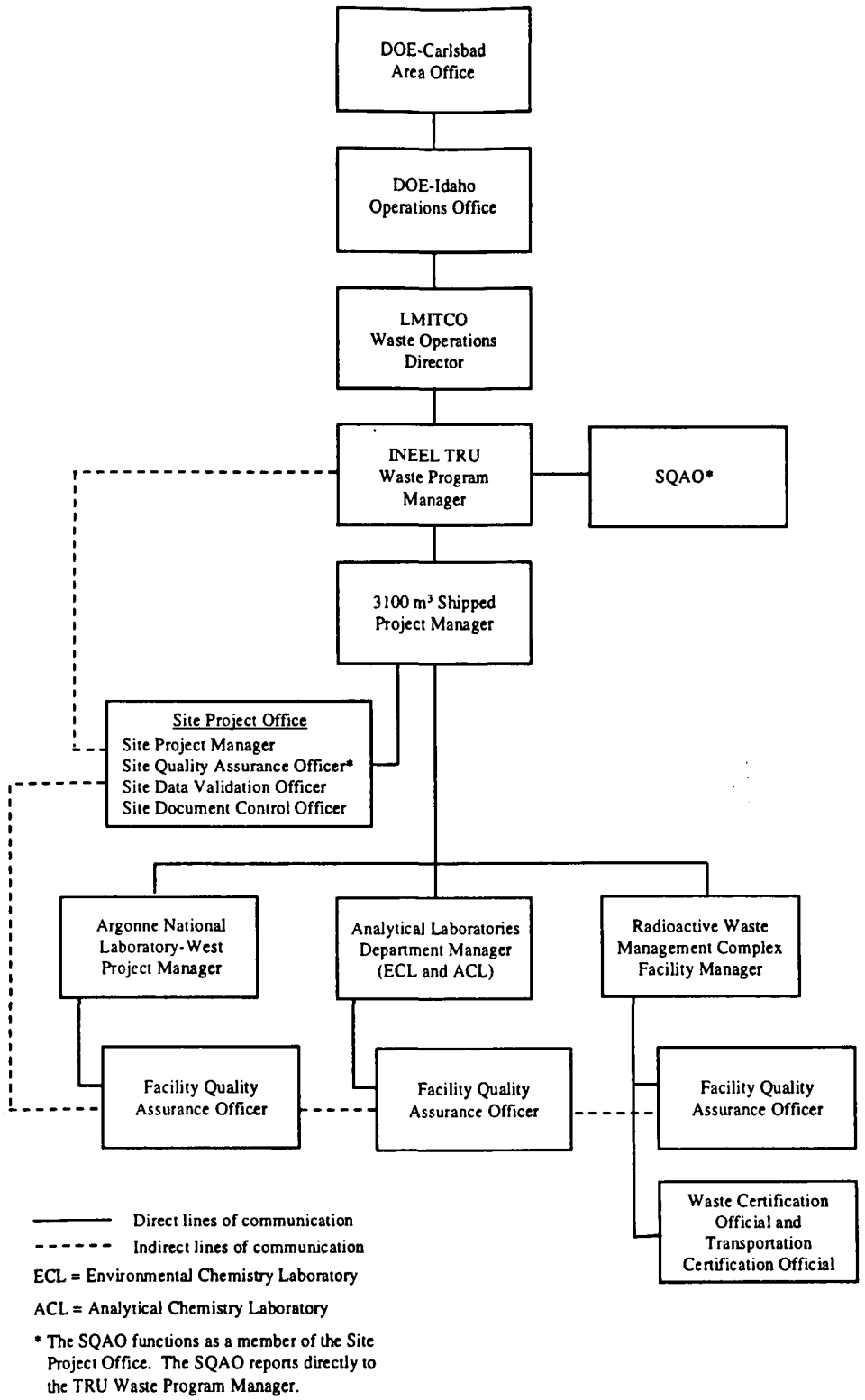


Figure 1-1. INEEL TWCP functional organizational chart.

1.1.3 Waste Operations Director

The Waste Operations Director (WOD) is responsible for contractor waste operations at the INEEL, and is the liaison between DOE-ID and the INEEL TRU Waste Program Manager.

1.1.4 INEEL TRU Waste Program Manager

The TRU Waste Program Manager is responsible for meeting production CH TRU waste certification goals established by DOE-ID, and for overseeing RWMC CH TRU waste storage, characterization, transportation, and certification activities in accordance with LMITCO and DOE-CAO requirements. The TRU Waste Program Manager is responsible for ensuring that methods and control programs have been implemented at the RWMC, and approved to meet characterization, certification, and transportation program objectives. The TRU Program Manager is responsible for resolution with the DOE-CAO of identified issues or concerns related to compliance with the DOE-CAO requirements.

1.1.5 3100 m^3 Shipped Project Manager

The 3100 m^3 Shipped Project Manager at the INEEL reports to the INEEL TRU Waste Program Manager. The 3100 m^3 Shipped Project Manager is responsible for retrieval, characterization, and transportation of 3100 cubic meters of certified TRU waste to WIPP by December 21, 2002.

1.1.6 INEEL Site Project Manager

The INEEL SPM is responsible for program planning and all operational aspects of the TWCP at the INEEL and reports directly to the 3100 m^3 shipped project manager. The SPM is responsible for managing activities in compliance with the QAPP, the QAPD, and this QAPjP. The SPM is responsible for the following program activities:

- Waste selection and tracking
- Program Change Notice approval
- Data validation/verification
- Data reconciliation with data quality objectives (DQOs)
- Assignment of EPA hazardous waste numbers
- Data transmission to CAO

The SPM provides technical direction to the SQAO, the SDCO, and the SDVO, as necessary, and coordinates the activities of TWCP participants. The SPM reviews and approves this QAPjP and FIPs, reviews variances.

1.1.7 Site Quality Assurance Officer

The SQAO ensures all TWCP QA requirements are implemented in accordance with this QAPjP; this includes verification and assessments of the QA program. The SQAO reviews and approves this QAPjP and FIPs and provides day-to-day guidance to TWCP staff on quality-related matters, as

necessary. The SQAO identifies and reports quality problems to the SPM and TRU Waste Program Manager and initiates, recommends, and tracks corrective actions to closure. To ensure the independence of the QA function, the SQAO reports directly to the TRU Waste Program Manager. The SQAO has the authority to stop TWCP activities at INEEL if quality is not assured or controlled. The SQAO is supported by QA personnel at each facility [collectively referred to as facility QA Officers (FQAOs) for the purposes of this QAPjP]. The following SQAO responsibilities are shared by the SQAO and FQAOs.

- Program Change Notice approval
- Laboratory/testing facility assessment
- Nonconformance tracking
- Corrective action verification
- Data validation/verification
- Data QA documentation verification
- Evaluating trends in compliance with TWCP objectives
- QA/QC reports to the TRU Program Manager

1.1.8 Site Data Validation Officer

The SDVO, under the supervision of the SPM, performs TWCP project-level data validation, verification, and reconciliation with DQOs, selects waste containers to be sampled, and reports data to CAO. The SDVO position was created by INEEL in order to comply with the QAPP requirements and meet internal project milestones, and reports to the 3100 m^3 Project Manager.

1.1.9 Site Document Control Officer

The SDCO maintains a document and records management system as described in the *Site Project Office Implementation Plan for the Transuranic Waste Characterization Program* (INEEL 1999i) (SPO implementation plan). The SDCO controls the data report and, upon completion of project-level data validation and reporting, transmits data to CAO and provides input to the Waste Isolation Pilot Plant (WIPP) Waste Information System (WWIS) as described in the SPO implementation plan. The SDCO reports to the 3100 m^3 Project Manager.

1.1.10 Argonne National Laboratory-West TWCP Project Manager

The ANL-W TWCP project manager (PM) is responsible for TWCP activities at ANL-W and coordinates activities and interfaces with DOE-ID, other ANL-W organizations, LMITCO, TWCP personnel, and DOE-CH, as required. ANL-W TWCP activities are described in the *Argonne National Laboratory-West* FIP *for the Transuranic Waste Characterization Program* (ANL-W 1999) (ANL-W implementation plan). The ANL-W PM interfaces with SPO personnel to establish methods that implement TWCP requirements; performs data generation level data review, validation, verification, and reporting to the SPO; and reviews and/or approves ANL-W TWCP documents and records, including (as

a minimum) the ANL-W implementation plan, nonconformance reports (NCRs), program change notices (PCNs), audit reports, and TWCP QA reports to the SQAO. The ANL-W PM is assisted in these responsibilities by project staff.

1.1.11 Radioactive Waste Management Complex Facility Manager

The Radioactive Waste Management Complex (RWMC) manager is responsible for the safe completion of activities for the TWCP at the Stored Waste Examination Pilot Plant (SWEPP) facility. The RWMC manager coordinates with other operational and laboratory personnel as required to correct any discrepancies in activities. RWMC activities are described in the *Radioactive Waste Management Complex Implementation Plan for the Transuranic Waste Characterization Program* (INEEL 1999g) (RWMC implementation Plan). The RWMC manager is responsible for RWMC operations and equipment used during the TWCP; ensures RWMC personnel are properly trained; and reviews, validates, verifies, and reports testing data to the SPO. The RWMC manager is assisted in these responsibilities by technical specialists, supervisors, and the FQAO.

1.1.12 Analytical Laboratories Department Manager

The Analytical Laboratories Department (ALD) manager is responsible for directing laboratory operations for headspace gas analyses at the Environmental Chemistry Laboratory (ECL) and solids and soil/gravel analyses at the Analytical Chemistry Laboratory (ACL) in compliance with the QAPP, the QAPD, and this QAPjP. ALD manager responsibilities include, but are not limited to, personnel training, equipment and systems maintenance, laboratory safety, customer interfacing, work status, data review, and cost control. The ALD manager is assisted in these responsibilities by supervisors, technical leaders, and the FQAO at each laboratory. Responsibilities within the ALD are described in the *Environmental Chemistry Laboratory Implementation Plan for the Transuranic Waste Characterization Program* (INEEL 1999b) (ECL implementation plan) and the *Analytical Chemistry Laboratory Implementation Plan for the Transuranic Waste Characterization Program* (INEEL 1999a) (ACL implementation plan).

1.1.13 Facility Quality Assurance Officers

Each FQAO is responsible for ensuring the facility-level QA TWCP requirements are implemented. To ensure the independence of the QA function, the FQAO interfaces with the SQAO as necessary. Each FIP plan describes specific FQAO responsibilities, that include data validation, NCR tracking, PCN approval, and preparing and submitting QA reports to management.

1.2 Program Documents

The INEEL TWCP is implemented by the of QA-related documents described in this section. In addition, the *INEEL Transuranic Waste Program Procedures Matrix for the DOE-CAO QAPD* (INEEL 1998d) (QAPD Procedures Matrix) identifies the procedures that implement each QAPD requirement. A cross reference of QA requirements and QA-related documents is provided as Table 1-1.

Table 1-1. Cross reference of quality assurance requirements.

QAPP and QAPjP Sections	CAO QAPD Requirements	10 CFR §830.120(c) Quality Assurance Criteria	ASME NQA-1 Basic Requirements[a]
Section 1.0 Program Management			
Program Organization	Quality Assurance Program and Organization	Management Program	Organization
Program Documents	Documents	Documents and Records	Document Control
Problem Definition and Background	Planning Scientific Investigations		
Program Description	Quality Assurance Program and Organization	Management Program	Quality Assurance Program
Data Quality Objectives for Measurement Data	Design Control Planning Scientific Investigations	Design	Design Control
Special Training Requirements and Certifications	Personnel Qualification and Training	Personnel Training and Qualification	Quality Assurance Program
Documentation and Records	Records	Documents and Records	Quality Assurance Records
	Data Documentation, Control, and Qualification		
Procurement	Procurement	Procurement	Procurement Document Control of Purchased Items and Services
Work Processes	Work Processes	Work Processes	Control of Processes
	Software QA Requirements		Identification and Control of Items

Table 1-1. (continued).

QAPP and QAPjP Sections	CAO QAPD Requirements	10 CFR §830.120(c) Quality Assurance Criteria	ASME NQA-1 Basic Requirements[a]
Section 2.0 Assessment and Oversight			
Assessment and Response Actions	Quality Improvement	Quality Improvement	Control of Nonconforming Items
	Management Assessment	Management Assessment	Corrective Action
	Independent Assessment	Independent Assessment	Audits
Reports to Management	Quality Improvement	Quality Improvement	Quality Assurance Program
	Independent Assessment		Corrective Action
Performance Demonstration Program	Inspection and Testing	Inspection and Acceptance Testing	Inspection
Section 3.0 Data Validation and Usability			
Data Review, Validation, and Verification Requirements	Data Documentation, Control, and Qualification	Work Processes	Control of Nonconforming Items
	Work Processes	Design	
	Design Control		
Validation Methods	Design Control	Design	Test Control
Reconciliation with Data Quality Objectives	Design Control	Design	Design Control
	Data Documentation, Control, and Qualification		
Data Reporting Requirements	Records	Documents and Records	Quality Assurance Records
	Data Documentation, Control, and Qualification		
Section 4.0 Acceptable Knowledge			
	Work Processes	Work Processes	Control of Processes
	Records	Documents and Records	Quality Assurance Records

Table 1-1. (continued).

QAPP and QAPjP Sections	CAO QAPD Requirements	10 CFR §830.120(c) Quality Assurance Criteria	ASME NQA-1 Basic Requirements[a]
Section 5.0 Sampling Process Design			
	Design Control	Design	Design Control
			Instructions, Procedures, and Drawings
Section 6.0 Sample Handling and Custody Requirements			
	Sample Control	Work Processes	Identification and Control of Items
	Sample Identification		
	Handling, Storing, and Shipping		Handling, Storage, and Shipping
	Samples		
	Disposition of Nonconforming		
	Samples		
	Work Processes		
Section 7.0 through 15.0 Techniques			
Quality Assurance Objectives	Design Control	Design	Design Control
Methods Requirements	Performing Scientific Investigation	Work Processes	Instructions, Procedures, and Drawings
	Work Processes		Control of Processes
Quality Control Requirements	Work Processes	Work Processes	Control of Processes
			Test Control

Table 1-1. (continued).

QAPP and QAPjP Sections	CAO QAPD Requirements	10 CFR §830.120(c) Quality Assurance Criteria	ASME NQA-1 Basic Requirements[a]
Instrument/Equipment Testing, Inspection, and Maintenance Requirements	Work Processes	Work Process	Inspection
	Inspection and Testing	Inspection and Acceptance Testing	Inspection, Test, and Operating Status
Instrument Calibration and Frequency	Work Processes	Work Processes	Control of Measuring and Test Equipment
	Inspection and Testing	Inspection and Acceptance Testing	
Data Management	Records	Documents and Records	Quality Assurance Record
	Data Documentation, Control, and Qualification		

a. American Society of Mechanical Engineers (ASME) *Quality Assurance Requirements for Nuclear Facility Applications* (ASME 1989) (NQA-1). Includes applicable requirements of ASME NQA-2a-1990, Part 2.7, "Quality Assurance Program Requirements of Computer Software for Nuclear Facility Applications" (ASME 1990).

1.2.1 LMITCO QAPD and ANL-W QAP

The *Program Description for LMITCO Quality Assurance Program* (INEEL 1999e) (LMITCO QAPD) and the *Argonne National Laboratory-West Quality Assurance Plan* (ANL-W 1998) (ANL-W QAP) describe the QA programs of LMITCO and ANL-W, respectively. They apply to all quality-affecting activities. These documents implement the 10 CFR §830.120 and DOE Order 5700.6C requirements.

1.2.2 INEEL Certification Plan and TRAMPAC

The Program Plan for Certification of INEEL Contact-Handled Stored Transuranic Waste (INEEL 1999f) (INEEL Certification Plan) and the RWMC Compliance Plan for TRUPACT-II Authorized Methods for Payload Control (TRAMPAC) (INEEL 1999h) describe how INEEL complies with the waste certification requirements of the Waste Acceptance Criteria for the Waste Isolation Pilot Plant (DOE 1996d) (WIPP WAC) and the waste transportation requirements of the TRUPACT-II Certificate of Compliance.

1.2.3 INEEL TRU Waste Characterization, Transportation, and Certification Quality Program Plan

The INEEL Transuranic Waste Characterization, Transportation, and Certification Quality Program Plan (INEEL 1999c) (QPP) implements the QAPD requirements that apply to waste characterization, certification, and transportation. These DOE-CAO QA requirements are project-specific and are not addressed in the LMITCO QAPD or ANL-W QAP; therefore, a project-specific Quality Program Plan (QPP) is necessary. The QPP is the INEEL QA plan that satisfies the WIPP WAC requirement for each participating site to develop and implement a site-specific QA plan for waste certification and TRUPACT-II payload control and cask usage. The ANL-W QA program for project-specific activities is also addressed in this INEEL document.

1.2.4 Quality Assurance Project Plan

This QAPjP follows the document format specified in the QAPP. This QAPjP is implemented by FIPs that address the characterization activities conducted at the facility. Figure 1-3 presents an overall idealized sequence of TWCP activities and indicates the INEEL facility responsible for each activity. Distribution of this QAPjP is controlled as specified in the SPO implementation plan. Table 1-2 specifies minimum review and approvals required before implementation of this QAPjP. SPO documents and procedures and related revisions are approved by the SPM and SQAO and controlled as described in the SPO implementation plan. Document and procedure revisions are reviewed and approved by the same level of approval authority as the original documents.

1.2.5 Facility Implementation Plans

FIPs implement this QAPjP. Table 1-3 lists the FIP. FIPs contain additional information pertinent to the TWCP, but not required by the QAPP. The plans contain references to procedures and additional internal INEEL program requirements and guidance pertinent to the TWCP, such as radiochemistry analysis of solid samples. In addition, the FIPs and/or procedures include report forms and data

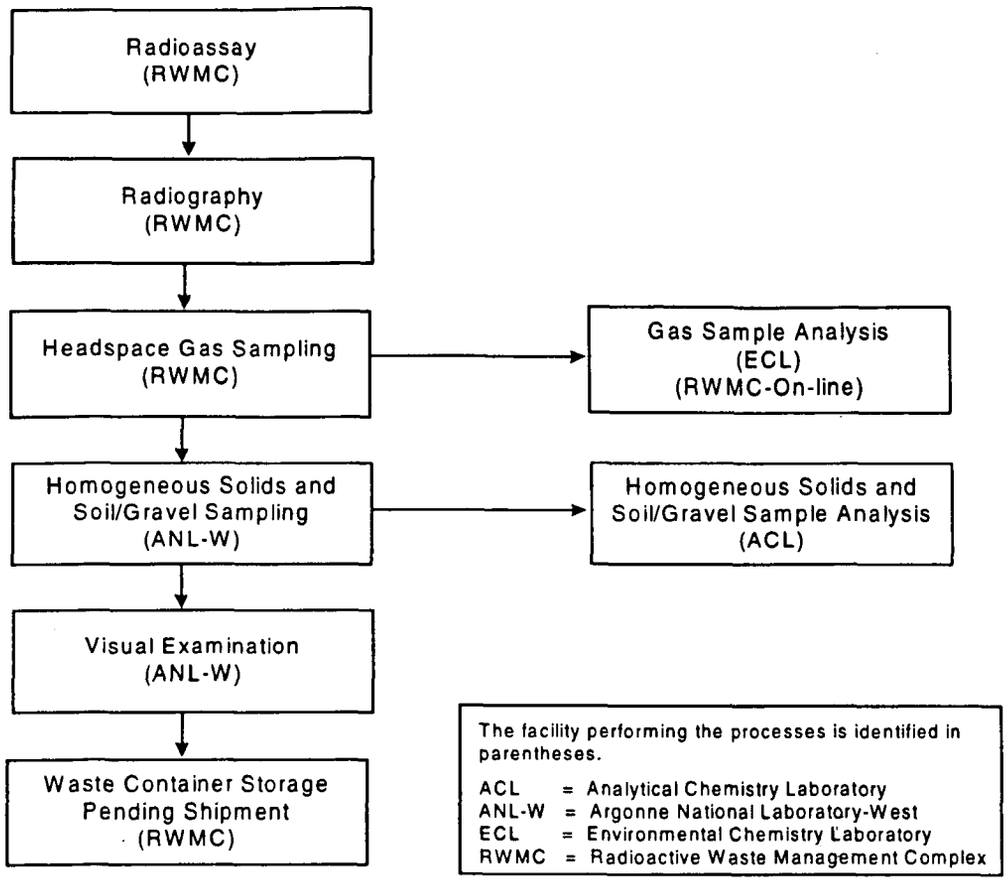


Figure 1-3. Idealized sequence of INEEL waste characterization processes.

Table 1-2. Review, approval, implementation, and control requirements for the INEEL QAPjP.

Reviewer	Review	Review and Approval	Change Approval	Change Control
Manager, CAO Quality Assurance	—	X	X	—
Team Leader, National TRU Program	—	X	—	—
DOE-ID Office/Project Manager	—	X	X	—
Department Manager, TRU Waste Programs	—	X	X	—
SPM	—	X	X	X
SQAO	—	X	X	—
SDVO	X	—	—	—
ALD Manager	X	—	—	—
ANL-W Project Manager	X	—	—	—
RWMC Manager	X	—	—	—
Facility QA Officers	X	—	—	—

NOTE: — = not required.

Table 1-3. INEEL TWCP FIPs.

Facility	Implementation Plan Number
SPO	PLN-188
RWMC	PLN-185
ANL-W	W0096-0481-ES
ECL	PLN-186
ACL	PLN-187

validation checklists. FIPs are approved at the facility level and by the SPM and SQAO. Each FIP specifies internal review and approval requirements. Other facility documents and procedures are approved and controlled at the facility level as described in each FIP and facility-specific document control procedure.

1.2.6 Transuranic Waste Characterization Program Project-Level Work Control

The SDCO maintains the *SPO Management Control Procedures* manual to identify and control procedures used to meet SPO requirements. Quality procedures that implement QAPD requirements and QAPP QA requirements are described in the QAPD Procedures Matrix (INEEL 1999d). Technical procedures that implement QAPP characterization requirements are listed in Sections 7 through 15 of this QAPjP. Procedures are also listed in FIPs. These procedures and the *INEEL Transuranic Waste Characterization, Transportation, and Certification Quality Program Plan* (INEEL 1999c) describe the

general administration, document control, quality, and data review, validation, verification, and reporting processes implemented by the SPO to comply with QAPP and QAPD requirements. The SDCO indexes, releases, retrieves, and stores documents and maintains a distribution list for SPO documents. The SDCO also distributes other controlled TWCP documents and revisions, including the QAPP, the *Transuranic Waste Characterization Sampling and Analysis Methods Manual* (DOE 1996c) (Methods Manual), and this QAPjP.

1.2.7 Facility-Level Work Control

Facility personnel perform TWCP activities in accordance with written procedures in compliance with the QPP and QAPD. These procedures are identified in the QAPD Procedures Matrix. The implementing procedures fall into three major categories: analytical, operational, and administration procedures. SPO personnel develop procedures in accordance with the SPO implementation plan. Each facility follows its established NQA-1 system for preparation and control of implementing procedures such as standard operating procedures (SOPs), technical procedures (TPRs), management control procedures (MCPs), or operating instructions (OIs). This QAPjP refers to these implementing documents collectively as procedures. Each FIP lists the procedures that implement the requirements of this QAPjP. Each facility supporting the TWCP distributes TWCP-related documents to the SPO and retains obsolete revisions of TWCP procedures. Each FIP describes the facility-specific format and document review, approval, and control process.

1.3 Problem Definition and Background

The INEEL currently stores approximately 130,000 drums of TRU waste in the Transuranic Storage Area (TSA) at the RWMC in inspectable storage or under earthen or geofabric cover. The majority of these wastes are general laboratory and secondary processing wastes received since 1970 from operations at the Rocky Flats Environmental Technology Site (RFETS). The remainder is from the INEEL and other DOE laboratories. This QAPjP applies to all TRU waste at the INEEL that is defined as contact-handled retrievably stored.

Any discussion in this QAPjP that refers to Resource Conservation and Recovery Act (RCRA) regulations promulgated by 40 CFR Parts 260 through 270 also refers to the Idaho Administration Procedures Act, Health and Welfare Department 16, Title 1, Chapter 5, Sections 004 through 012 (IDAPA 16.01.05.004 through 16.01.05.012). Mixed waste refers to waste regulated by both the Atomic Energy Act and RCRA. In this QAPjP, the term TRU waste refers to TRU and TRU mixed waste.

Over the WIPP facility's 35-year disposal phase, the INEEL plans to dispose of approximately 29,000 cubic meters (m^3) of retrievably stored CH TRU waste and approximately 220 m^3 of retrievably stored RH TRU waste. The INEEL characterizes TRU waste using the waste selection, acceptable knowledge, testing, sampling, and analytical techniques described in this QAPjP.

1.4 Program Description

The TWCP at the INEEL characterizes and certifies retrievably stored TRU waste for shipment to the WIPP. All retrievably stored waste at the INEEL is stored in drums. This QAPjP applies to the characterization of retrievably stored TRU waste drums. Any retrievably stored TRU waste drums that are repackaged during conduct of the TWCP are considered newly generated waste. A future revision of

this QAPjP will address characterization of newly generated waste. The INEEL facilities implement the QAPP requirements as described in this QAPjP and FIPs.

1.4.1 General Overview

The INEEL temporarily stores TRU waste at the RWMC; much of this waste is planned for disposal at WIPP. The INEEL characterizes this waste and provides the results to CAO in accordance with the QAPP.

The TWCP is designed to characterize TRU waste on a waste stream basis. A waste stream is defined as waste material generated from a single process or activity that is similar in material, physical form, and hazardous constituents. The TWCP has adopted the waste description nomenclature outlined in the most current revision of the *Waste Isolation Pilot Plant Transuranic Waste Baseline Inventory Report* (DOE 1995e) (TWBIR) and the *DOE Waste Treatability Group Guidance* (DOE 1995a). The TWCP considers three broad matrix parameter categories of waste: homogeneous solids (summary category S3000), soil/gravel (summary category S4000), and debris wastes (summary category S5000). These matrix parameter categories are used to provide a description of the waste physical form and determine characterization requirements for the TWCP.

INEEL uses several different classification systems for RFETS waste. These include item description codes (IDCs), TRUPACT-II content codes (TRUCONs), and matrix parameter categories (MPCs). The most specific description is provided by the IDC, which is the code assigned by RFETS. All other classification systems in use are groupings of the IDCs. The characterization programs at the INEEL are conducted at the IDC level to take advantage of the most detailed information available.

1.4.2 INEEL Site Project Office

The INEEL SPO provides overall management and coordination for the characterization of TRU waste at the INEEL. SPO personnel statistically select waste containers for core sampling and visual examination; validate and verify all sampling, testing, and analytical data; prepare summary data reports for each drum characterized; and transmit data to CAO.

1.4.3 INEEL Waste Characterization Facilities

The INEEL conducts TRU waste characterization activities at several INEEL facilities, as depicted on Figure 1-3. The facility activities are summarized as follows:

- **RWMC Activities**: RWMC personnel retrieve the selected TRU waste drums from storage at the SWEPP. RWMC personnel initiate drum tracking, weigh drums, collect drum headspace gas samples, perform on-line gas analysis, perform real-time radiography (RTR), and nondestructive radioassay (RA), and report the results of headspace gas sampling, RTR, and RA characterization activities to the SPO. RWMC personnel package some of the characterized waste drums in Transuranic Package Transporter-IIs (TRUPACT-IIs) for transportation to ANL-W personnel at the Hot Fuel Examination Facility (HFEF) for further characterization and ship headspace gas samples to the ECL for analysis.

- **ANL-W Activities**: ANL-W personnel receive the shipment of waste drums from the RWMC and temporarily store the drums at the HFEF in a specified area until waste characterization of each drum proceeds. ANL-W personnel move each drum into the HFEF

waste characterization area (WCA), perform visual examination on a portion of the waste drums, and obtain samples from a portion of matrix parameter summary category S3000 and S4000 drums. ANL-W personnel ship solid samples to the ACL for analysis and return the waste drums to RWMC for storage after characterization at WCA. ANL-W personnel report the visual examination and solids sampling results to the SPO.

- **ECL Activities**: ECL personnel analyze the headspace gas samples collected in SUMMA® canisters for volatile organic compounds (VOCs), hydrogen (H_2), and methane (CH_4), and report results to the SPO. ECL personnel clean and recertify the SUMMA® canisters.

- **ACL Activities**: ACL personnel analyze the S3000 and S4000 samples for total VOCs, total semivolatile organic compounds (SVOCs), and total metals and report analysis results to the SPO.

1.5 Data Quality Objectives

Table 1-4 lists the DQOs established for the TWCP. Table 1-5 lists the characterization techniques, parameters, and facilities used at the INEEL to obtain data in support of the DQOs. Appropriate sections of this QAPjP and FIPs identify the quality assurance objectives (QAOs) for each characterization technique listed in Table 1-5.

Table 1-4. Data quality objectives.

Characterization Technique	Data Quality Objective
Radioassay	To classify waste by activity as low-level versus TRU waste by demonstrating with a 95% probability that the total TRU activity is less than or equal to 100 nCi/g of waste. The quality assurance objectives (QAO) for the minimum detectable concentration for TRU measurements was selected to help ensure that measurements in the 60 to 80 nCi/g region can be made with sufficient precision to avoid designating excessive quantities of alpha contaminated TRU waste as low-level waste.
	To confirm the radionuclide inventory on which the 40 CFR Part 191 certification application is based and assess compliance with the individual protection requirements, ground water protection standards, and containment requirements (40 CFR Part 191).
	To obtain the total activity in TRU waste to support revision of the thermal power restrictions for shipment of waste in the Transuranic Package Transporter-II (TRUPACT-II).
Radiography	To classify/verify the TRU waste inventory by matrix parameter category and waste material parameter (DOE 1995e) on which the 40 CFR Part 191 certification application is based.
	To verify the TRU IDCs by matrix parameter category (DOE 1995e) for purposes of physical waste form identification and determination of sampling and analytical requirements (Section 5).
Gas sampling and analysis	To quantify the concentrations of H_2, CH_4, and flammable VOCs by waste container and determine the potential flammability of TRU waste headspace gases.
	To quantify the concentrations of volatile organic hazardous constituents in the total waste inventory to support a demonstration that volatile organic hazardous constituents will not migrate through the air beyond the WIPP unit boundary in concentrations greater than EPA-determined health-based limits during the WIPP disposal phase.
	To quantify H_2 and CH_4 headspace concentrations in waste containers to support revision of the thermal power restrictions for shipment of TRU waste in the TRUPACT-II.
Homogeneous solids and soil/gravel sampling and analysis	To compare the upper 90% confidence limit (UCL_{90}) values for the mean measured contaminant concentrations in an IDC to the specified regulatory levels (40 CFR Part 261, Subpart C). That is, to determine if an IDC exhibits a toxicity characteristic.
	To report the average concentrations, standard deviation, UCL_{90}, and number of samples collected for hazardous constituents in an IDC, as specified in 40 CFR Part 261, Appendix VIII.

Table 1-5. Summary of wastes characterization requirements.

Parameter		Characterization Techniques	Characterization Facility/Laboratory
Radionuclide		**Radioassay**	
Pu-239 fissile gram equivalents		Nondestructive Assay	
Total alpha activity		(QAPjP **Section 9)**	RWMC
TRU activity			
Individual radioisotopes			
Thermal power			
Physical Waste Form		**Waste Inspection Procedures**	
Matrix Parameter Categories		Radiography	
		(QAPjP Section 10)	RWMC
Summary Category	*Names*	Visual Examination	ANL-W
S3000	Homogeneous Solids	(QAPjP Section 10)	
S4000	Soil/Gravel		
S5000	Debris Wastes		
Waste Material Parameters			
Iron -Based Metals/Alloys			
Aluminum-Based Metals/Alloys			
Other Metals			
Other Inorganic Materials			
Cellulosics			
Rubber			
Plastics (waste material)			
Organic matrix			
Inorganic matrix			
Soil			
Steel (packaging material)			
Plastics (packaging material)			
Headspace Gases		**Gas Analysis**	
Hydrogen		Gas mass spectrometry or gas chromatography	Gas sample collection:
Methane		On-line mass spectrometry for hydrogen	RWMC
		(QAPjP Section 11)	

Table 1-5 (continued).

Parameter	Characterization Techniques	Characterization Facility/Laboratory
Volatile Organic Compounds *flammable* acetone benzene butanol chlorobenzene cyclohexane 1,1-dichloroethane 1,2-dichlorethane 1, 1-dichloroethylene cis-1,2-dichloreoethylene ethyl benzene ethyl ether methanol methyl ethyl ketone methyl isobutyl ketone toluene 1,2,4-trimethylbenzene 1,3,5-trimethylbenzene xylenes *Nonflammable* bromoform carbon tetrachloride chloroform methylene chloride 1,1,2,2-tetrachloroethane tetrachloroethylene 1,1-2-trichloro-1,2,2-trifluoroethane 1,1,1-trichloroethane trichloroethylene	Fourier transform infrared spectroscopy for methane (QAPjP Section 12) Gas chromatography/mass spectrometry Gas chromatography/flame ionization Detector Fourier transform infrared spectroscopy (QAPjP Section 12)	Gas analysis: ECL (SUMMA® canisters): and RWMC (on-line) Gas analysis: ECL (SUMMA® canisters): and RWMC (on-line)

Table 1-5 (continued).

Parameter		Characterization Techniques	Characterization Facility/Laboratory
Volatile Organic Compounds in Solid Samples		**Total Volatile Organic Compound Analysis**	
acetone	Methylethyl ketone	Gas chromatography/mass spectrometry	Sample collection:
benzene	methylene chloride	Gas chromatography/flame ionization detector	ANL-W
bromoform	1,1,2,2-tetrachloroethane	(QAPjP Section 13)	Sample analysis:
butanol	tetrachloroethylene		ACL
carbon disulfide	toluene		
carbon tetrachloride	1,2,3-trichloro-1,2,2-trifluoroethane		
chlorobenzene	1,1,1-trichloroethane		
chloroform	trichloroethane		
1,2-dichloroethane	trichloroethylene		
1,1-dichloroethene	vinyl chloride		
ethyl benzene	xylenes		
ethyl ether			
isobutanol			
methanol			
Semi-Volatile Organic Compounds		**Total Semi-Volatile Organic Compound Analysis**	
cresols		Gas chromatography/mass spectrometry (QAPjP Section 14)	Sample collection: ANL-W
ortho-dichlorobenzene			
1.4-dichlorobenzene			
2,4-dinitrophenol		Acceptable knowledge for debris wastes (Matrix parameter summary category S5000) (QAPjP Section 4)	Sample analysis: ACL
2,4-dinitrotoluene			
hexachlorobenzene			
hexachloroethane			
nitrobenzene			
pentachlorophenol			
pyridine			
polychlorinated biphenyls (PCBs) (IDC 003 only)			

Table 1-5 (continued).

Parameter	Characterization Techniques	Characterization Facility/Laboratory
Metals	**Total Metals Analysis**	
antimony arsenic barium beryllium cadmium chromium lead mercury nickel selenium silver thallium vanadium zinc	Inductively coupled plasma atomic emission spectroscopy Cold vapor atomic fluorescence spectrophotometry (QAPjP Section 15) Acceptable knowledge for debris wastes (Matrix parameter summary category S5000) (QAPjP Section 4.0) TCLP, when non-mixed determinations are required.	Sample collection: ANL-W Sample analysis: ACL

1.6 Special Training Requirements and Certification

The SPM and facility line management ensure all TWCP personnel receive indoctrination into the scope, purpose, and objectives of the program and the specific QAOs of the task being performed. All SPO personnel receive training requisite with their activities and level of responsibility and maintain minimum qualifications as described in the SPO implementation plan. Facility personnel receive initial and continuing training requisite with their activities and level of responsibility and maintain minimum qualifications as described in facility training programs. TWCP personnel performing activities affecting quality are trained to ensure they achieve and maintain suitable proficiency. Table 1-6 specifies the minimum qualifications for analytical personnel. Job performance is evaluated and documented at periodic intervals not to exceed three years.

Training programs for facility personnel meet or exceed the minimum training and qualification requirements in Table 1-6 and are the responsibility of the training coordinator at each facility. Facility training programs are described in FIPs. The FIP sidentify the facility-specific job titles that correspond to the positions listed in Table 1-6. FIPs identify the documents that describe the training program. The facility training programs comply with the requirements specified in the QAPP, the QAPD, and 10 CFR §830.120. The personnel qualification and training criteria specified in the QAPD are implemented as described in the QAPD Procedures Matrix. Training records are maintained in the facility files in accordance with Section 1.7.

1.7 Records Management

Quality records at the INEEL are maintained consistent with the requirements of the QAPP and the QAPD to ensure objective evidence of quality is retrievable. The SDCO maintains TWCP files in accordance with the SPO implementation plan. The SDCO is responsible for SPO records administration. Facility personnel specified in Section 1.1 are responsible for TWCP records administration at the RWMC, ANL-W, ACL, and ECL.

The SPO implementation plan addresses the major elements of records identification, maintenance, control, and disposition. The SDCO ensures records are collected, processed, stored, and maintained in accordance with the SPO implementation plan and ASME NQA-1, Supplement 17S-1, as either lifetime records or nonpermanent records. Special processed records (e.g., microfilm, optic, and magnetic media) are physically protected from damage or deterioration from excessive light, stacking, electromagnetic fields, temperature, and humidity. FIP address identification, control, maintenance, and disposition of records pertinent to the facility. Table 1-7 lists the TWCP records to be maintained as lifetime or nonpermanent. TWCP personnel maintain the records in TWCP files in accordance with the QAPD and an approved records inventory and disposition schedule (RIDS) or LMITCO uniform filing code. Lifetime records are maintained at the SPO for the life of the TWCP at INEEL plus six years then offered to CAO or transferred to the appropriate Federal Records Center. Nonpermanent records are maintained at the SPO or facility (as necessary) for 10 years from the date of record generation and dispositioned according to the approved RIDS or LMITCO Uniform Filing Code.

Table 1-6. Minimum training and qualifications requirements[a].

Personnel	Requirements[a]
Radiography Operators[c]	Site-specific training based on matrix parameter categories and waste material parameters; requalification every two years
Gas Chromatography Technical Supervisors[b] Gas Chromatography Operators[c]	B.S. or equivalent experience and six months previous applicable experience
Gas Chromatography/Mass Spectrometry Operators[c] Mass Spectrometry Operators[c]	B.S. or equivalent experience and one year independent spectral interpretation or demonstrated expertise
Gas Chromatography/Mass Spectrometry Technical Supervisors[b] Mass Spectrometry Technical Supervisors[b] Atomic Absorption Spectroscopy Technical Supervisors[b] Atomic Absorption Spectroscopy Operators[c] Atomic Mass Spectrometry Operators[c] Atomic Emission Spectroscopy Operators[c]	B.S. or equivalent experience and one year applicable experience
FTIRS Technical Supervisors	B.S. or equivalent experience and one year applicable experience.
FTIRS Operators	Applicable training and demonstrated expertise.
Atomic Mass Spectrometry Technical Supervisors[b]	B.S. and specialized training in Atomic Mass Spectrometry and two years applicable experience
Atomic Emission Spectroscopy Technical Supervisors[b]	B.S. and specialized training in Atomic Emission Spectroscopy and two years applicable experience.

a. Based on requirements contained in USEPA Contract Laboratory Program Statement of Work for Organics Analysis (Document Number OLM 01.0) and Statement of Work for Inorganics Analysis (Document Number ILM 03.0).

b. Technical Supervisors are those persons responsible for the overall technical operation and development of a specific laboratory technique. FIP include the facility-specific title for this position.

c. Operators are those persons responsible for the actual operation of analytical equipment. FIPs include the facility-specific title for this position.

Table 1-7. Classification for waste characterization QA records.

Records
Lifetime
• Field sampling data forms
• Field and laboratory chain-of-custody forms
• Test facility and laboratory analytical data reports
• Summary data packages
• Sampling Plans
• Data reduction, validation, and reporting documentation
• Acceptable knowledge documentation
• Data reconciliation report
• Waste Stream Profile Form
Nonpermanent
• Nonconformance documentation
• PCN documentation
• Assessment documentation
• Gas canister tags
• Methods performance documentation
• Performance Demonstration Program documentation
• Sampling equipment certifications
• Calculations and related software documentation
• Training/qualification documentation
• QAPP (CAO)/QAPjPs (INEEL) documentation (all revisions)
• Calibration documentation
• Analytical raw data
• Procurement documentation
• QA procedures (all revisions)
• Technical implementing procedures (all revisions)
• Audio/video recording (RTR, visual, etc.)

1.8 Procurement

All INEEL facilities implement procedures to ensure procured items and services meet requirements and perform as intended. Procurement controls specified in the QAPP and QAPD are applicable to equipment and services that directly affect testing, sampling, and analytical data quality. The procurement criteria specified in the QAPD are implemented as described in the QAPD Procedures Matrix.

1.8.1 Procurement Document Control

The SPM and facility line management ensure TWCP personnel control procurement documents in accordance with procedures identified in FIPs for procurement document control.

1.8.2 Control of Purchased Items and Services

The SPM and facility line management ensure TWCP personnel control items and services purchased, including supplier evaluations and inspections. FIPs identify additional implementing procedures for control of purchased items and services.

1.8.3 Control of Subcontractors

Section 1.8.2 specifies requirements that also apply to subcontractors who perform work that directly affects the quality of characterization data. The SPO negotiates any subcontracts for analytical services.

1.9 Work Processes

TWCP personnel perform TRU waste characterization processes to approved procedures. These procedures comply with the requirements specified in the QAPP and QAPD. Quality procedures that implement QAPD requirements are described in the QAPD Procedures Matrix. TRU waste characterization procedures (including procedures to ensure testing equipment is properly controlled, calibrated, and maintained) are listed in FIPs and Sections 7 through 15 of this QAPjP.

1.9.1 Control of Processes

The SPM and facility line management ensure TWCP activities are controlled and conducted in accordance with controlled procedures. FIPs identify the TRU waste characterization procedures applicable to their work. Major technical procedures are also listed in Sections 7 through 15 of this QAPjP.

1.9.2 Identification and Control of Items

Facilities establish and implement procedures to identify items (e.g., items with a limited shelf life or operating life, materials, equipment, samples) and ensure that only correct and accepted items are used. These procedures comply with the QAPP and QAPD requirements. The item identification and control criteria specified in the QAPD are implemented as described in the QAPD Procedures Matrix. Technical procedures are listed in Sections 7 through 15 of this QAPjP. FIPs also identify procedures for

identifying and controlling items. Identification and control of waste containers and samples are covered in subsequent sections of this QAPjP.

1.9.3 Computer Hardware and Software

Computer software and hardware/software configurations used in data collection, analysis of samples, data reduction, data processing, and data evaluation are developed, verified, validated, and tested prior to use in compliance with QAPP, QAPD, and *Quality Assurance Requirements of Computer Software for Nuclear Facility Operations* (NQA-2a) (ASME 1990) requirements. FIP define the specific procedures to be implemented for computer software development, validation, and verification.

1.9.4 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. FIPs identify the facility procedures pertinent to this section of the QAPjP.

2. ASSESSMENT AND OVERSIGHT

Participants in the INEEL TWCP implement an assessment and oversight program to meet QAPP and QAPD requirements. Assessment and oversight actions include audits, assessments, reports to management, and Performance Demonstration Program (PDP) participation.

2.1 Assessment and Response Actions

The SQAO has overall responsibility for ensuring INEEL TWCP personnel conduct assessment and response actions in accordance with the QAPP and QAPD. The INEEL TWCP facility personnel participate in and conduct management and independent assessments. FIPs identify personnel responsible for ensuring corrective action is taken when assessments and audits identify conditions or significant conditions adverse to quality and reporting assessment actions to the SQAO.

2.1.1 Audits

Facilities participating in the INEEL TWCP are subject to audits by CAO. A CAO audit of the INEEL TWCP is required before waste is shipped to the WIPP and annually thereafter. These audits are the responsibility of the CAO QA manager, who coordinates these audits through the SPM and SQAO. The SQAO ensures all conditions adverse to quality are resolved and appropriate corrective actions are implemented in a timely manner. The SQAO develops a schedule, in association with TWCP facility personnel, that details follow-up activities and final resolution of all corrective actions. The SQAO tracks corrective actions to completion.

2.1.2 Nonconformances

Nonconformances are uncontrolled and unapproved deviations from an approved plan, procedure, or expected result. Nonconforming items and activities are those that do not meet TWCP requirements (see definition section). TWCP participants report any conditions that do not comply with TWCP requirements. The SQAO, under the supervision of the SPM, evaluates and tracks nonconformances and reports this information to DOE-ID in the semiannual report.

All TWCP participants are responsible for identifying and reporting nonconforming items and processes. Facility and SPO line management are responsible for evaluating nonconformances and taking appropriate corrective action. Each FIP identifies the procedure used to control nonconformances in compliance with the QAPP and QAPD. The procedure identifies the person(s) responsible for evaluating, dispositioning, and tracking nonconformances. An NCR is prepared by the individual identifying the nonconformance. Each NCR includes the following information:

- Identification of individuals(s) identifying or originating the NCR
- Description of the nonconformance
- Method(s) of corrective action
- Schedule for completing the corrective action

- Cause of nonconformance (if known) and action to prevent reoccurrence
- A copy of, or reference to, appropriate background information (e.g., analytical results, QC tests, audit report, internal memoranda, letters)
- Indication of the potential ramifications and overall useability of the data, if applicable
- Approval signatures of facility personnel

Facility personnel report TWCP-related nonconformances to the SQAO and transmit copies of NCRs to the SQAO. The SQAO oversees the NCR process and coordinates with facility personnel to track nonconformances and verify corrective action completion.

2.1.3 Program Change Notices

Program Change Notices (PCNs) are approved and controlled temporary deviations from program-related plans and procedures. PCNs may not be used to deviate from higher-tier customer requirements (e.g.QAPP). PCNs may be initiated at either the characterization facilities or the SPO. PCNs to approved operational procedures, administrative procedures, or analytical and sampling protocols are changes caused by identification of improvement opportunities or unusual or nonroutine occurrences that affect planned activities, but not the ability to achieve performance standards or quality requirements. When a need to deviate from established procedures is identified, it is the responsibility of the person performing the work to initiate a PCN.

Each INEEL FIP identifies the process to document and control PCNs. These PCNs are approved by the facility line manager and FQAO and promptly transmitted to the SPO for SQAO and SPM signature before initiation of the activity. PCNs initiated by the SPO are controlled in accordance with the SPO implementation plan. Example PCN forms are included in FIPs or procedures and include the following information:

- Title or heading, Program Change Notice
- Drum or sample identification number, if applicable
- Reason for the deviation from the requirement
- A description of the variation from an approved process or procedure
- A description of special equipment or personnel required
- Initiator's signature and date
- Facility line manager's signature and date (for facility initiated PCNs)
- FQAO's signature and date (for facility initiated PCNs)
- SPM's signature and date

- SQAO's signature and date

The person initiating the PCN completes the PCN form and forwards it to the SQAO. The SQAO assesses the significance of the PCN and determines whether changes to applicable procedures and further notifications are necessary. The SQAO monitors the status of PCNs until close-out actions are completed.

2.1.4 Quality Improvement

TWCP personnel detect and prevent quality problems and ensure quality improvement as described in Sections 2 and 3.1 of this QAPjP. The SQAO accomplishes this by analyzing the quality related information discussed in Sections 2 and 3.1 of this QAPjP. FIPs describe facility processes for detecting and preventing quality problems and ensuring quality improvement. Quality improvement criteria are specified in the QAPD.

2.1.5 Management Assessment

TWCP management at all levels periodically assess the integrated TWCP and its performance. Management assessments focus on identifying, correcting, and preventing management and technical problems that hinder the achievement of the organization's objectives. Management assessments evaluate customer and employee perceptions relative to the following key areas:

- Organization mission and strategic objectives
- Employee roles in the organization
- Customer expectations and degree to which expectations are being met
- Opportunities for improving quality and cost-effectiveness
- Recognition and enhancement of human resource capabilities

Management assessment results are documented in writing and used as input to the quality improvement process. TWCP management assessment activities focus on the key areas identified above and are explained in the SPO implementation plan. These management assessments include all facilities.

2.1.6 Independent Assessment

All facilities that perform characterization activities are audited by the SPO at least annually. Also, facilities are subject to quarterly (every three months) surveillances performed by an independent assessment team assembled by the SQAO. At a minimum, these assessments will include a repeat of level 1 data review, validation, and verification for one randomly selected drum. These surveillances focus on performance of work in accordance with the requirements of this QAPjP, FIPs, and applicable procedures.

In addition, each facility may perform independent assessments of TWCP activities. The FIPs describe the personnel, roles, and responsibilities for these assessments. FQAOs report results of these assessments to the SQAO (see Section 2.2). The SPO is assessed annually by individuals independent of the TWCP SPO. All independent assessment results are reported to the SPM (as described in Section

2.2). The SQAO and FQAOs track assessment results and corrective actions. Independent assessments comply with the QAPD.

2.1.7 Trend Analysis

FIPs identify the specific quality-related information that will be analyzed to identify trends that adversely impact quality. The SQAO reports this trend information in the semiannual QA report.

2.2 Reports to Management

The SQAO prepares and transmits a semiannual QA report to the DOE-ID. The QA reports include the following information, as appropriate:

- Any QAPjP changes
- Identification of any significant QA problems, recommended solutions, and corrective actions
- An assessment of QC data collected during the period, including the frequency of repeated analyses, reasons they were repeated, and corrective actions
- Discussions of whether QAOs have been met and any resulting impact on decision making
- Limitations on the use of measurement data
- Status of PDP sample results
- Results of any audits, assessments, and surveillances conducted during the period
- All nonconformances and PCNs that could impact the results of the work, as described in Section 2.1.2 of this QAPjP
- QA trend analysis results

The report may include information provided in facility QA reports. FQAO reports are described in the FIPs. The SQAO provides copies of this semiannual QA report to facility program managers and the SPM. The SQAO also sends a copy of QA reports (including facility QA reports) to the SDCO, who maintains them as TWCP records.

2.3 Performance Demonstration Program

The RWMC, ACL, and ECL participate in the PDP, as specified in Section 2.3 of the QAPP and summarized in Table 2-1. FIPs describe how compliance with this requirement is achieved. The INEEL SPO is not involved in administration of the PDP.

2.4 Initial and Continuing Procedure Performance Demonstration

ACL, ECL, and RWMC personnel demonstrate acceptable performance of procedures before analyzing any samples, and semiannually thereafter. Demonstration of acceptable procedure performance is initially performed on seven replicate method performance samples; thereafter, four method performance samples are analyzed semiannually, as required in Sections 11 through 15. ACL, ECL, and RWMC implementation plans address how compliance with these requirements is achieved.

2.5 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 2-2 lists the major procedures pertinent to this section of the QAPjP. FIPs identify other procedures.

Table 2-1. Performance Demonstration Program participation.

INEEL facility	PDP plan
RWMC	*Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program* (NDA PDP Plan) (DOE 1995c or most current revision)
ECL and RWMC	*Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases for the TRU Waste Characterization Program* (Gas PDP Plan) (DOE 1995b or most current revision)
ACL	*Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes* (Solid PDP Plan) (DOE 1995d or most current revision)

Table 2-2. Section 2 implementing procedures.

Procedure number	Title
MCP-2531	*PCN and Nonconformance Reporting*
MCP-598	*Process Deficiency Resolution*
MCP-2532	*Internal Audits*
MCP-2533	*QA Reports to Management*
MCP-2534	*Level 1 Surveillances*
MCP-2992	*QA Program Surveillances*
MCP-1757	*TWCP Management Assessments*

3. DATA VALIDATION, USABILITY, AND REPORTING

Data generated during TWCP testing, sampling, and analytical activities are reviewed, validated, verified, and reported. Data generated from testing, sampling, and analytical operations are reported to the SPO as testing, sampling, analytical, or on-line batch data reports. RWMC personnel collect headspace gas samples in SUMMA® canisters in sampling batches and perform RA or RTR on drums in testing batches. RWMC personnel sample/analyze headspace gas from drums in on-line batches using fourier transform infrared spectroscopy (FTIRS) and residual gas analysis (RGA). ANL-W personnel core samples in sampling batches. ECL and ACL personnel analyze samples in analytical batches. Sections 7 and 8 include the requirements for sampling batch data reports, Sections 9 and 10 include the requirements for testing batch data reports, and Section 3.4.1 includes the requirements for analytical and on-line batch data reports. The Definitions section provides definitions for sampling, testing, analytical, and on-line batches.

3.1 Data Review, Validation, and Verification

To ensure TWCP data meet the level of quality required in the QAPP, INEEL TWCP personnel review, validate, verify, and report data at two levels: level 1 (data generation) and level 2 (project level). Figure 3-1 depicts the overall data validation and reporting flow. The SPO implementation plan describes the SPO process for data validation, verification, and reporting.

The INEEL has recently implemented an electronic data processing system called the Transuranic Reporting and Inventory Processing System (TRIPS). The TRIPS complies with all QAPP data requirements and functions as an alternative to the paper based process. All data processing activities described in this QAPJP are performed regardless of the media (paper or electronic).

TRIPS will be implemented into the INEEL TWP in phases. Presently, electronic data validation and reporting have been implemented at RWMC for RTR, RA, and online gas sampling/analysis processes; and at the SPO for Level 2 data validation.

Future electronic data processing capabilities for the other characterization processes (manual gas sampling, ECL, ACL, and ANL-W visual and sludge sampling) are in the planning/development stages.

Table 3-1 indicates the present implementation status of TRIPS.

TABLE 3-1. Facility Data process.

Characterization Process	Facility	Data Process
RTR	RWMC	Electronic (TRIPS)
RA	RWMC	Electronic (TRIPS)
Online Gas Sampling/Analysis	RWMC	Electronic (TRIPS)
Manual (Summa) Gas Sampling	RWMC	Paper
HS Gas Analysis	ECL	Paper

Solid sample analysis	ACL	Paper
Visual Exam	ANL-W	Paper
Solid Sample Collection	ANL-W	Paper

In the TRIPS, data are entered, signed electronically (password protected) and progressively promoted through the Level 1 and Level 2 review/approval process. TRIPS replaces paper signatures with electronic approvals.

Data are collected by the operator, entered into TRIPS, approved electronically and promoted for Level 1 review and validation. The data are progressively reviewed at Level 1 by the technical supervisor, independent technical reviewer, and facility QA officer using electronic data validation checklists. Data are promoted to one reviewer at a time. If data are approved, the data are automatically promoted to the next reviewer. If the data are rejected, the data are automatically demoted to the data generator for resolution of the problem.

When the data are approved by Level 1 QA, they are promoted to Level 2 and the progressive review process is repeated at the SPO by the SQAO and SDVO. The SQAO and SDVO complete electronic data validation checklists.

TRIPS data processing is controlled by implementing procedures as described in the RWMC FIP and SPO FIP.

3.1.1 Level 1: Data Generation

Facility line management ensure TWCP personnel perform the following minimum requirements for raw data collection and management:

- Sign and date all raw data in permanent, reproducible ink; or equivalent electronic signature.
- Record clearly, legibly, and accurately all data in field and laboratory records (e.g., bench sheets, logbooks, TRIPS input screens), and include applicable sample identification numbers.
- Line out, initial, and date all changes to original data or perform comparable electronic changes. Include justification for changing the original data. Do not obliterate or otherwise disfigure original data so as not to be readable.
- Transfer and reduce all data completely and accurately from field and laboratory records.
- Maintain all field and laboratory records in permanent files according to National Enforcement Investigation Center (NEIC) guidelines.

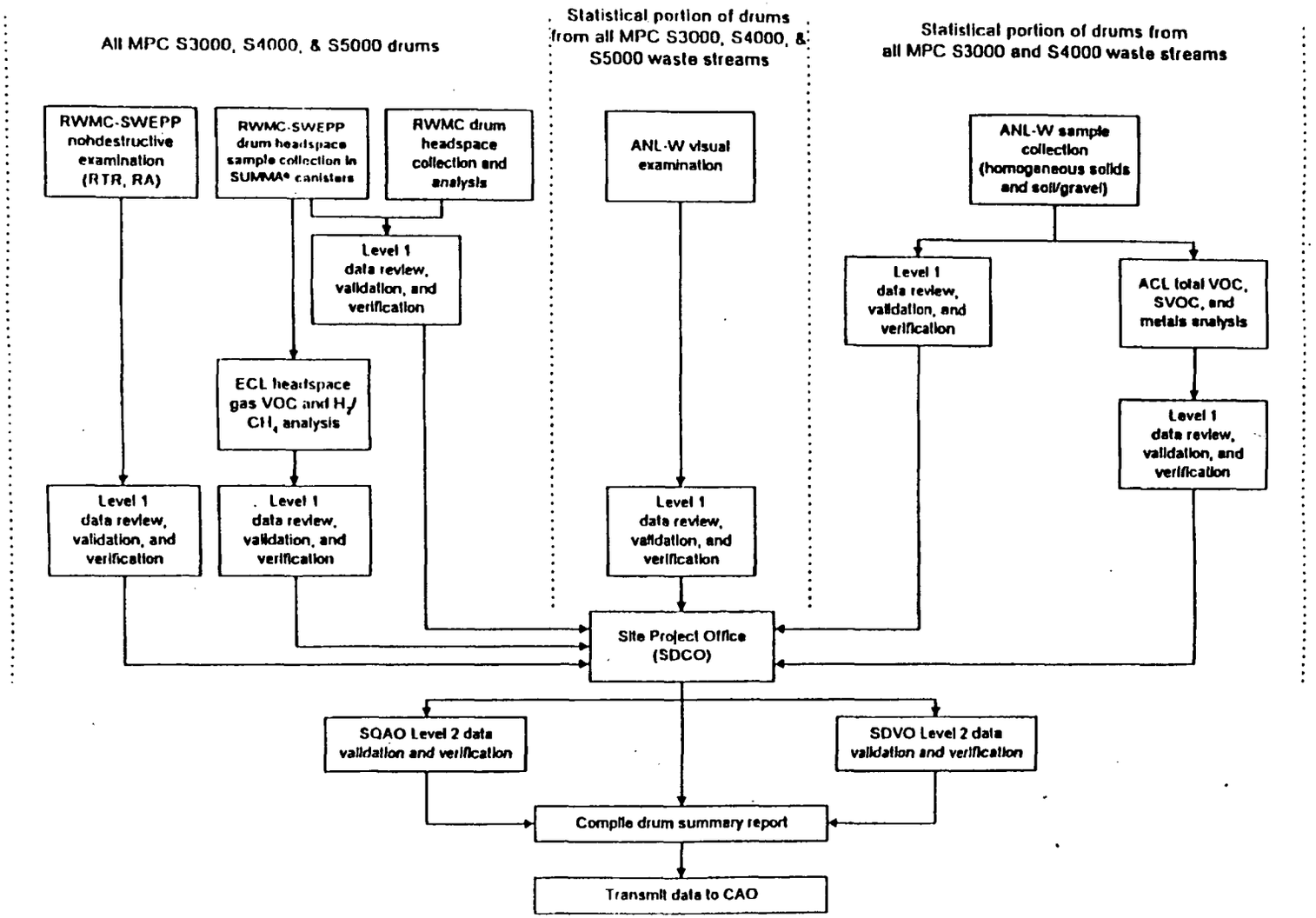


Figure 3-1. Overall flow of data for the TWCP.

- Organize data into a standard format for reporting purposes (testing, sampling, analytical, or on-line batch data report), as outlined in specific testing, sampling, and analytical techniques (Sections 7 through 15).

- Store all electronic and video data in accordance with the QAPD requirements to ensure waste container, sample, and associated QC data are readily retrievable (Section 1.7).

Facility line management ensure level 1 data review, validation, and verification is performed per the FIPs and includes signature release from qualified independent technical reviewer(s), technical supervisor(s), and a QA officer, as specified below. Facility personnel review, validate, and verify data using checklists (examples provided in FIPs or referenced implementing procedures) that address all items included in this section. The completed checklists are transmitted with batch data reports (or electronic versions) to the SPO.

- Facility line management ensure 100% of the data receives an independent technical review by an individual other than the data generator who is qualified to have performed the initial work. The reviewer releases the data as evidenced by signature and consequently ensures the following:

  - Data generation and reduction were conducted in a technically correct manner in accordance with the methods used. Data are reported in the proper units and correct number of significant figures.

  - Calculations were verified by a valid calculation program, a spot check of verified calculation programs, and/or 100% check of all hand calculations.

  - All PCNs from an accepted method and the rationale for the variations were documented and approved (Section 2.1.3).

  - The data were reviewed for transcription errors.

  - The testing, sampling, or analytical data QA documentation (testing, sampling, analytical, or on-line batch) is complete and includes raw data, calculation records, sample chain-of-custody (COC) forms, calibration records, QC sample results, and gas canister sample tags (if applicable).

  - QC sample results are within established control limits and if not, the data are appropriately qualified (Sections 7 through 15).

  - Reporting flags were assigned correctly.

  - Sample holding time and preservation requirements were met or exceptions documented.

  - Radiography tapes were reviewed, at a minimum, for one out of 10 waste containers against the data reported on the radiography form to ensure the data are correct and complete.

- Field sampling records are complete and include the documentation specified in Section 6.1 of this QAPjP.

- Facility line management ensure 100% of the data receives combined QA and technical supervisory signature release for each testing, sampling, and analytical batch. These releases ensure the following:

  - The data are technically reasonable based on the technique used.

  - All data received independent technical review with the exception of radiography tapes, which received periodic technical review as specified above.

  - The testing, sampling, or analytical data QA documentation (testing, sampling, analytical, or on-line batch) is complete and includes raw data, calculation records, sample COC forms, calibration records, QC sample results, and gas sample canister tags (if applicable).

  - Sample holding time requirements were met or exceptions documented.

  - Field sampling records are complete and include the documentation specified in Section 6.1 of this QAPjP.

  - Independent technical and technical supervisory reviews were performed as evidenced by the appropriate signature releases.

  - The testing, sampling, or analytical data QA documentation (testing batch, sampling batch, analytical batch, or on-line batch) is complete as appropriate for the point of data generation (i.e., radiography, RA, sampling, and analysis).

  - Sampling and analytical QC checks were properly performed. QC criteria not met are documented.

  - QAOs were met according to the methods outlined in Section 3.2.

If minor data package errors or omissions are identified, the FQAO and facility line management evaluate the nature of the problem and the data package is revised as necessary. If the problem cannot be rectified by correcting the data package, or if the problem is of a recurring nature, an NCR is generated as described in Section 2.1.2 of this QAPjP. After data packages undergo level 1 review, validation, and verification, they are forwarded to the SPO along with the required signature releases.

3.1.2 Level 2: Site Project Office Data Validation and Verification

Data validation and verification at this level involves scrutiny and signature release from the SPM, the SQAO, and SDVO to ensure minimum requirements are met for each drum. If minor data package errors or omissions are identified, or if the problem is of a recurring nature, the nature of the problem is evaluated and the data package is revised as necessary. If the problem cannot be rectified by correcting the data package, an NCR is generated as described in Section 2.1.2 of this QAPjP.

By SPM, SQAO, and SDVO signature release of 100% of the testing, sampling, and analytical data, SPO personnel ensure the following:

- Data generation level independent technical, technical supervisory, and QA officer review, validation, and verification were performed as evidenced by the appropriate signature releases.
- Testing, sampling, analytical, and on-line batch data review checklists are complete.
- Testing, sampling, analytical, and on-line batch data reports are complete and data are properly reported (i.e., data are reported in the correct units, with the correct number of significant figures and with qualifying flags).
- Reconciliation with the DQOs was performed (Section 3.3).
- Sampling batch QC checks (e.g., equipment blank, field duplicate, field reference standard) were properly performed and meet the established QAOs (Sections 7 and 8).
- Testing batch QC checks (e.g., replicate scans, measurement system checks, replicate counts) were properly performed (Sections 9 and 10).
- Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix spikes, matrix spike duplicates, laboratory control samples) were properly performed and meet the established QAOs (Sections 11 through 15).
- On-line QC checks (on-line duplicates, on-line blanks, on-line control samples) were properly performed and meet the established QAOs (Section 12).
- Proper procedures were followed to ensure representative samples of headspace gas and homogeneous solids and soil/gravel were taken.
- Radiography data are complete and acceptable based on the video data review of one drum per batch, at a minimum. For drums visually examined, a comparison with the RTR results has been performed and documented.
- RA data are complete and acceptable.
- All laboratory holding requirements were met.
- Data reduction conducted at facilities is correct.
- 100% of the batch data report has been checked for correctness and completeness.

The SQAO and SDVO ensure level 2 data validation and verification to meet the requirements specified above. Level 2 data validation, verification, and reporting are further described in the SPO implementation plan. In association with level 2 data validation and verification, the SDVO prepares a Data Validation Summary and the SQAO prepares a Site Project QA Officer Summary for each batch data package. After batch data packages are validated, SPO personnel notify the data generator. SPO

personnel also perform quarterly repeats of level 1 data review, validation, and verification in accordance with the SPO implementation plan.

3.2 Validation Methods

TWCP personnel validate data (qualitative as well as quantitative) to ensure data used for WIPP compliance programs are of known and acceptable quality. Validation includes a quantitative determination of precision, accuracy, completeness, comparability, and method detection limit (as appropriate) for analytical data (headspace hydrogen, methane, and VOC data and total VOC, SVOC, and metals data). Quantitative data validations are performed in accordance with the conventional procedures outlined below [Equations (3-1) through (3-9)] including comparison with the quantitative determinations to the QAOs specified in Sections 11 through 15. Section 9 of the QAPP presents validation methods used to determine compliance with the QAOs for RA.

The qualitative data or descriptive information generated by radiography is not amenable to statistical analysis. However, radiography and visual examination are complementary techniques yielding similar data for determining the matrix parameter category and waste material parameter weights of waste present in a drum. Therefore, the SQAO uses visual examination results to verify the matrix parameter category and waste material parameter weights determined by radiography, as described in Section 10.

The applicable facility ensures representativeness is achieved through proper implementation of sampling procedures. Representativeness of drums from IDCs subject to visual examination and homogeneous solids and soil/gravel sampling and analysis are validated through documentation that a true random sample was collected. The SPM documents that the selected drums from within an IDC were randomly selected (see Section 5 for details).

3.2.1 Precision

Precision is a measure of the mutual agreement among multiple measurements of a single analyte, either by the same or different methods. Precision is expressed either as the relative percent difference (RPD) for duplicate measurements or as the percent relative standard deviation (%RSD) for three or more replicate measurements. For duplicate measurements, the precision expressed as the RPD is calculated as follows:

$$RPD = \frac{C_1 - C_2}{\frac{(C_1 + C_2)}{2}} * 100 \qquad (3\text{-}1)$$

where C_1 and C_2 are the two values obtained by analyzing the duplicate samples and C_1 is the larger of the two observed values.

For three or more replicate measurements, the precision expressed as the %RSD is calculated as follows:

$$\%RSD = \frac{s}{\bar{y}} * 100 \qquad (3\text{-}2)$$

where s is the standard deviation and $\bar{y}$ is the mean of the replicate sample analyses.

The standard deviation, s, is calculated as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n-1}} \qquad (3\text{-}3)$$

where y_i is the measured value of the i^{th} replicate sample analysis measurement, and n equals the number of replicate analyses.

Another aspect of precision is associated with analytical equipment calibration. In these instances, the percent difference (%D) between multiple measurements of an equipment calibration standard is calculated as follows:

$$\%D = \left| \frac{C_1 - C_2}{C_1} \right| * 100 \qquad (3\text{-}4)$$

where C_1 is the initial measurement and C_2 is the second or other additional measurement.

3.2.2 Accuracy

Accuracy is the degree of agreement between a measured analyte concentration (or the average of replicate measurements of a single analyte concentration) and the true or known concentration. Accuracy is determined as the percent recovery (%R).

For situations where a standard reference material is used, the %R is calculated as follows:

$$\%R = \frac{C_m}{C_{srm}} * 100 \qquad (3\text{-}5)$$

where C_m is the measured concentration value obtained by analyzing the sample and C_{srm} is the "true" or certified concentration of the analyte in the sample.

For measurements where matrix spikes are used, the %R is calculated as follows:

$$\%R = \frac{S - U}{C_{sa}} * 100 \qquad (3\text{-}6)$$

where S is the measured concentration in the spiked aliquot, U is the measured concentration in the unspiked aliquot, and C_{sa} is the actual concentration of the spike added.

3.2.3 Method Detection Limit

The method detection limit (MDL) is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL for all quantitative measurements except for those using FTIRS is defined as follows:

$$MDL = t_{(n-1,1-\alpha=.99)} * s \quad (3\text{-}7)$$

where $t_{(n-1,1-a=.99)}$ is the *t*-distribution value appropriate to a 99% confidence level and a standard deviation estimate with *n-1* degrees of freedom, *n* is the number of observations, and *s* is the standard deviation of replicate measurements. This equation is also used to determine the instrument detection limit (IDL) for total metals analysis.

For headspace gas analysis using FTIRS, MDL is defined as follows:

$$MDL = 3s \quad (3\text{-}8)$$

where *s* is the standard deviation. Initially, a minimum of seven samples of ambient air or seven blanks are used to establish the MDLs. MDLs are constantly updated using the results of the on-line control sample.

3.2.4 Completeness

Completeness is a measure of the amount of valid data (see completeness definition) obtained from the overall measurement system compared to the amount of data collected and submitted for analysis. Completeness is expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Completeness, expressed as the percent complete (%C), is calculated as follows:

$$\%C = \frac{V}{n} * 100 \quad (3\text{-}9)$$

where *V* is the number of valid analytical results obtained and *n* is the number of samples submitted for analysis.

3.2.5 Comparability

Comparability is the degree to which one data set can be compared to another. Facility personnel ensure that data generated at different facilities over the lifetime of the TWCP are comparable through the use of standardized approved testing, sampling, and analytical techniques, and by meeting the QAOs specified in Sections 7 through 15 of the QAPP.

3.3 Reconciliation with Data Quality Objectives

The SPM assesses whether data of sufficient type, quality, and quantity were collected. The SPM determines whether the variability of the data set is small enough to provide the required confidence in the results. The SPM also determines whether, based on the desired error rates and confidence levels, a

sufficient number of valid data points were determined. In addition, the SPM documents that random sampling of drums was performed for the purposes of IDC characterization. In association with the data validation and verification described above, the SDVO, as the SPM designee, ensures all data reported meet the DQOs provided in Section 1.5 of the QAPP.

For each IDC characterized, the SDVO determines whether sufficient data were collected to determine:

- Matrix parameter category
- Waste material parameter weights
- Average mass and activity of each radionuclide of concern
- Whether each drum of waste is TRU or low-level radioactive waste
- Average concentration of H_2, CH_4, and each VOC in the headspace gas of drums in the IDC
- Total mass of H_2, CH_4, and VOCs in the headspace gas of the IDC
- Potential flammability of TRU waste headspace gases
- Mean concentrations, UCL_{90} for the mean concentrations, and standard deviations for headspace as VOCs included in the RCRA descriptions for F001, F002, F003, and F005 listed wastes
- Mean concentrations, UCL_{90} for the mean concentrations, standard deviations, and number of solid number of drums was visually examined to determine with a samples collected for VOCs and SVOCs included in the RCRA descriptions for F001, F002, F003, F004, and F005; and toxicity characteristic (TC) VOCs, SVOCs, and metals in the IDC (if applicable)
- Total masses of VOCs, SVOCs, and metals in the IDC
- Whether the IDC is listed for the presence of spent solvents under 40 CFR Part 261, Subpart D
- Whether the IDC exhibits a TC under 40 CFR Part 261, Subpart C
- Whether the IDC can be classified as hazardous or nonhazardous at the 90% confidence level
- Whether a sufficient reasonable level of certainty that the UCL_{90} for the miscertification rate is less than 14%

The SDVO performs the DQO reconciliation and reports results to the SPM. The SPO implementation plan describes the specifics of this process. If the SPM determines insufficient data were collected to make the determinations listed above, facilities collect additional data.

The SPM evaluates and reports waste characterization data from the analysis of homogeneous solids and soil/gravel following the statistical procedure presented in Section 5. The procedure, which calculates UCL_{90} values, is used to assess compliance with the DQOs in Section 1.5 as well as with RCRA regulations, and is applied to all laboratory analytical data for total VOCs, total SVOCs, and total metals. For RCRA regulatory compliance (40 CFR §261.24), the SDVO compares data from the analysis of the appropriate metals and organic compounds to the TC levels expressed as total values. These total values are considered the regulatory threshold limit (RTL) values for the program listed in Table 3-1. The SDVO verifies the assignment of EPA hazardous waste numbers for the presence of spent solvents (40 CFR §261.31), data from the analysis of the appropriate headspace VOCs, total VOCs, and total SVOCs will be compared to the program required quantitation limits (PRQLs) in Tables 12-1, 13-1, and 14-1. The SPO implementation plan identifies the SPO procedures that address this data evaluation process.

3.4 Data Reporting

3.4.1 Level 1: Data Generation

Facility personnel assign a unique identification number to every sampling, testing, analytical, and on-line batch and every field, laboratory, and on-line sample. Sampling facility personnel assign identification numbers to samples (i.e., gas samples, homogeneous solids samples, and soil/gravel samples) as described in Section 6.2 of this QAPjP. Each facility assigns batch numbers as described in Section 6.5. Facility personnel assign unique serial numbers to batch data reports and ensure each page is numbered at the bottom. The serial number used for data reports may be the same as the batch number.

Facility personnel transmit a hard copy (or electronic equivalent) of all testing, sampling, analytical, and on-line batch data reports and data review checklists to the SPO. FIPs or referenced procedures include report forms and checklists required by the applicable testing, sampling, and analytical methods. The batch data reports include the signature releases that document the data generation level review, validation, and verification as described in Section 3.1 of this QAPjP. FIPs identify the procedures that implement level 1 data reporting requirements.

RWMC personnel submit final sampling batch data reports and testing batch data reports to the SDCO within 28 days of sampling or testing the last drum in the batch. ANL-W personnel submit the final sampling batch data report to the SDCO within 28 days of collection of the last sample in the sampling batch. Sections 7 and 8 identify batch data report requirements for sampling methods and Sections 9 and 10 identify those for testing methods. FIPs or SOPs include the format for batch data reports.

Analytical laboratory personnel at the ECL, ACL, and RWMC (for on-line analysis) submit analytical batch data reports to the SDCO within 28 days of the validated time of sample receipt (VTSR) of the last sample in an analytical batch. RWMC personnel submit on-line batch data reports within 28 days of analyzing the last sample in an on-line batch. Analytical and on-line batch data report requirements are included in Sections 11 through 15.

3.4.2 Level 2: Site Project Office Data Reporting

The SDCO reports data on an individual drum basis to the CAO management and operating (M&O) contractor and CAO National TRU Program team using the WWIS as specified in the WIPP WAC. In addition to the reporting for individual drums, once an IDC is fully characterized, the SPM

prepares a Waste Stream Profile Form (WSPF). The SPM submits the WSPF, AK summary report, and the reconciliation with DQOs report to the CAO M&O contractor and CAO National TRU Program team. If the CAO or its M&O contractor requests hard-copy data reports, the INEEL submits these reports. The SPO data reporting process is described in the SPO implementation plan and referenced procedures. When a hard copy of the data package is requested by CAO, the SPM will ensure that the data will be compiled into a data package and reported as requested. The data package will include a cover page identifying the site and waste container numbers included in the data package; appropriate signature releases; table of contents; a narrative (as described in the QAPP); a table that relates sample numbers to waste container numbers; *Site Project QA Officer Summary; Data Validation Summary;* radiography and radioassay results; headspace gas hydrogen, methane, and VOC analytical results; and total VOC, SVOC, and metal analytical results for homogeneous solids and soil/gravel.

3.5 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 3-2 lists the major project level implementing procedures pertinent to this section of the QAPjP. FIPs identify data generation level procedures.

Table 3-1. TC levels expressed as RTL values in the waste.

Analyte	RTL Value (mg/kg)[a]
Metals[b]	
Arsenic	100
Barium	2000
Cadmium	20
Chromium	100
Lead	100
Mercury	4
Selenium	20
Silver	100
Semi-VOCs[b]	
Cresols	4000
1,4-Dichlorobenzene	150
2,4-Dinitrotoluene	2.6
Hexachlorobenzene	2.6
Hexachloroethane	60
Nitrobenzene	40
Pentachlorophenol	2000
Pyridine	100
VOCs[c]	
Benzene	10
Carbon tetrachloride	10
Chlorobenzene	2000
Chloroform	120
1,2-Dichloroethane	10
1,1-Dichloroethylene	14
Methyl ethyl ketone	4000
Pyridine	100
Tetrachloroethylene	14
Trichloroethylene	10
Vinyl chloride	4

a. The calculations assume 1) the maximum amount of material suggested by the TCLP is used, 2) wastes are 100% solid (no liquid fraction), 3) the maximum amount of extraction fluid is used, and 4) all analytes are 100% soluble in the extraction fluid.

b. For metals and SVOCs, RTL value (mg/kg) = (TC level, mg/L) (volume of extraction fluid, 2 L)/(weight of sample, 0.100 kg)

c. For VOCs, RTL value (mg/kg) = (TC level, mg/L) (volume of extraction fluid, 0.5 L)/(weight of sample, 0.025 kg)

Table 3-2. Section 3 implementing procedures.

Procedure number	Title
MCP-2536	*Project Level Data Validation and Verification by the SDVO*
MCP-2529	*Drum Data Review by Site QA Officer*
MCP-2527	*DQO Reconciliation at SPO Level*
MCP-2539	*Data Report Preparation*
MCP-2530	*SQAO Drum Data Review Checklists*
MCP-2996	*Electronic Data Review by SQAO*
MCP-2997	*SQAO Electronic Data Review Checklists*
MCP-2995	*Project Level Electronic Data Validation and Verification by the SDVO*

4. ACCEPTABLE KNOWLEDGE

As described in the QAPP, acceptable knowledge is used to assign waste containers to waste streams and to assign the appropriate EPA hazardous waste numbers to each waste stream. Several different classification systems are utilized at INEEL. Among these are IDCs, TRUCONs, and MCPs. The most specific description is provided by the IDC, which was the item description code assigned by Rocky Flats. The INEEL uses the detailed IDC information to define waste streams. Similar IDCs may be grouped into a waste stream using acceptable knowledge and verified by characterization data. Occasionally, a single IDC could be split into multiple waste streams.

SPO personnel initially evaluate the information available at an IDC level. Based on this analysis, IDCs are assigned to the appropriate waste streams. MCPs, EPA hazardous waste numbers, waste material parameters, and radionuclides present are then determined based on acceptable knowledge. This process is described in Section 5.

4.1 Quality Assurance Objectives

SPO personnel use acceptable knowledge documentation (e.g., records; management, procedural, and quality controls associated with the waste generating processes; past sampling and analytical data; material inputs to the waste generating process; time period of waste generation) to characterize waste in accordance with the QAPP. This primarily qualitative information is assessed according to the QAOs established by the CAO to ensure DOE sites consistently apply the acceptable knowledge process. To demonstrate compliance with the QAOs, TWCP personnel:

- Measure accuracy based on the percentage of drums that require reassignment to a new MPC and/or designation of different EPA hazardous waste numbers and the reevaluation of acceptable knowledge and sampling and analysis data.

- Ensure completeness by compiling 100% of the required TRU waste management program information and TRU IDC information described in Sections 4.3.1 and 4.3.2 for each IDC.

- Ensure comparability of data by meeting the training requirements and complying with the minimum standards outlined for procedures used to implement the acceptable knowledge process. SPO personnel assign MPCs and EPA hazardous waste numbers and identify waste material parameters and radionuclides in accordance with the QAPP requirements.

- Ensure representativeness by obtaining, evaluating, and documenting acceptable knowledge information in compliance with the QAPP standards. SPO personnel assess and document the limitations of the acceptable information used [e.g., purpose and scope of the information, date of publication(s), type and extent to which waste parameters are addressed, and limitations of information in identifying hazardous waste(s)].

4.2 Procedural Requirements

SPO personnel compile the minimum acceptable knowledge documentation in an auditable record; confirm acceptable knowledge information using radiography and headspace gas and solidified waste sampling and analysis; and assess acceptable knowledge records for completeness.

4.2.1 Assembling Acceptable Knowledge Documentation

Procedures identified in the SPO implementation plan describe the SPO process for assembling acceptable knowledge documentation. TWCP personnel ensure that acceptable knowledge documentation:

- Is compiled in an auditable record and includes a road map for all applicable information.
- Correlates specific IDC information to the overview of the facility and TRU waste management operations in the context of the INEEL mission.
- Describes correlations between IDCs, with regard to the time of generation, waste generating processes, and site-specific facilities.
- Includes a reference list that identifies documents, databases, QA protocols, and other sources of information that support acceptable knowledge information.

The procedures also describe or provide the following:

- The specific methodology used to assemble acceptable knowledge records, including the origin of the documentation, how it will be used, and any limitations associated with the information (e.g., identify the purpose and scope of a study that included limited sampling and analysis data)
- The process for assembling and evaluating available documentation in the following priority: a) relevant information from published documents and controlled databases; b) unpublished data; c) internal procedures and notes, such as logbooks; and d) correspondence (e.g., memoranda, letters, telephone logs, and interviews)
- The process for identifying the physical form of the waste for use in assigning an appropriate MPC to each IDC
- The process for identifying the waste material parameters and radionuclides present in each IDC
- The process for identifying hazardous wastes and assigning the appropriate EPA hazardous waste numbers to each IDC
- The processes used to ensure unacceptable wastes are identified and segregated and that waste is certified for shipment to the WIPP facility
- The management controls used to ensure nonconforming items are documented and managed
- A reference to radiography and visual examination procedures that list nonconforming items the operator verifies are not present in each drum of waste (i.e., corrosives, ignitables, reactives, incompatible waste)

- The process for confirming acceptable knowledge in accordance with Section 4.4.3 of the QAPP

- A cross reference to the applicable matrix parameter summary category (i.e., S3000, S4000, and S5000) for each IDC to verify all required confirmation data have been evaluated and proper EPA hazardous waste numbers have been assigned

- The process for evaluating acceptable knowledge information and resolving any discrepancies in documentation

- For debris IDCs, the process for documenting changes to matrix parameter categories, IDC assignment, and any associated EPA hazardous waste numbers based on material composition

- For all IDCs, the process for reevaluating acceptable knowledge information if radiography or visual examination results in the reassignment of a different MPC [e.g., Plastic/Rubber (S5310) versus Paper/Cloth (S5330)]

- For all IDCs, the process for reassigning waste to a different IDC and assigning appropriate EPA hazardous waste numbers

- How the acceptable knowledge data is used to assign waste containers to waste streams (see Section 5).

SPO personnel develop documentation to demonstrate consistency in assigning MPCs and EPA hazardous waste numbers, and in determining waste material parameters and radionuclides to meet QAPP requirements and to defend these assignments and determinations to independent auditors. The procedures describe the following steps in the process for assembling and using acceptable knowledge:

- Compiling all of the required information, including any acceptable knowledge information, regarding the materials and processes that generate a specific IDC, in an auditable record

- Reviewing the required information to determine the appropriate MPC

- Reviewing the required information to determine the waste material parameters and radionuclides present

- Reviewing the required information to determine whether the waste is listed under 40 CFR Part 261, Subpart D and assign all listed EPA hazardous waste numbers

- Reviewing the required information to determine whether the waste may contain hazardous constituents included in the toxicity characteristics specified in 40 CFR Part 261, Subpart C. If an identified toxicity characteristic contaminant is not included as a listed waste, the toxicity characteristic EPA hazardous waste number is assigned to the IDC. Unless data are available from the sampling and analysis of a representative sample of the IDC that demonstrates the concentration of the constituent in the waste is less than the toxicity characteristic regulatory level, no judgement is made regarding the concentration of the

constituent. When analytical data are not available, the toxicity characteristic EPA hazardous waste number for the identified hazardous constituent is applied to the IDC.

- The SPM (or designee) ensures all potential EPA hazardous waste numbers are assigned to the IDC

- Identifying the specific drums of retrievably stored waste in each IDC and correlating the IDC to the mandatory acceptable knowledge information (Sections 4.3.1 and 4.3.2)

- Identifying data used for assigning specific waste containers to waste streams (See Section 5).

4.2.2 Confirmatory Testing

SPO personnel use waste characterization results to confirm acceptable knowledge information in accordance with Section 4.2.2 of the QAPP. Waste characterization methods used to confirm acceptable knowledge information are summarized below and described in Sections 7 through 15 of this QAPjP.

4.2.2.1 Radiography and Visual Examination. SPO personnel use RTR and visual examination results to confirm the MPC and waste material parameters identified using acceptable knowledge. RWMC personnel perform RTR on retrievably stored wastes to confirm the MPC. If waste needs repackaging, ANL-W personnel perform visual examination of the waste during repackaging and the SDVO uses these results (rather than RTR) to confirm acceptable knowledge. SPO personnel assign the toxicity characteristic EPA hazardous waste numbers (to debris wastes) based on the presence of hazardous constituents, regardless of quantity or concentration.

4.2.2.2 Sampling and Analysis. SPO personnel use headspace gas data to confirm acceptable knowledge concerning the presence or absence of F-listed solvents in accordance with the QAPP. SPO personnel may use headspace gas data to assist in confirming the characterization of waste contaminated with F-listed solvents by the "mixture rule" (e.g., solvent-contaminated rags mixed with other waste materials). SPO personnel collect documentation to support any determination that organic constituents are associated with packaging materials or other uses not consistent with solvent use. If the source of the detected solvents cannot be identified, the appropriate spent solvent EPA hazardous waste number is conservatively applied to the IDC (i.e., it is assumed the solvent was used for its solvent properties).

SPO personnel confirm the assignment of spent solvent EPA hazardous waste numbers (40 CFR §261.31) by evaluating the mean concentrations of each VOC detected in container headspace gas and/or the solidified waste matrix. The UCL_{90} for the mean constituent concentration is compared to the PRQL or RTL for the constituent. If the UCL_{90} for the mean constituent concentration is greater than or equal to the PRQL or RTL, SPO personnel reevaluate acceptable knowledge information and determine the potential source of the constituent.

If the source of the constituent is identified as spent solvent used in the process or is determined to be the result of mixing a listed waste with a solid waste during waste packaging, SPO personnel either 1) assign the applicable listed spent solvent EPA hazardous waste number to the entire IDC, or 2) segregate the drums containing detectable concentrations of the solvent into a separate IDC and assign applicable EPA hazardous waste numbers. SPO personnel document, justify, and consistently define IDCs and assign EPA hazardous waste numbers based on INEEL permit requirements and other state-enforced agreements.

SPO personnel confirm the EPA hazardous waste numbers associated with S3000 and S4000 IDCs based on the results of the total VOC and SVOC analyses. If discrepancies between the results obtained from the solidified waste sampling and headspace gas sampling and analysis exist (i.e., a VOC is detected in the solidified waste but not in the headspace), SPO personnel use the solidified waste data to confirm acceptable knowledge and assign EPA hazardous waste numbers.

To determine the mean concentration of solvent VOCs, SPO personnel use all headspace gas data and solidified waste data for a IDC or IDC lot, including data qualified with a "J" flag (i.e., less than the PRQL but greater than the MDL) or qualified with a "U" flag (i.e., undetected). For data qualified with a "U" flag, SPO personnel use one-half the MDL to calculate the mean concentration. In the case of elevated dilutions (MDLs above the PRQL), SPO personnel use only the concentrations of detected constituents to calculate the mean for the purpose of assigning F-listed EPA hazardous waste numbers. SPO personnel do not remove EPA hazardous waste numbers established using acceptable knowledge if hazardous waste constituents are not detected in the headspace.

4.3 Acceptable Knowledge Information

SPO personnel ensure the required information listed in Sections 4.3.1 and 4.3.2 is included or referenced in the acceptable knowledge record. If the information is referenced, the record specifies locations where the required information can be found.

4.3.1 TRU Waste Management Program Information

The SPO implementation plan provides information related to TRU waste certification procedures and the types of documentation used to summarize acceptable knowledge. *Acceptable Knowledge Document for INEL Stored Transuranic Waste-Rocky Flats Plant Waste* (LMITCO 1998) (INEL-96/0280) provides an overview of the Rocky Flats Plant (RFP) and their TRU waste management operations during production of the waste. INEL-96/0280 establishes the basis for the more detailed TRU IDC information (Section 4.3.2), provides an overall perspective of TRU waste management operations at RFETS, and serves as a guide to IDC-specific information. The TRU waste management program information included in INEL-96/0280 clearly defines waste categorization schemes and terminology used as RFETS, provides a breakdown of the types and quantities of TRU waste stored at the INEEL, and describes how waste is tracked and managed at RFETS. INEEL specific information on current storage, inventories, mission statement, and certification processes are documented in the Program Plan for Certification of INEEL Contact-Handled Stored Waste (INEEL 1999f) (INEL-96/0345), the Project Plan for shipment of 3,100 m^3 of Certified Transuranic Waste to WIPP (INEEL 1997j) (PLN-129), and the semi-annual report to the State of Idaho, the RWMC HWMA/RCRA Permit, Permit Condition IIK-4 Report. The above acceptable knowledge documents include the following information.

- Map(s) of the site(s) with the areas and facilities involved in TRU waste generation, treatment, and storage identified
- Facility mission description(s) as related to TRU waste generation and management
- Description of the operations that generated TRU waste at the site(s)
- Waste identification or characterization schemes used at the facility(ies)

- Types and quantities of TRU waste generated
- Correlation of IDCs generated from the same building and process, as appropriate
- Waste certification procedures for wastes to be sent to the WIPP facility
- How specific waste containers are assigned to waste streams (see Section 5).

4.3.2 TRU Item Description Code Information

For each IDC, INEL-96/0280 includes all process information and data that support the acceptable knowledge used to characterize that IDC. At a minimum, the waste process information includes:

- Area(s) and building(s) from which the IDC was generated
- IDC volume and time period of generation
- Waste generating process described for each building
- Process flow diagrams
- Material inputs or other information that identifies the chemical and radionuclide content of the IDC and the physical waste form

INEL-96/0280 includes a summary traceable to referenced documents to identify all information sources and the basis and rationale for defining each IDC based on the parameters of interest. INEL-96/0280 also identifies and justifies any assumptions made in defining each IDC. IDC information is used to assign specific waste containers to waste streams (see Section 5).

4.3.3 Supplemental Acceptable Knowledge Documentation

INEL-96/0280 includes supplemental acceptable knowledge documentation for particular IDCs. This supplemental documentation may include, but may not be limited to, the following:

- Process design documents
- Procedures that describe raw materials or reagents, the process or experiment generating the waste, or the wastes generated and how they are managed at the point of generation
- Preliminary and final safety analysis reports and technical safety requirements
- Waste packaging logs
- Test plans or research project reports that describe reagents and other raw materials used in experiments
- Site databases

- Information from site personnel
- Industry documents
- Previous analytical data relevant to the IDC, including results from fingerprint analyses, spot-checks, or routine verification sampling
- Material Safety Data Sheets, product labels, or other product package information
- Sampling and analysis data from comparable or surrogate IDCs
- Laboratory notebooks that detail the research processes and raw materials used in an experiment

If supplemental documentation is included for a particular IDC, the specific, relevant information is identified and justification is provided for its use (e.g., identification of a toxicity characteristic). If discrepancies exist between supplemental information and the required documentation, the SDVO includes all potential EPA hazardous waste numbers to the subject IDC. The SDVO prioritizes the sources of information used to assign EPA hazardous waste numbers in terms of accuracy of information. Published documents and controlled databases are considered the most reliable information. Second priority is given to unpublished data, internal procedures, and notes. Correspondence (e.g., memoranda, letters, telephone logs, and interviews) are considered the least defensible. The pages from large documents (e.g., safety analysis reports) are flagged with the relevant information provided.

4.4 Acceptable Knowledge Program Control

To ensure compliance with the requirements for assembling, evaluating, and assessing acceptable knowledge, responsible SPO personnel receive training and follow procedures described or referenced in the SPO implementation plan. The SPO and facility personnel coordinate activities to segregate any waste drum determined to be unacceptable and resolve any discrepancies in acceptable knowledge before the waste is shipped.

4.4.1 Training

SPO personnel responsible for assessing information and resolving discrepancies associated with acceptable knowledge are trained and qualified to the following:

- WIPP Waste Analysis Plan, WIPP WAC, and QAPP requirements
- State and federal RCRA regulations associated with solid and hazardous waste determinations
- MPC and waste material parameter designations
- The nonconformance process, including discrepancy resolution and reporting (see Section 2.1.2)
- Procedures related to waste characterization using acceptable knowledge

4.4.2 Management Controls of Nonconforming Items

Nonconformances are managed as described in Section 2.1.2. FIPs (or related procedures) describe management controls used to ensure that nonconforming items or waste are segregated from certifiable populations. FIPs or SOPs include the following minimum elements associated with management controls:

Identification of the organization(s) responsible for compliance with management controls

Identification of the oversight procedures and frequency of actions to verify compliance with management controls

On-the-job training specific to management control procedures

A stop work process in the event of noncompliance with management controls

A nonconformance process that complies with the requirements in Section 2.1.2

A corrective action process that assesses the potential time frame of the noncompliance, the potentially affected waste population(s), and the reassessment and recertification of those wastes.

4.4.3 Confirmation of Acceptable Knowledge

Prior to shipping waste to the WIPP, the SPM (or designee) reports to CAO the required data associated with waste stream characterization including the results from radiography, visual examination, headspace gas sampling and analysis, solidified waste sampling and analysis; and the Waste Stream Profile Form on which EPA hazardous waste numbers for the waste stream are designated. The SDVO tracks and documents changes to waste determinations as part of the reconciliation with DQOs process (Section 3.3).

4.4.4 Resolution of Discrepancies

SPO personnel resolve discrepancies in acceptable knowledge documentation by including all available information in the auditable records and assigning all potential EPA hazardous waste numbers indicated by all of these records.

SPO personnel handle any unresolved discrepancies associated with hazardous waste determinations by reassessing the materials and processes that generated the waste. SPO personnel also reassess the EPA hazardous waste numbers assigned based on headspace gas sampling and analysis, radiography or visual examination, and solidified waste sampling and analysis. Discrepancies will be resolved prior to shipping waste.

4.4.5 Reevaluation of Acceptable Knowledge

SPO personnel reevaluate acceptable knowledge as described in the SPO implementation plan. They take the following minimum steps in the event a waste must be assigned to a different MPC based on radiography or visual examination:

- Review existing information based on the drum identification number and document all differences in EPA hazardous waste number assignments
- If differences exist in the EPA hazardous waste numbers that were assigned, they reassess and document all required acceptable knowledge information associated with the new designation
- Reassess and document all sampling and analytical data associated with the waste
- Reevaluate waste material parameter determinations and document any changes
- Reevaluate the radionuclide content and document any changes
- Verify and document that the reassigned MPC was generated within the specified time period, area, buildings, and waste generating process; and that the process material inputs are consistent with the waste material parameters identified during radiography or visual examination
- Record all changes to acceptable knowledge records
- If discrepancies exist in the acceptable knowledge information for the reassigned MPC, they complete an NCR, document the segregation of this drum, and define the corrective actions necessary to fully characterize the waste

4.5 Audits

The SQAO evaluates the acceptable knowledge process and IDC documentation through internal assessments. For these assessments, the SQAO assembles a team knowledgeable of RCRA regulations and EPA guidance regarding the use of acceptable knowledge for waste characterization, hazardous waste determinations, MPCs, waste material parameters, and QAPP requirements. Team members are independent of all INEEL TRU waste management operations. Internal assessments of acceptable knowledge include the following elements:

- Documentation of the process used to compile, evaluate, and record acceptable knowledge is available and implemented.
- Personnel training and qualifications are documented.
- All of the required acceptable knowledge documentation has been compiled in an auditable record.
- A procedure and records exist for assigning EPA hazardous waste numbers to IDCs.
- A procedure and records exist for assigning a MPC to an IDC.
- A procedure exists for determining waste material parameters present in an IDC.

- A procedure exists for determining the radionuclides present in an IDC.
- A procedure exists for grouping IDCs into waste streams
- A procedure exists for resolving inconsistencies in acceptable knowledge documentation.
- A procedure exists for confirming acceptable knowledge information through: a) radiography or visual examination, b) headspace gas sampling and analysis, and c) solidified waste sampling.
- Results of other audits of the TRU waste characterization programs at the facility are available in facility records.

Team members will evaluate all acceptable knowledge documentation associated with one debris IDC and one solidified IDC and will evaluate compliance with written site procedures for developing the acceptable knowledge record. Team members will evaluate the logic and defensibility of the acceptable knowledge documentation, the completeness and traceability of the information, clarity of presentation, degree of compliance with requirements of Section 4.0 of the QAPP, nonconformance procedures, and oversight procedures. Team members will review records for correlation to specific IDCs and the basis for making waste determinations. They will verify that sites include all required information and conservatively include all potential EPA hazardous waste numbers indicated by the acceptable knowledge documentation. They will include all deficiencies found in the acceptable knowledge documentation in a report to the SQAO.

4.6 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 4-1 lists the major technical procedures pertinent to this section of the QAPjP. The SPO implementation plan identifies additional procedures.

Table 4-1. Section 4 implementing procedures.

Document number	Title
MCP-2988	*Confirmation, Resolution and Reevaluation of Acceptable Knowledge Information Records*
MCP-2989	*Collection, Review and Management of Acceptable Knowledge Documentation*
INEL-96/0280	*Acceptable Knowledge Document for INEL Stored Transuranic Waste - Rocky Flats Plant Waste*

5. SAMPLING PROCESS DESIGN

TWCP personnel implement the sampling process design described in Section 5 of the QAPP for retrievably stored waste. All drums at the INEEL included in the TWCP undergo RTR examination, RA examination, and headspace gas sampling and analysis. INEEL TWCP personnel assess all drums in all MPCs (summary categories S3000, S4000, and S5000) for the presence of spent solvents based on headspace gas and total VOC sampling and analysis results, as appropriate. A randomly selected portion of the drums undergoes further examination based on the approaches presented in following sections of this QAPjP. The SPO establishes the selection process for drums in homogeneous solids and soil/gravel waste streams (summary categories S3000 and S4000) for RCRA characterization. The SPO establishes the selection process for drums from waste streams in all MPCs (summary categories S3000, S4000, and S5000) for visual examination to confirm the results of RTR.

5.1 Description of Acceptable Matrix Parameter Categories

Using acceptable knowledge, SPO personnel sort drums into waste streams. SPO personnel assign each waste stream an MPC based on the physical and chemical composition of the waste following the waste stream designations in the TWBIR and the *DOE Waste Treatability Group Guidance* (DOE 1995a). The MPCs are divided into three broad groups: homogeneous solids (summary category S3000), soil/gravel (summary category S4000), and debris wastes (summary category S5000). Sampling and analysis activities are based on the MCP summary category as described in Section 5.2. The SPO implementation plan documents these waste stream and MPC assignments. IDCs are also grouped into waste streams on the basis of TRUCONS, MPCs, and the presence of regulated substances at the INEEL. These waste streams are documented in the EDF titled "Identification of Transuranic Waste Stream" (INEEL 1997k).

5.2 Parameters, Rationale, and Test Methods

Once a waste stream is identified, TWCP personnel develop characterization information. INEEL TWCP personnel characterize all retrievably stored waste containers by RA (Section 9), RTR (Section 10), and headspace gas sampling and analysis (Sections 7, 11, and 12). In addition, the SPO establishes the selection process for retrievably stored homogeneous solids and soil/gravel for sampling and analysis as described in Section 5.4. All retrievably stored debris waste is characterized as described in Section 5.2.2.

5.2.1 Homogeneous Solids and Soil/Gravel

ANL-W personnel obtain samples of homogeneous solids (summary category S3000) and soil/gravel (summary category S4000) as described in Section 8. ACL personnel analyze these samples for total RCRA-regulated VOCs, SVOCs, and metals as described in Sections 13, 14, and 15.

5.2.2 Debris Wastes

TWCP personnel characterize debris wastes (for RCRA-regulated VOCs, SVOCs, and metals) based on acceptable knowledge. Acceptable knowledge procedures are described in Section 4 of this QAPjP.

Much of the planning for characterization will continue to take place at the IDC level, and hazardous waste numbers will be assigned at the IDC level. This ensures that information will continue to be available at the most specific level possible. However, IDC will be rolled up to the waste stream level for data reconciliation and waste stream profile forms. The term waste stream will be used throughout this section, but it should be recognized that some of the work will actually take place at the more specific IDC level.

5.3 Confirmation of EPA Hazardous Waste Numbers for Spent Solvents Using Headspace Gas Analysis

The SDVO evaluates headspace gas VOC analytical results from each waste stream to confirm and update EPA hazardous waste numbers for spent solvents (i.e., F001, F002, F003, F005) in accordance with the QAPP. The SDVO uses a statistical method for determining whether spent solvent EPA hazardous waste numbers should be added to an waste stream. The SDVO calculates the UCL_{90} for the mean concentration of each spent solvent constituent and compares it to the PRQLs (listed in Table 12-1). If the UCL_{90} for the mean is equal to or greater than the PRQL, the SDVO assigns the appropriate spent solvent EPA hazardous waste number to the waste stream if it has not already been assigned based on acceptable knowledge. If the UCL_{90} for the mean is less than the PRQL, the assignment of spent solvent EPA hazardous waste numbers remains unchanged. The SPO implementation plan describes the process used to document RCRA waste stream characterization and includes statistical equations used for evaluating the analytical results to confirm or assign EPA hazardous waste numbers. The SPO implementation plan includes a description of how SPO personnel transform data to normality (if needed) and handle less-than-detectable analytical results.

It may not be logistically feasible to characterize some waste streams in their entirety with a single sampling episode because of staging and transportation requirements. In these cases, an available portion, or lot, of an waste stream is characterized. The characterization then applies to the waste stream lot only.

5.4 Sampling Plan

Representativeness expresses the degree to which a sample or group of samples represents the population being studied and, for the TWCP, is based on the random selection of drums for both sampling and analysis of homogeneous solids and soil/gravel wastes and visual examination of all wastes. The SPM verifies that a true random sample is collected from a waste stream.

- The engineering design file (EDF), *Transuranic Waste Sample Plan for the INEEL* (EDF-RWMC-909) (INEL-96/295) (INEL 1998) describes the INEEL statistical approach used to select drums for characterization to obtain a preliminary estimate of the coefficient of variation (CV). The Phase II sampling program (EDF-RWMC-909) describes the strategy for randomly selecting drums of homogeneous solids and soil/gravel for final waste stream characterization. The EDF includes the following information:

- Name of the site to which the sampling plan applies

- Waste to which the sampling plan applies (i.e., retrievably stored and newly generated TRU waste)

- Specific facilities or waste-generating processes addressed
- Characterization activities to which the sampling plan applies (i.e., selection of containers for RCRA characterization and visual examination)
- Issues, operational constraints, or as low as reasonably achievable (ALARA) concerns related to container selection and retrieval
- Identification and summary description of waste streams to be sampled (including a citation of the basis used for identification and description)
- Correlation to applicable TWBIR waste streams
- Description or citation of procedures for obtaining data for preliminary estimates of mean, variance, and CV as described in Section 5.4.1 of the QAPP
- Identification of preliminary sample data (if available), justification for its use for a particular waste stream, and preliminary estimates (or citation of documents containing the preliminary estimates)
- Description or citation of a procedure for selecting CV and documenting the calculation of the number of containers to sample
- An indication that the number of containers sampled will be compared to the number of containers calculated from the CV for the sampling episode to determine if additional sampling is required
- Description or citation of procedure used for random selection of containers and sampling locations
- Description or citation of procedure for determining the miscertification rate, determining the number of containers to be selected, and randomly selecting containers for visual examination as described in Section 5.4.2
- Description or citation of procedures for interfacing with operations personnel regarding retrieval of selected containers
- Newly generated waste characterization strategies

The SPM is responsible for review and approval of this EDF.

It may not be logistically feasible to characterize some waste streams in their entirety with a single sampling episode because of staging and transportation requirements. In these cases, an available portion, or lot, of an waste stream is characterized. The characterization then applies to the waste stream lot only.

5.4.1 RCRA Characterization of Retrievably Stored Homogeneous Solids and Soil/Gravel

The TWCP statistical approach for RCRA characterization relies on acceptable knowledge to segregate drums of homogeneous solids and soil/gravel into waste streams so that it is reasonable to classify the entire waste stream as hazardous or nonhazardous. INEEL TWCP personnel sample and analyze a minimum of five containers in each waste stream. SPO personnel determine preliminary estimates of the mean concentration and variance for each RCRA-regulated toxicity characteristic contaminant in the waste by preliminary sampling of the waste stream or from previous sampling of the waste stream. SPO personnel calculate preliminary estimates of the CV for each contaminant using the equations in section 5.4.1 in the QAPP and use the highest CV to determine the number of samples to collect and analyze to finalize RCRA characterization for each waste stream. INEL-96/295 describes the applicability of the preliminary estimates to the waste stream to be sampled and the process for determining the estimates.

TWCP personnel randomly select and sample the statistically selected drums and analyze the samples a minimum of five containers must be sampled and analyzed in each waste stream. If samples for the preliminary mean and variance estimates were randomly collected from the same waste stream lot being examined and were collected and analyzed in the manner required for characterization samples, then these samples are counted toward meeting the required number. The number of drums sampled is dependent on defined levels of acceptable error for the hazardous versus nonhazardous determination, as described in the QAPP.

SPO personnel verify the samples collected within an waste stream were selected randomly. Upon completion of the required sampling, SPO personnel determine the final mean, variance, and CV for each contaminant. SPO personnel then compare the observed CV against the preliminary estimate of CV used to determine the number of samples to collect. If the observed CV is greater than the preliminary CV estimate, SPO personnel recompute the required number of samples using the observed CV. SPO personnel randomly select additional drums for sampling if the recomputed number of required samples is greater than 20% of the number of samples collected.

When sampling and analysis is completed for an waste stream, the SPM (or designee) calculates the UCL_{90} for the mean concentration of each contaminant. The SPO implementation plan includes a description of the calculations (including all equations) for mean, variance, CV, and UCL_{90} for the mean concentrations used for the RCRA characterization of waste streams. The SPO implementation plan also describes how data transformations (if necessary) and less-than-detectable analytical results are addressed.

EPA hazardous waste numbers are assigned according to the process described by the SPO FIP (INEEL 1999i).

5.4.2 Visual Examination of Retrievably Stored Homogeneous Solids, Soil/Gravel, and Debris Wastes

As a QC check on radiography, ANL-W personnel visually examine a statistically selected portion of the waste containers certified by the RWMC in one year to check the MPC and waste material parameter weights. SPO personnel determine, with acceptable confidence, the percentage of miscertified waste containers from the visual examination results on a twelve month period. Miscertified containers are those that radiography indicates meet the WIPP WAC radiography-determined and TRUPACT-II authorized methods for payload control requirements, but visual examination indicates do not meet these

requirements. Experience at the INEEL indicates that less than 2% of the RTR-certified waste containers were miscertified when compared with visual examination results.

The *Description of the SWEPP Certified Waste Sampling Program* (EDF-RWMC-363) (INEEL 1998a) describes how drums are selected for visual examination. Each year, the number of drums to be visually examined is determined based on the number of drums that RWMC is expected to certify and the previous years' miscertification rate.

At a minimum, the INEEL will visually examine enough drums to achieve the level of confidence required by the QAPP. The INEEL may also choose to use the replacement strategy described in the QAPP for drums undergoing homogeneous solids and soil/gravel coring operations. This strategy allows randomly selected drums for coring to replace drums randomly selected for visual examination from the same waste stream or waste stream lot. The implementation of this replacement strategy is described in INEEL 1998a.

5.5 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 5-1 lists the major technical implementing documents pertinent to this section of the QAPjP. FIPs identify additional facility procedures.

Table 5-1. Section 5 implementing procedures.

Document number	Title
INEL-95/029	*Matrix Parameter Category Groups (MPCGs)*
INEL-96/295	*Transuranic Waste Sampling Plan for the INEEL*
INEL-96/104	*Description of the SWEPP Certified Waste Sampling Program*

6. DRUM AND SAMPLE HANDLING AND CUSTODY REQUIREMENTS

TWCP personnel observe the sample handling and custody practices and requirements described in this QAPjP to ensure TWCP data meet accepted standards for legal admissibility and defensibility. Facility sample handling and custody procedures comply with the QAPP requirements and EPA guidelines as prescribed in the *National Enforcement Investigation Center Practices and Procedures* (EPA 1991). FIPs reference the applicable procedures that implement the requirements specified in this section. Procedures contain the forms (or describe the electronic equivalents) used to implement the requirements of this section.

6.1 Field Documentation

RWMC and ANL-W personnel record information pertinent to field sampling in reproducible, permanent ink or in INEEL-approved electronic format. The individual making a record entry dates and signs the record. At a minimum, sampling personnel record the following pertinent information:

- Sampling site
- Waste drum identification number
- Sample identification number (referenced to drum from which sample was taken)
- Sample matrix (e.g., headspace gas, sludge)
- Sampling equipment used
- Time and date of sample collection
- Quantity of sample collected
- Type and number of sample containers along with the equipment cleaning batch or manufacturer's lot number, if applicable
- Sample preservatives
- Analysis requested
- QC designation, if applicable
- COC record number
- Analytical laboratory performing the analysis
- Shipping information
- Sampler's name

- Procedure document number and revision
- Real-time instrument readings, if applicable [e.g., organic vapor analyzer (OVA) ppm indication]
- Comments pertinent to sampling activities

For headspace gas sampling using SUMMA® canisters, RWMC personnel record the following additional information:

- Ambient temperature and pressure measurements at the time of sample collection
- Canister pressures before and after sample collection

For sampling homogeneous solids and soil/gravel, ANL-W personnel record the following additional information:

- Coring tool identification
- Randomly selected coring location
- Depth of waste, depth of core, and core recovery
- Visual observations of waste and recovered core
- Randomly selected sample location
- Information on smaller waste container sampling, when applicable

6.2 Labeling

Facility personnel label drums and samples to meet the requirements specified in the QAPP. Facilities maintain procedures for drum and sample labeling and describe or cite those procedures in FIPs.

6.2.1 Drum Identification Numbers

All drums stored at the INEEL are presently identified with a unique identification number which is affixed to, inscribed on, or otherwise attached to the drum. This identification number is used on related COC documents. During waste characterization activities, some waste is transferred from the original drum to a new drum. RWMC personnel assign a unique identification number to the new drum.

RWMC Form-155
Page 1 of 1
Rev. 0, 03/15/99

TWCP GAS SAMPLE CANISTER TAG

Z Z	M M D D Y Y	E I A A X X X
Sampling Site	Date	Canister ID

Batch Number: _____ Drum Number: _____

Sampling Organization: _____ Sample Description: _____

Location	Canister Pressure C or M (1)	Ambient P and T (2)	Date MMDDYY	Time 24 Hour	Initials
Certifying Laboratory	C= M=	T= P=			
Field- Before Sample Collection	C= M=	T= P=			
Field- After Sample Collection	C= M=	T= P=			
Analytical Laboratory	C= M=	T= P=			

BLANK SAMPLE Y / N

ANALYSIS REQUESTED	VOCs	Hydrogen	Methane

Remarks:

_____

_____

Sampler Signature: _____

Notes: (1) C = Canister pressure gauge reading inches Hg (evacuated), or Psi (pressurized);
M = Manifold pressure gauge in mm Hg.
(2) P = pressure in mm Hg; T = Temperature in C.

Figure 6-1. Example of canister tag form.

6.2.2 Headspace Gas Sample Identification Number

Sampling personnel assign each SUMMA® canister sample a unique identification number at the time of sample collection. The 13-digit number is in the following format:

ID	MMDDYY	EI XXX
Idaho sampling site	Date	Canister identification

6.2.3 Headspace Gas Sample Canister Tag

The RWMC implementation plan and referenced procedures include instructions for completing the sample canister tags (or equivalent documentation). Figure 6-1 is an example of the sample canister tag currently used in the TWCP. Certifying laboratory personnel document the canister pressure (i.e., both the pressure from the manifold gauge and the canister gauge reading) for each field and field QC sample canister after cleaning. This information is recorded in permanent ink on a canister tag which is securely fastened to the canister prior to shipment to the field or is recorded on equivalent documentation traceable to the canister. During sampling and analysis, TWCP personnel record the following information in permanent ink on the canister tag (or equivalent documentation):

- Sample identification number
- Drum number
- Sampler's initials
- Ambient temperature (°C) and pressure (mm Hg)
- Sampling organization
- Sample description
- Comment section
- Requested analyses
- Date and time of sample collection
- Designation of whether the sample is a blank

NOTE: The individual responsible for each of the following entries records the date and time, and initials the corresponding documentation.

- RWMC personnel record the canister gauge reading in the field immediately before use.
- RWMC personnel record the sampling manifold pressure when a sampling manifold is used.

- ECL personnel record canister pressure and ambient pressure and temperature within 24 hours of VTSR. Canisters are thermally equilibrated to ambient temperature before these measurements are made.

After sample receipt, ECL personnel place the sample canister tag (or equivalent documentation) in their files.

6.2.4 Homogeneous Solids and Soil/Gravel Sample Identification Number

Sampling personnel assign a 13-digit sample identification number to each sample of solid process residues and soils collected. This sample numbering scheme complies with the QAPP; however, a thirteenth digit has been added for complete container identification; this digit (N) defines how many containers make up each subsample. The sample identification number is in the following format:

ID XXXXXX Y Z T A N

where:

ID	=	Idaho sampling site
XXXXXX	=	6-digit drum identification (bar code) number
Y	=	Yth core sample number
Z	=	zero, if between core sample compositing is not required
	or	
	=	number of the second core sample in the composite
T	=	Tth subsample in the core sample
	or	
	=	C, when single core sample composite is required
	or	
	=	B, when between core compositing is required
A	=	specified analysis:
		V, for VOC analysis
		M, for metal and SVOC analysis
N	=	Nth container in the subsample

Example:

Sample number, ID 023691 1 0 1 V 2 indicates

ID: Idaho sample

XXXXXX: from drum 023691

Y: the first core sample

Z: no between core composite

T: the first subsample, no composite

V: VOC analysis sample

2: second vial in the subsample

6.2.5 Homogeneous Solids and Soil/Gravel Sample Label

ANL-W personnel label each sample container prior to shipping the sample to the ACL. A copy of the labels (or description of the electronic system) used is included in the ANL-W FIP or referenced procedures. The completed label is affixed to each sample container with the following information recorded in permanent ink:

- Applicable waste container identification number
- Sample identification number
- Time and date of sample collection
- Type and number of sample containers
- Sample preservatives
- Analysis requested
- Sampler's initials
- Remarks

6.3 Chain of Custody

TWCP personnel track original waste drums and new waste drums (for repackaged waste) and initiate and maintain sample COC to meet the requirements specified in the QAPP. Facilities maintain tracking and COC procedures and describe or cite those procedures in FIPs. TWCP personnel initiate sample COC at the time of sample generation and maintain sample COC until analysis is complete, level 2 data validation is complete, and the sample is removed from the program. TWCP personnel initiate and maintain the following custody chains during the conduct of the TWCP:

- Homogeneous solid and soil/gravel samples from MPC S3000 and S4000 waste drums
- Headspace gas samples from MPC S3000, S4000, and S5000 waste drums

Documentation of tracking and custody chains creates a record that can be used to trace possession of the original waste drums, new waste drums, solid, soil, and gas samples. Documentation and tracking of drum and sample custody ensure the integrity of the waste or sample container and that characterization data are traceable to the waste drum.

A sample is considered under effective custody control if it is sealed (i.e., unopened) with the custody seal intact, and one or more of the following are true:

- It is in the possession of an authorized individual
- It is in the view of an authorized individual, after being in the possession of that individual

- It was in the possession of an authorized individual, and access to the sample(s) was controlled by locking or placement of signed custody seals that prevent undetected access

- It is in a designated secure area, such as a controlled access location with complete documentation of personnel access or a radiological containment area (hot cell or glove box)

Whenever a transfer of custody takes place, both parties sign and date a COC form (or electronic equivalent) and the relinquishing party retains a copy of the form. The receiving custodian inspects the custody form and all accompanying documentation (e.g., custody seals, sample tags, shipping forms) to ensure the information is complete and accurate and resolves any discrepancies or omissions with the organization responsible for collecting the sample. The receiving custodian also inspects all waste containers and samples for signs of damage or tampering. Any discrepancies in information, signs of damage, or tampering are documented on custody documentation (e.g., COC form, tags, labels) or on receiving checklists by the receiving custodian. Original COC forms are maintained in facility or SPO files. An NCR may be initiated if discrepancies cannot be resolved, omitted information is unrecoverable, or in cases of repeated documentation problems.

6.3.1 Drums

RWMC personnel track drums as described in the RWMC implementation plan and referenced procedures.

6.3.2 Gas Canister COC Form

For gas canister samples, RWMC personnel initiate sample COC, including field QC sample COC, immediately after sample collection into SUMMA® canisters. Sample COC is maintained until the required analyses are completed. SPO personnel release the canisters for cleaning after the analysis is completed. Sample COC is recorded on a sample COC form (example shown in Figure 6-2). The comment field is used to describe final sample container disposition.

6.3.3 Homogeneous Solids and Soil/Gravel COC Form

For homogeneous solids and soil/gravel, ANL-W personnel initiate sample COC, including field QC sample COC, immediately after sample collection. Sampling and laboratory personnel maintain sample COC until the required analyses are completed, level 2 data validation is complete and the samples are removed from the program. Sample COC is recorded on a solid/soil sample COC form (example shown in Figure 6-3). The comment field may be used to describe final sample container disposition.

6.4 Waste Drum and Sample Handling

Sampling, testing, and analytical facilities describe processes for waste drum and sample handling in FIPs and referenced procedures. These procedures include sample preservation, sample holding times, and waste drum and sample tracking.

INEL

TWCP GAS SAMPLE CHAIN OF CUSTODY FORM

1002

FORM L-0435.22
(08 96 - Rev. #00)

Sampling Location:			Bin No:		Field Batch No:				
Sampler(s)					Comments:				
Field Sample ID No.			Sampling Time		Sample Description			Remarks	
	Sampling Date (MMDDYY)	Canister ID							
Relinquished by: (signature)		Date/Time	Received by: (signature)	Location:	Relinquished by: (signature)	Date/Time	Received by: (signature)	Location:	
Relinquished by: (signature)		Date/Time	Received by: (signature)	Location:	Relinquished by: (signature)	Date/Time	Received by: (signature)	Location:	

Figure 6-2. Sample COC form.

Argonne National Laboratory
Waste Characterization Area
Solid/Soil Sample Chain of Custody

COC No.: COC Number

Sampling Location: ANL-W / HFEF / WCA
Waste Container No.: Drum Number
Date: _____

Sample Batch No.: Batch Number
Sampler: _____

Sample ID Number	Sample Date	Sample Time	Container Size	Sample Weight	VOC	NHVOC	Semi VOC	PCB	Metals	RAD	Comments

Relinquished By:	Date	Time
Received By:	Date	Time
Relinquished By:	Date	Time
Received By:	Date	Time

Relinquished By:	Date	Time
Received By:	Date	Time
Relinquished By:	Date	Time
Received By:	Date	Time

Discrepencies

TID# _____

Figure 6-3. Solid/soil sample COC form.

6.4.1 Waste Drum Sampling

Drums and their contents are allowed to equilibrate to the temperature of the sampling area for at least 72 hours prior to sampling. To ensure waste characterization data are collected that will represent conditions in the WIPP repository, drums are characterized at a temperature range of 18 to 29°C (65 to 84°F).

6.4.2 Gas Samples

RWMC personnel ensure gas samples collected in SUMMA® canisters are promptly transferred to the ECL or sampled and analyzed in integrated on-line sampling/analysis systems. Sampling personnel ensure sample holding times and storage conditions meet the requirements specified in Table 6-1. Sample handling adheres to an overall holding time of 34 days (4 days field holding time plus 2 days transfer shipping allowance plus 28 days laboratory holding time). Sampling and sample handling personnel maintain headspace samples between zero and 40°C (32 and 104°F).

RWMC personnel load gas samples in SUMMA® canisters into a shipping container and affix a signed and dated custody seal across the lid and body of the shipping container to provide visual evidence of tampering. A copy of the custody seal used is included in the RWMC implementation plan or procedures.

6.4.3 Homogeneous Solids and Soil/Gravel Samples

TWCP personnel ensure handling requirements for samples of homogeneous solids and soil/gravel meet the requirements for preservation and holding time specified in Table 6-2. ANL-W sampling procedures describe sample quantities and containers. ANL-W and transporting personnel ensure samples shipped to the ACL are maintained at a temperature of ≤ 4°C from the time of collection through sample transport to the ACL.

Before shipping samples to the ACL, ANL-W sampling personnel wrap sample jars in plastic (e.g., bubble wrap) to prevent breakage and place them in a cooler or other appropriate container for shipment. The ANL-W implementation plan and referenced procedures describe the process for sample shipment. ANL-W sampling personnel place the COC form in a waterproof plastic bag taped to the underside of the shipment container and affix a signed and dated custody seal or tamper-indicating device to the shipping containers. If a tamper-indicating device is used, the signature and date documenting the sample shipment container custody may be on documents traceable to the drum. The ANL-W implementation plan or referenced procedures provide example custody seals and accompanying documentation format. If more than one shipment container is being used, documentation is placed in the same container as the samples listed on that documentation.

The SPO implementation plan describes the INEEL solid/soils sample tracking system. The ANL-W implementation plan and referenced procedures include instructions to notify the SPO of sample shipments.

Table 6-1. Headspace gas holding conditions and times.

Parameter	Container	Minimum Drum Headspace Sample Volume[a]	Holding Temperature	Field Holding Time[b]	Shipping Allowance	Laboratory Holding Time[c]
H_2, CH_4	SUMMA® Canister	100 mL	0-40°C	4 days	2 days	28 days
VOCs	SUMMA® canister	250 mL	0-40°C	4 days	2 days	28 days

a. Alternatively, if available headspace is limited, a single 100 mL sample may be collected for determination of VOCs, H_2, and CH_4.

b. From time of headspace sample collection to shipment.

c. Programmatic-based maximum holding time. Holding time begins at validated time of sample receipt.

Table 6-2. Sample handling requirements for homogeneous solids and soil/gravel.

Parameter	Required Preservative	Maximum Holding Time[a]
VOCs	Cool to 4°C	14 days prep/40 days analyze[b]
SVOCs	Cool to 4°C	14 days prep/40 days analyze[b]
Metals	Cool to 4°C	180 days[c]

a. Holding time begins at sample collection (holding times are consistent with SW-846 requirements).

b. 40-day holding time allowable only for methanol extract—14-day holding time for nonextracted VOCs.

c. Holding time for mercury analysis is 28 days.

6.4.4 Laboratory Chain of Custody Program

ACL and ECL implementation plans describe each laboratory's documented sample custody program and reference procedures for sample receipt and log-in, sample storage and numbering, sample tracking in the laboratory, and storage of laboratory data. At a minimum, the sample custody programs include written procedures for the following:

- Chronological sample number sequencing
- Sample log-in (including determination of proper sample preservation)
- Identification of sample custodian
- Internal sample numbering and tracking systems
- Custody transfers within the laboratory
- Example custody forms with instructions for use
- Sample storage

- Sample disposal
- Analytical data maintenance and custody

6.5 Batch Numbering Convention

All TWCP facilities use a numbering convention to assign unique numbers to testing, sampling, analytical, and on-line batches. These numbers appear on the batch data reports forwarded to the SPO. TWCP personnel assign a batch number based on a format described in the FIP.

6.6 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 6-3 lists the major technical implementing procedures pertinent to this section of the QAPjP. FIPs identify additional facility procedures.

Table 6-3. Section 6 implementing procedures.

Document Number	Title
MCP-2525	*Drum Core Sample Plan*

7. HEADSPACE GAS SAMPLING

RWMC personnel collect headspace gas samples directly under the drum lid from the headspace of all TRU waste drums. To ensure representativeness, samples are collected after allowing each drum to equilibrate for a minimum time period (known as the drum age criterion) established in the QAPP. The drum age criterion is 225 days for Waste Types I and IV and 142 days for Waste Types II and III. RWMC personnel collect headspace gas samples using a sampling manifold as part of an on-line integrated sampling/analysis system or into SUMMA® canisters using the direct canister method. The RWMC implementation plan and referenced procedures detail headspace gas sampling activities. Table 7-1 identifies the type of headspace gas samples collected and corresponding analyses.

Table 7-1. Analyses required for each type of headspace gas sample collected.

Sample Type	VOCs[a]	H_2, CH_4
Drum lid	X	X
Field duplicate[b]	X	X
Field blank	X	—
Equipment blank[b,c]	X	—
Field reference standards[b]	X	X

a. See Table 12-1 for a list of VOCs.

b. For on-line integrated sampling/analysis systems, sampling and analytical QC samples are combined as on-line QC samples as described in Section 7.3.

c. For manifold only.

7.1 Quality Assurance Objectives

RWMC personnel collect field QC headspace gas samples for analysis at the frequency specified in Section 7.3 to demonstrate QAOs have been met. The QC headspace gas samples include equipment blanks (EBs), field reference standards (FRSs), field blanks (FBs), and field duplicates (FDs). For the on-line integrated sampling/analysis system, RWMC personnel combine the sampling and analytical QC samples as described in Section 7.3. Table 7-1 lists the required analytes for each type of headspace gas sample collected.

SPO personnel monitor field QC samples that are collected into SUMMA® canisters. RWMC personnel monitor on-line QC sample results and ensure corrective action is taken if acceptance criteria are not met. RWMC personnel prepare, submit, and resolve an NCR if final, reported QC sample results do not meet the acceptance criteria. The RWMC implementation plan and related procedures identify the methods used (summarized below) to demonstrate compliance with the QAOs. RWMC personnel:

- Collect FDs simultaneously into SUMMA® canisters or collect and analyze on-line duplicates sequentially for determination of VOCs, H_2, and CH_4 to assess the precision of the headspace gas sampling and analysis operation. RWMC personnel calculate the RPD for

on-line FDs, and the SPO calculates the RPD for canister FDs. Corrective action is initiated if the RPD exceeds 25.

- Collect an FRS into a SUMMA® canister or collect and analyze an on-line control sample using the on-line integrated equipment to assess the accuracy of the headspace gas sampling and analysis operation. RWMC personnel calculate the %R for FRS on-line samples, and the SPO calculates %R for canister FRSs. Corrective action is initiated if the %R of the FRS or on-line control sample is less than 70 or greater than 130.

- Conduct sufficient headspace gas sampling and on-line integrated sampling/analysis to ensure a minimum 90% completeness. The importance of any lost or contaminated headspace gas samples is evaluated by the SQAO and the RWMC FQAO and corrective action is initiated as appropriate. The RWMC implementation plan and related procedures describe how RWMC personnel document any nonroutine events or occurrences that may affect the quality of the headspace gas sample collected.

- Apply uniform procedures and equipment consistently, as specified in the Methods Manual to ensure headspace gas sampling operations are comparable to those performed at other sampling facilities.

- Follow specific headspace gas sampling steps to ensure samples are representative, including:

  - Sample canister cleaning and leak check
  - Sampling equipment cleaning or disposal after use
  - Sampling equipment leak check
  - Use of sample canisters with passivated internal surfaces
  - Use of low internal volume sampling equipment
  - Collection of small sample volume: low sample volume to available headspace volume ratio
  - Careful pressure regulation
  - Performance audits
  - Collection and analysis of QC samples

7.2 Methods Requirements

RWMC personnel obtain headspace gas samples at the SWEPP in a controlled radiological area using direct canister or manifold sampling methods. A sample of the headspace gas is obtained directly under the drum lid using the direct canister method, portable sample pump method, or manifold method (as part of an on-line integrated sampling/analysis system) as described in the RWMC implementation

plan and procedures that meet the requirements of Section 7.2 of the QAPP and appropriate Methods Manual procedures.

7.2.1 Direct Canister

In the direct canister method, SUMMA® canisters are used to collect headspace gas samples as described in the RWMC implementation plan and referenced procedures. The canisters and procedures meet the requirements specified in Procedure 110.2 in the Methods Manual. A portable sample pump may be used in the field to pressurize the canister to 15 to 30 psig as the sample is collected into the SUMMA® canister.

7.2.2 On-Line Integrated

The on-line integrated method described in the RWMC implementation plan is used to sample headspace gas for FTIRS analyses (VOCs and CH_4) and MS analysis (H_2). The on-line integrated method is called a residual gas analyzer (RGA), uses a manifold, and meets the requirements specified in Procedure 430.7 in the Methods Manual.

The headspace sampling system (HSS) for the drum vent facility consists of a sampling point, valve manifold, FTIRS instrument, RGA, tritium monitor, clean air machine, and vacuum pump. The sampling point is the punch that is located within the hot cell, called the silo. Drums to be punched and sampled are moved by conveyor through a rollup door to the punch position within the silo. The doors are closed and ventilation started to prevent contamination releases. At this time, the filter insertion machine (FIM) punches the drum. The punch then remains in the drum until a sample of the headspace gas can be obtained through the punch to the HSS system. To obtain this sample, a vacuum of 100 mTorrs is maintained in the HSS system. Once the sample is obtained, (HSS system reaches atmospheric pressure) valves are opened automatically to allow the various instruments to analyze the gas. When the gas is analyzed, the system is purged with clean air. The process is repeated for the next drum.

The HSS valve manifold, instruments, and associated equipment are located outside the silo area. Gas is transferred to these areas from the punch area using heat-traced 1/4-inch stainless steel tubing. Penetrations through the silo wall are sealed to prevent the spread of contamination to the equipment area.

7.2.3 Sampling Heads

The RWMC implementation plan describes and implements the sampling requirements specified in Procedures 110.3 and 110.4 in the Methods Manual. RWMC personnel collect a sample of the headspace gas from each drum. This ensures that a representative sample of headspace gas is collected.

7.3 Quality Control

RWMC personnel collect QC samples on a sampling batch or on-line batch basis. Sampling and on-line batches are defined in the Definitions section of this QAPjP. Table 7-2 summarizes QC sample collection requirements. Table 7-3 summarizes QC sample acceptance criteria. The RWMC implementation plan and referenced procedures implement these QC requirements.

For on-line integrated sampling/analysis systems, the on-line batch QC samples serve as combined sampling batch/analytical batch QC samples as follows:

- The on-line blank replaces the equipment blank and laboratory blank
- The on-line control sample replaces the field reference standard and laboratory control sample
- The on-line duplicate replaces the field duplicate and laboratory duplicate

RWMC FTIRS personnel also collect a comparison sample into a SUMMA® canister for analysis by gas chromatography/mass spectrometric detection (GC/MS) once per on-line batch. The acceptance criterion for this comparison sample is addressed in Section 12 of this QAPjP.

Table 7-2. Summary of headspace gas QC sample frequencies.

QC Samples	On-Line Integrated Manifold	Direct Canister and Portable Sample Pump
Field blanks[a]	1 per on-line batch[d]	1 per sampling batch[d]
Equipment blank or on-line blank[b]	1 per on-line batch[d]	once[e]
Field reference standard or on-line control sample[c]	1 per on-line batch[d]	once[e]
Field duplicate or on-line duplicate	1 per on-line batch[d]	1 per sampling batch[d]
FTIRS/SUMMA® comparison	1 per on-line batch[d,f]	Not applicable

a. Analysis of field blanks for VOCs (Table 12-1) only.

b. One equipment blank sample is collected, analyzed for VOCs (Table 12-1) and demonstrated clean prior to first use of the headspace gas sampling equipment with each of the sampling heads, then at the specified frequency for VOCs only thereafter. Daily, prior to work, the sampling manifold (if in use) is verified clean using an organic vapor analyzer (OVA).

c. One field reference standard is collected, analyzed, and demonstrated to meet acceptance criteria prior to first use and thereafter at the specified frequency.

d. See Definitions Section.

e. One equipment blank and field reference standard is collected after equipment purchase, cleaning, and assembly.

f. For comparison purposes, one sample per on-line batch is analyzed by GC/MS. This involves collecting the sample in a SUMMA® canister and comparing the FTIRS and GC/MS results.

Table 7-3. Summary of sampling quality control sample acceptance criteria.

QC Sample	Acceptance Criteria	Corrective Action[a]
Field blank	VOC amounts < 3 * MDLs in Table 12-1 for GC/MS and GC/FID; < PRQLs in Table 12-1 for FTIRS	Nonconformance if any VOC amount ≥ 3 * MDLs in Table 12-1 for GC/MS and GC/FID; ≥ PRQLs in Table 12-1 for FTIRS
Equipment blank or on-line blank	VOC amounts < 3 * MDLs in Table 12-1 for GC/MS and GC/FID; < PRQLs in Table 12-1 for FTIRS	Nonconformance if any VOC amount ≥ 3 * MDLs in Table 12-1 for GC/MS and GC/FID; ≥ PRQLs in Table 12-1 for FTIRS
Field reference standards or on-line control samples	70%R-130 %R	Nonconformance if %R < 70 or > 130
Field duplicates or on-line duplicates	RPD ≤ 25[b]	Nonconformance if RPD > 25
FTIRS/SUMMA® comparison	RPD ≤ 25[b]	Nonconformance if RPD > 25

FTIRS	=	Fourier Transform Infrared Spectroscopy
GC/FID	=	Gas Chromatography/Flame Ionization Detection
GC/MS	=	Gas Chromatography/Mass Spectrometry
MDL	=	Method detection limit
%R	=	Percent recovery
RPD	=	Relative percent difference

a. Corrective action only if the final, reported QC sample results do not meet the acceptance criteria; nonconformance procedures are outlined in Section 2.1.2.

b. Applies to concentrations greater than the PRQLs listed in Tables 11-1 and 12-1.

The SQAO and RWMC FQAO monitor and document QC sample results. The RWMC FQAO initiates an NCR if final, reported QC sample results do not meet the acceptance criteria. The SPM and RWMC manager ensure appropriate corrective action is taken if acceptance criteria are not met.

RWMC personnel collect FBs before sample collection at a frequency specified in Table 7-2. For the on-line integrated sampling/analysis system, FBs are collected through the entire manifold. If the results of the FB are within acceptance criteria, a separate EB or on-line blank may not be collected and analyzed. SPO personnel use the FB data to assess impacts of ambient contamination (if any) on the sample results.

After the initial cleanliness check, RWMC personnel collect on-line blanks through the manifold at a frequency of one per on-line batch for VOC analysis. For the direct canister method, RWMC personnel may use FBs instead of EBs. SPO personnel use the EB data to assess impacts of potentially contaminated sampling equipment on the sample results and ensure corrective action is taken when EB data indicate equipment contamination.

RWMC personnel use FRSs and on-line control samples to assess the accuracy with which the sampling equipment collects VOC, H_2, and CH_4 samples before first use. They ensure FRSs for direct sampling contain a minimum of six of the analytes listed in Table 12-1 at concentrations within a linear range of zero to 100 ppmv, and H_2 and CH_4 greater than or equal to the PRQLs listed in Table 11-1. They ensure on-line control samples contain at least 10 of the VOC analytes listed in Table 12-1 and methane (if analyzing methane by FTIRS) at concentrations within a linear range of zero to 100 ppmv for the VOCs and greater than or equal to the PRQL for methane (if applicable). They ensure these QC samples have a known valid relationship to a nationally recognized standard [e.g., National Institute of Standards and Technology (NIST)]. If commercial gases are used, they obtain a certification of analysis from the manufacturer, documenting traceability. Commercial stock gases are not used beyond their manufacturer-specified shelf-life. After the initial accuracy check, RWMC personnel collect FRSs and on-line control samples at the frequency specified in Table 7-2 and submit them blind to the laboratory. For the direct canister method, FRS collection may be discontinued if the FRS results meet acceptance criteria. FRS and on-line control sample %R are calculated using Equation (3-5) in Section 3.3. SPO personnel monitor FRS and on-line control sample results. SPO personnel ensure corrective action is taken when the QAOs for accuracy are not met.

RWMC personnel collect FD samples simultaneously into SUMMA® canisters and consecutively collect and analyze on-line duplicates for on-line integrated sampling/analysis systems at the frequency specified in Table 7-2 to assess the precision with which the sampling procedure can collect samples. SPO personnel monitor the FD and on-line duplicate results and calculate RPD using Equation (3-1) in Section 3.3. The SPM ensures corrective action measures are taken when acceptance criteria are not met.

For comparison purposes, RWMC personnel collect one sample per on-line batch for analysis by GC/MS. For the on-line integrated sampling/analysis system, this involves the collection of a sample in a SUMMA® canister. The results of this comparison are acceptable if the RPD between the FTIRS results and the GC/MS results is less than or equal to 25.

7.4 Equipment Testing, Inspection, and Maintenance Requirements

Sampling equipment components that come into contact with headspace gases are constructed of relatively inert materials, such as stainless-steel or Teflon®. To minimize the potential for cross-

contamination of samples, RWMC personnel clean and leak check the sampling equipment before headspace gas sampling. ECL personnel clean and leak check SUMMA® canisters after analyzing samples, prior to returning canisters to the RWMC. Procedures for cleaning and preparing the sampling equipment and sample canisters are described in the RWMC and ECL implementation plans and are based on Section 7.4 of the QAPP and the following Methods Manual procedures:

- Procedure 110.1 for cleaning and leak-checking the manifold
- Procedure 110.2 for cleaning and leak-checking the direct canister equipment
- Procedure 210.1 for SUMMA® canister cleaning and certification

7.5 Equipment Calibration and Frequency

For on-line integrated sampling and analysis, RWMC personnel certify the manifold pressure sensor before initial use, then annually using NIST or equivalent standards. If necessary, the pressure indicated by the pressure sensor(s) is temperature compensated. RWMC personnel certify the ambient air temperature sensor prior to initial use, then annually, to NIST traceable or equivalent temperature standards. The RWMC implementation plan or referenced procedures detail the specific requirements.

RWMC sampling personnel calibrate the OVA once per day, before first use, or as necessary according to manufacturer specifications using calibration gases certified to contain known analytes at known concentrations. They ensure the balance of the OVA calibration gas is consistent with the manifold purge gas when the OVA is used (i.e., zero air, nitrogen, or helium).

7.6 Data Management

For on-line integrated sampling/analysis systems, the RWMC combines the sampling batch and analytical batch data reports as on-line batch reports. These reports are described in Section 3.4.

RWMC sampling personnel document headspace gas sample collection for each waste drum. RWMC personnel review, validate, and verify sample collection records (as specified in Section 3.1) and control and report the data as described in the RWMC implementation plan. RWMC personnel submit a sampling batch data report (or electronic equivalent) to the SPO, consisting of the following:

- The sampling facility name, sampling batch number, sample numbers included in that sampling batch, and signature releases (Section 3.1.1)
- Data review checklist verifying data generation level review, validation, and verification have taken place (Section 3.1.1)
- Sampling information as specified in Section 6.1
- NCRs, if applicable

RWMC personnel maintain records in accordance with the RWMC implementation plan. They maintain the following records in their files, documented and retrievable by sampling batch number:

- Copies of the sampling batch reports submitted to the SPO
- Copies of the completed sample COC records
- Reference standard gas cylinder certification information
- Instrument calibration, maintenance, and repair records

The RWMC implementation plan or referenced procedures include an example form or describe the electronic system used to record field and field QC sample collection (either QC sample record form or COC form). Sampling personnel complete this form when samples are collected and submit the form to the SDCO. The SPO uses this information for canister tracking and analytical data validation.

7.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 7-4 lists the major technical implementing procedures pertinent to this section of the QAPjP. The RWMC implementation plan identifies additional facility procedures.

Table 7-4. Section 7 implementing procedures.

Document Number	Title
TPR-1728	*Drum Gas Sampling in WMF-610*
TPR-1584	*Headspace Sampling/RGA/FTIRS System operation*
MCP-1815	*On-Line HSS/RGA/FTIRS Level 1 Data Validation*

8. SAMPLING OF HOMOGENEOUS SOLIDS AND SOIL

The TWCP techniques for sampling homogeneous solids (MPC S3000) and soil/gravel (MPC S4000) are designed to obtain a representative sample to characterize a waste stream. These techniques ensure samples are randomly selected in both the horizontal and vertical planes of waste. Sampling procedures are identified in the ANL-W implementation plan and implement QAPP requirements. ANL-W personnel sample homogeneous solids and soil/gravel at the HFEF WCA from drums in MPC S3000 and MPC S4000 that are statistically selected by SPO personnel as specified in Section 5.

Drums may contain homogeneous solids or soil/gravel within smaller containers (e.g., 1-gal. polyethylene bottles). ANL-W personnel sample the waste in the drum or in one randomly selected smaller container within a drum. The samples are analyzed for VOCs, SVOCs, and metals as described in Sections 13, 14, and 15, respectively.

8.1 Quality Assurance Objectives

For 55-gal. drums containing homogeneous solids and soil/gravel, ANL-W personnel collect a core sample at a location randomly selected in the horizontal plane of the waste. They then collect representative subsamples from a location randomly selected along the core's length. For drums that contain smaller containers (e.g., 1-gal. polyethylene bottles), the smaller containers are grouped according to IDC and ANL-W personnel collect a representative sample from one randomly chosen container. The SPO provides sampling instructions to ANL-W. The ANL-W implementation plan and related procedures identify the methods used (summarized below) to demonstrate compliance with the QAOs. ANL-W personnel:

- Collect duplicates (e.g., colocated cores) once per sampling batch (see Definitions section) or once per week during sampling operations, whichever is more frequent. The SQAO determines precision by calculating and reporting the RPD between colocated samples.

- Comply with methods and requirements described in this section to minimize sample degradation and maximize sampling accuracy. Because drums containing homogeneous solids and soil/gravel with known quantities of analytes are not available, sampling accuracy cannot be determined.

- Measure completeness by calculating the number of valid samples collected as a percent of the total number of samples collected. ANL-W personnel achieve a minimum 90% completeness. The SQAO evaluates the importance of any lost or contaminated samples and determines whether corrective action is appropriate. The ANL-W implementation plan describes the process for documenting any nonroutine events or occurrences that may affect the quality of the samples collected.

- Apply uniform procedures, sampling equipment, and measurement units consistently to ensure sampling operations are comparable. They collect and evaluate colocated cores as described in Section 8.3.1. In addition, INEEL participates in the RCRA PDP program.

- Ensure they collect representative samples as follows:

- They ensure coring tools and sampling equipment are clean prior to sampling

- They core the entire depth of the waste (less a small safety factor) and ensure the core collected has a length greater than or equal to 50% of the depth of the waste. This is called the core recovery and is calculated as follows:

$$Core\ recovery = \frac{y}{x} * 100 \qquad (8\text{-}1)$$

where x is the depth of the waste in the container (as described in the Methods Manual) and y is the length of the core collected from the waste.

- They visually examine the core to verify minimal waste disturbance and describe the observation (e.g., undisturbed, cracked, pulverized) in field records. Coring operations and tool selection are designed to minimize alteration of the in-place waste characteristics.

If core recovery is less than 50% of the depth of the waste, a second coring location is randomly selected and the sample is collected from the core with the greatest recovery.

8.2 Methods Requirements

The ANL-W FIP and related procedures describe the sampling apparatus and process used to obtain samples from homogeneous solids and soil/gravel. The core sampling and subsampling processes and coring tools comply with the requirements specified in Sections 8.2.1 and 8.2.2 of the QAPP and Procedure 120.1 in the Methods Manual.

8.2.1 Core Sample Collection

ANL-W personnel use either a rotational or nonrotational coring tool to collect samples from drums of homogeneous solids and soil/gravel. They use these coring tools in such a way that minimizes disturbance to the core.

The ANL-W implementation plan and related procedures allow for the collection of multiple vertical cores from each drum. If the waste is packaged in smaller containers, procedures allow for the collection of a vertical core from one or more randomly selected containers. The number and location of cores to be collected from each drum are based on programmatic and regulatory needs and are influenced by such factors as waste content code and acceptable knowledge of the IDC. The SPO works closely with ANL-W personnel to determine individual drum core sampling requirements by completing a core sample plan for each drum.

8.2.2 Subsample Collection

The ANL-W implementation plan and related procedures allow for (a) extracting subsamples from anywhere along the length of a core, (b) preparing composite samples from the same core (except for those undergoing VOC analysis), and (c) preparing composite samples from different cores within the same drum (except for those undergoing VOC analysis). ANL-W personnel provide the minimum

amount of sample required by ACL analytical methods in order to minimize the quantity of investigation-derived waste. This will be documented in FIPs, or referenced procedures.

8.3 Quality Control

QC requirements for solidified waste sampling include the collection and analysis of collocated samples to determine precision and EBs to verify cleanliness of the coring tools and subsampling equipment; and analysis of reagent blanks to ensure reagents [e.g., deionized or high-pressure liquid chromatography (HPLC) water] are of sufficient quality. ANL-W sampling and subsampling of homogeneous solids and soil/gravel comply, at minimum, with QAPP QC requirements as described in the ANL-W implementation plan and related procedures.

8.3.1 Collocated Cores

ANL-W personnel collect collocated cores side by side and as close as feasible to one another at a frequency of one per week during sampling operations or one per sampling batch, whichever is more frequent. ANL-W personnel handle collocated samples in the same manner, visually inspect them through the transparent liner, and sample them in the same manner at the same randomly selected sample location. If the visual inspection reveals inconsistencies in the waste at the sample location, another sampling location is randomly selected, or two new collocated samples are obtained.

SPO personnel develop control charts for establishing acceptance criteria for collocated cores for each constituent for each waste matrix or waste type, as needed, as described in Section 8.3 of the QAPP. The SPO implementation plan identifies the procedure for developing, updating, and evaluating control charts.

8.3.2 Equipment Blanks

ANL-W personnel clean, identify, and seal in protective wrapping sampling equipment in compliance with the QAPP. ANL-W personnel collect EBs from fully assembled coring tools, liners (cleaned separately from the coring tools), and subsampling equipment prior to first use at a frequency of one per equipment cleaning batch (see Definitions section). (If ANL-W personnel discard liners and subsampling equipment after one use, they do not collect EBs from that equipment.) EBs are collected as described in the QAPP using clean water (e.g., deionized or HPLC water). ANL-W personnel collect the water in an "EPA certified clean" sample container and analyze the water for the analytes listed in Tables 13-1, 14-1, and 15-1. If analytes are detected at a concentration greater than or equal to three times the MDLs listed in Tables 13-1 and 14-1, or three times the program-required detection limits (PRDLs) listed in Table 15-1, ANL-W personnel clean the associated equipment cleaning batch of coring tools, liners, or subsampling equipment again and collect and analyze another EB before use. EB results are traceable to the items in the equipment cleaning batch and are reviewed before using coring tools, liners, and subsampling equipment, and a sufficient quantity is maintained clean in storage to prevent disruption of sampling operations.

Each coring tool has a unique identification number. ANL-W personnel reference the coring tool identification number in field records and relate this number to the drum number on which it is used. They test one coring tool for cleanliness from each equipment cleaning batch before use and record the identification number of this coring tool in the field records.

8.4 Equipment Testing, Inspection, and Maintenance Requirements

ANL-W personnel test, inspect, and maintain sampling areas and all sampling tools, equipment, and protective wrappings in accordance with the requirements in Section 8.4 of the QAPP. They test and maintain all sampling tools and equipment in accordance with applicable manufacturers' specifications. The ANL-W implementation plan describes specific testing, inspecting, and maintenance procedures. The ANL-W project records coordinator (PRC) maintains testing and maintenance records as described in the ANL-W implementation plan.

8.5 Equipment Calibration and Frequency

ANL-W personnel calibrate the balance used for weighing subsamples to maintain its operation within the manufacturer's specifications, and after repair or routine maintenance as described in the ANL-W implementation plan and referenced procedures. Weights used for calibration are traceable to a nationally recognized standard. The PRC maintains calibration records as described in the ANL-W implementation plan.

8.6 Data Management

ANL-W personnel record sample collection information for each waste drum and ensure its availability to data users. The ANL-W implementation plan describes procedures for recording, reviewing, and reporting sampling information. The ANL-W FIP, or referenced procedures, includes an example of the form used to record field and field QC sample collection information. The ANL-W PM submits a sampling batch data report for each sampling batch to the SPO on approved standard forms (or electronic equivalent). Sampling batch data reports consist, at a minimum, of the following:

- The ANL-W facility identifier, sampling batch number, sample numbers included in that sampling batch, and the signature releases of the sampling personnel as specified in Section 3.1
- Data review checklists for each sampling batch verifying that the data generation level review, validation, and verification (as described in Section 3.1) have taken place
- Information specified in Section 6.1
- NCRs, if applicable

In addition, the PRC maintains the following items in the files, documented and retrievable by sampling batch number:

- Copies of the sampling reports submitted to the SPO, filed in accordance with the sampling batch number
- Copies of the completed COC form(s) used to transfer the samples in that sampling batch to the ACL

- Coring information (e.g., coring duration, downward pressure applied, rotational speed, and torque applied, if applicable)
- Heat and dust generation observations
- Coring tool, liner, sampling equipment, and reagent certification information
- Instrument (e.g., scale) calibration, maintenance, and repair records

8.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 8-1 lists the major technical implementing procedures pertinent to this section of the QAPjP. The ANL-W and SPO implementation plans identify additional procedures.

Table 8-1. Section 8 implementing procedures.

Document Number	Title
HFEF OI 6910	*Core Drilling Operation*
HFEF OI 6921	*Sludge Sample Preparation*
MCP-2525	*Drum Core Sample Plan*

9. NONDESTRUCTIVE ASSAY

RWMC personnel perform RA measurements of waste drums at the SWEPP using a passive/active neutron (PAN) system and a high-resolution passive gamma ray spectroscopy (SGRS) system. With these two systems, they determine the radioactive material composition, quantify the radionuclide masses, and compute the associated derived quantities, such as total and TRU alpha activity. The RWMC implementation plan describes the RA processes and implementation of the requirements specified in Section 9.0 of the QAPP.

9.1 Quality Assurance Objectives

The QAOs for RA are precision, accuracy, sensitivity limits, minimum detectable concentration (MDC), total uncertainty, total bias, completeness, and comparability. The sensitivity limits criteria are specified by using an MDC requirement. The QAOs for precision, accuracy, MDC, completeness, and total bias are summarized in Table 9-1. The methods used for demonstrating compliance with the QAOs are described in the RWMC implementation plan and comply with Section 9.6 of the QAPP.

The RWMC implementation plan and referenced procedures describe processes for meeting QAOs. To demonstrate compliance with the QAOs, RWMC personnel:

- Determine precision through replicate processing of a drum containing a noninterfering matrix and known quantity(ies) of plutonium (Pu) source(s) in accordance with the nominal activity compliance points specified in Table 9-1. RWMC personnel determine precision for the range in which the assay system is operated.

- Determine accuracy through replicate processing of a drum containing a noninterfering matrix and known quantities of Pu sources in accordance with the nominal activity compliance points specified in Table 9-1. RWMC personnel determine accuracy for each range where the system is operated.

- Determine MDC from replicate processing of calibration drums with no radioactive sources added. The MDC is determined using calculations that set the risk for concluding falsely that activity is present above the critical level (α) and the predetermined degree of confidence for correctly detecting its presence above the critical level ($1-\beta$) at 5% and 95%, respectively.

- Determine total uncertainty for the SWEPP assay measurement process on a waste form basis and document the evaluation for review by an expert review team.

- Determine total bias as part of the determination of total uncertainty and document the evaluation for review by an expert review team.

- Demonstrate completeness by obtaining acceptable RA data for 100% of the waste drums characterized for disposal.

- Participate in the NDA PDP to demonstrate comparability between INEEL and other DOE sites. Results of the NDA PDP are reported to CAO for evaluation.

9.2 Methods Requirements

The SWEPP assay process uses the PAN system and the SGRS system. The SGRS is used to establish radionuclide ratios relative to a radionuclide whose mass is directly determined. The SWEPP assay process utilizes the SGRS data in conjunction with the PAN data to determine the radioactive material composition, quantify radionuclide masses, and compute the associated derived quantities.

Table 9-1. Quality assurance objectives for nondestructive assay.

Range of Waste Activity in ^{a}-Curies[a]	Nominal Compliance Point ^{a}-Curies[a] (g WG Pu)[b]	Precision[c] (%RSD)	Accuracy[d] (%R)	Parameter Total bias[e] (%)	Completeness[f] (%)	MDC (nCi/g)[g]
0	0					60
>0.002 to 0.02	0.008 (0.1)	≤ 20	75-125	Low 25 High 400	100	
>0.02 to 0.2	0.08 (1.0)	≤ 15	50-150	Low 35 High 300	100	
>0.2 to 2.0	0.8 (10)	≤ 10	75-125	Low 67 High 150	100	
> 2.0	12.5 (160)	≤ 5	75-125	Low 67 High 150	100	

a. Applicable range of TRU activity in a 208-liter (55-gal.) drum to which the QAOs apply, units are Curies of alpha-emitting TRU isotopes with half-lives greater than 20 years.

b. The nominal activity (or weight of Pu) in the 208-liter (55-gal.) drum used to demonstrate that QAOs can be achieved for the corresponding range in column 1, values in parentheses are the approximate equivalent weights of WG Pu, 15 years after purification; for purposes of demonstrating QAOs, "nominal" means within □10%.

c. □ One standard deviation based on 15 replicate measurements of a noninterfering matrix.

d. Ratio of measured to known values based on the average of 15 replicate measurements of a noninterfering matrix.

e. 95% confidence bounds for system bias established by studies to determine contributions to total uncertainty from all significant sources. Units are confidence bound divided by true value, expressed as a percent. Requirement for the QAO for total uncertainty is to determine and document.

f. Valid radioassay data are required for all waste containers.

g. As defined in Sections 9.1 and 9.6 of the QAPP and the RWMC implementation plan.

RWMC personnel demonstrate and document the performance of software associated with RA in accordance with ASME NQA-1, Element 11, Supplement 11S-2 (ASME 1989) and NQA-2, Part 2.7 (ASME 1990). They ensure software testing covers the full range of expected system application.

RWMC personnel ensure the methods and systems used for RA meet the QAOs listed in Table 9-1 for the applicable ranges in which they operate. They have developed procedures that detail all aspects of RA operation. RWMC RA procedures instruct operators to perform all necessary background and performance checks prior to performing any drum assays. RWMC personnel check performance check

data against predetermined acceptance criteria as documented in the procedures. They initiate corrective action if any acceptance criterion is not met. They document and justify the disposition and use of any assays performed during a period ending with a suspect performance check.

RWMC RA procedures require the use of proper calibration standards, proper equipment and equipment setup, avoidance of practices known to result in inaccurate assays, attention to proper record-keeping and equipment maintenance, and safe operation of equipment. These procedures contain all necessary instructions for the operation of computerized data acquisition systems. Instructions include explanations of required input, options, and prohibitions for operators when exercising any interactive portions of the software. These procedures are written, approved, and controlled under the provisions of this QAPjP. The procedures have been internally demonstrated in the RWMC and include documented performance characteristics which meet the QAOs listed in Table 9-1 for the waste activity ranges in which the systems are operated.

9.3 Quality Control

RWMC personnel implement a documented QA program as described in the RWMC implementation plan. This QA program specifies qualitative and quantitative acceptance criteria for QC checks and the corrective action necessary when acceptance criteria are not met. The RWMC FQAO is responsible for monitoring and documenting procedure performance, including the analysis of QC samples. RWMC personnel initiate and resolve an NCR if the final reported QC measurements do not meet the acceptance criteria. The FQAO and the RWMC manager are responsible for implementing corrective action when acceptable procedure performance is not met.

RWMC personnel perform routine performance checks on the PAN and SGRS systems, as described in the RWMC implementation plan and referenced procedures. They operate the RA systems in statistical control as determined by the control limits established in RA procedures. The FQAO reports the results of these performance checks to the SQAO, who forwards these results to CAO on a semiannual basis.

RWMC personnel perform routine performance checks of efficiency, background, and energy resolution as described below. They use control charts to track trends in the parameters measured in the performance checks. They log data, plot the data on control charts, and compare this data to preset control limits. They report these data with RA results to the SPO. Performance checks include efficiency checks, background checks, energy calibration checks, and energy resolution checks, as appropriate for the RA system. System performance check implementation and documentation are described in the RWMC implementation plan and referenced RA procedures.

For the PAN system, RWMC personnel perform and document an efficiency check at least twice per work shift, prior to making measurements on any waste drums, and after completing all waste drum measurements for that shift. They perform and document a background check at the beginning of each shift.

For the SGRS system, RWMC personnel perform and document efficiency checks, energy calibration checks, and energy resolution checks continuously via a pulsar input system. They perform and document additional efficiency checks, energy calibration and energy resolution checks on a weekly basis. They perform and document a background check on a monthly basis.

RWMC personnel perform and document a duplicate measurement (i.e., replicate count) on one of every 20 drums, or once per operating day (24 hours), whichever is more frequent. RA procedures detail duplicate measurement and documentation processes.

The RWMC participates in the NDA PDP to provide measurements for comparing performance with that of other organizations performing measurements for the same analytes under comparable conditions. Data resulting from the PDP is reported to CAO.

RWMC personnel who operate the assay equipment are trained in accordance with ASME NQA-1, Element 2, with the exception of Supplement 2S-2 (ASME 1989) and are requalified every two years. The RWMC training coordinator ensures training records are current and that unsatisfactory performance results in disqualification, retraining, and demonstration of satisfactory performance prior to allowing an operator to again operate RA systems.

9.4 Equipment Testing, Inspection, and Maintenance Requirements

RWMC personnel calibrate RA systems in accordance with the controls established in Section 9.5 of this QAPjP as described in the RWMC implementation plan and referenced procedures. RA procedures detail system maintenance, routine system calibration, performance checks, and operation. The procedures are consistent with applicable sections of the American National Standards Institute (ANSI) and American Society for Testing and Materials (ASTM) standards listed below:

ANSI N15.36-1994, Nondestructive Assay Measurement Control and Assurance

ANSI N15.20-1975, American National Standard Guide to Calibrating Nondestructive Assay Systems

ANSI N42.14-1991, Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides

ASTM C 1207-91 Standard Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting

9.5 Calibration Procedures and Frequencies

RWMC personnel perform calibrations on the RA system consistent with consensus standards (e.g., ASTM, ANSI). The RWMC implementation plan describes the calibration processes and the associated measurement parameters. RA procedures detail all aspects of system calibration and documentation. RWMC personnel calibrate RA systems at least annually and support these calibrations with QA records which can be tracked to standards obtained from NIST or from suppliers maintaining measurement systems traceable to NIST.

RWMC personnel prepare calibration standards from primary standards obtained from suppliers maintaining measurement systems traceable to NIST, whenever these standards are available. When these standards are not available, they calibrate standards against primary standards obtained from suppliers maintaining measurement systems traceable to NIST. They document the cross-calibration and maintain this documentation as a QA record. Traceable standards using material of nominal weapons-grade plutonium composition are used to calibrate the PAN system.

Traceable standard Eu-152 is used to calibrate the SGRS detectors consistent with ANSI N42.14-1991. RA procedures specify the range of applicability of system calibrations. If a particular assay falls outside this range, RWMC personnel initiate and document corrective action. They derive matrix correction factors from correlation algorithms embedded in the PAN system software. They determine and document the range of waste types to which any given calibration and set of correction factors apply.

RWMC personnel ensure that all computer programs (and any revisions) are documented, verified, and validated in accordance with the QAPD prior to use for data generation. They ensure verification includes both verification of the algorithm and test runs comparing program output to true values. They ensure test runs exercise all default and boundary values of parameters. They ensure programs are documented in accordance in with *Standard for Software User Documentation* (ANSI 1987) and include the following minimum information:

- Program name
- Revision number
- Revision date
- Author(s)
- Program application
- Programming language (including version numbers of all compilers, linkers, etc.)
- Operating system
- Required hardware
- Descriptions of algorithms used
- User's manual
- Listing of code
- Examples of input and output forms
- Results of test cases
- Copies of external data files
- Lists of default parameters
- Records of review and approval

RWMC procedures identify the individuals responsible for the following functions:

- System operation and maintenance, including documentation and training

- Database integrity, including data entry, data updating and QC
- Data and system security, backup, and archiving

9.6 Data Management

RWMC personnel collect and reduce RA data using computer software designed for the PAN and SGRS. The computer software is controlled to comply with the QAPD and ANSI (1987) as described in the RWMC implementation plan. Data reduction is detailed in RA documents, which include the algorithms used.

RWMC personnel review, verify, and validate each testing batch data report as described in Section 3.1.1 of this QAPjP. RWMC methods for validation, including verification that the QAOs have been met, are described in the RWMC implementation plan and related procedures and comply with Section 9.6 of the QAPP.

RWMC personnel compile data reports on a testing batch basis (see Definitions section). RWMC personnel assign each testing batch data report a unique report number (this number may be the testing batch number—see Section 6.5) and number each page of the data report at the bottom. RWMC personnel transmit the final RA testing batch data report on approved standard forms (or electronic equivalent). The RWMC implementation plan or referenced procedures provide example reporting forms. The report is transmitted to the SPO and contains the following information:

- RWMC identifier, testing batch number, drum numbers included in that testing batch, and signature releases of RA testing personnel as described in Section 3.1
- Table of Contents
- RWMC level 1 data review checklists for each testing batch verifying the data generation level review (as described in Section 3.1) has taken place.
- Separate testing report sheet(s) for each sample in the testing batch, including:
  - Title "Radioassay Data Sheet"
  - Method used for RA (i.e., procedure identification)
  - TRUCON code, Item Description Code, and matrix parameter category, as applicable
  - Date of RA examination
  - Total Pu-239 fissile gram equivalents (g) and associated uncertainty
  - Total alpha activity and associated uncertainty (Ci)
  - TRU activity and associated uncertainty (nCi/g)
  - Listing of individual radioisotopes present and associated uncertainty (Ci)

- Thermal power and associated uncertainty (W)
- QC replicate (yes or no); if yes, brief description of comparison results
- Operator signature and date
- Reviewer signature and date
- – Pu-239 equivalent activity

NOTE 1: *The RTR data (Section 10) and RA data may be combined into a single RWMC data report.*

NOTE 2: *TRIPS electronic reporting includes all of the above information. The data is validated using electronic checklists. TRIPS utilitizes a password protected electronic signature process.*

The RWMC retains testing batch records as defined in Section 9.6 of the QAPP.

9.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 9-2 lists the major technical implementing procedures pertinent to this section of the QAPjP. The RWMC implementation plan identifies additional procedures.

Table 9-2. Section 9 implementing procedures.

Document Number	Title
TPR-1573	*Passive/Active Neutron Drum Assay System*
TPR-1588	*Gamma Ray Spectroscopy System*
TPR-1726	*TRU Waste Characterization Examination*
INEL-96/0008	*SWEPP Non-Destructive Assay Software Verification and Validation Plan*

10. RADIOGRAPHY

RWMC personnel perform RTR of waste drums at the SWEPP. Through RTR, RWMC personnel determine the MPC and estimate weights of the waste material parameters listed in Table 10-1. To verify RTR results, ANL-W personnel perform visual examination of a portion of the waste drums at the HFEF. This section describes the RTR and visual examination processes.

10.1 Quality Assurance Objectives

The RWMC and SPO implementation plans and referenced procedures identify the methods used to meet the QAOs. To demonstrate compliance with the QAOs, RWMC and SPO personnel:

- Calculate the RPD (performed by the SQAO) between the estimated waste material parameter weights as determined by RTR, and these same parameters as determined by visual examination.

- Determine accuracy with which the MPC is assigned by submitting a randomly selected statistical portion of drums to ANL-W for visual examination (Section 5). The SQAO calculates the percentage of waste drums that require a new MPC after visual examination and reports the result as a measure of RTR accuracy.

- Meet the completeness QAO by documenting the RTR examination on an audio/videotape and on a RTR data form for 100% of the waste drums in the TWCP.

- Use standardized RTR procedures and qualify operators in accordance with the requirements of the QAPP to enhance the comparability of radiography data from different sites.

10.2 Methods Requirements

RWMC personnel use RTR developed by the DOE to aid in the examination and identification of containerized waste. The procedures used to achieve the RTR objectives are described in the RWMC implementation plan. Trained RTR operators record data on a RTR data form (or electronic equivalent) and an audio/videotape. The RWMC implementation plan or referenced procedures include example RTR data reporting forms or describe the electronic system on which RTR results are recorded. RWMC personnel use procedures that meet all QAPP requirements and are based on Procedure 310.1 in the Methods Manual. ANL-W personnel use procedures that meet all QAPP requirements and are based on Procedure 310.2 in the Methods Manual.

10.3 Quality Control

RWMC personnel ensure QC of RTR through operator training and experience and qualitative and semi-quantitative evaluations of visual displays. Additionally, SPO personnel verify the RTR results through visual examination data provided by ANL-W for a statistically determined portion of waste containers.

Table 10-1. Waste material parameters and descriptions.

Waste Material Parameter	Description
Iron-based metals/alloys	Iron and steel alloys in the waste; does not include the waste drum materials
Aluminum-based metals/alloys	Aluminum or aluminum-based alloys in the waste materials
Other metals	All other metals found in the waste materials
Other inorganic materials	Nonmetallic inorganic waste, including concrete, glass, firebrick, ceramics, sand, and inorganic sorbents
Cellulosics	Materials generally derived from high polymer plant carbohydrates Examples are paper, cardboard, wood, cloth, etc.
Rubber	Natural or man-made elastic latex materials. Examples are surgeons' gloves, leaded rubber gloves, etc.
Plastics (waste materials)	Generally man-made materials, often derived from petroleum feedstock. Examples are polyethylene, polyvinyl chloride, etc.
Organic matrix	Cemented organic resins, solidified organic liquids and sludges
Inorganic matrix	Any homogeneous materials consisting of sludge or aqueous-based liquids that are solidified with cement, calcium silicate, or other solidification agents. Examples are wastewater treatment sludge, cemented aqueous liquids, and inorganic particulates, etc.
Soils	Generally consists of naturally-occurring soils contaminated with inorganic waste materials
Steel (packaging materials)	208-L (55-gal.) drums
Plastics (packaging materials)	90-mil polyethylene drum liner and plastic bags

Source: Waste Isolation Pilot Plant Transuranic Waste Baseline Inventory Report (DOE 1995e) Revision

10.3.1 Radiography

Only trained personnel are allowed to operate RTR equipment to ensure QC in regard to RTR system operation and for interpretation and disposition of RTR results. RTR operators are trained in accordance with the RWMC implementation plan or referenced procedures to meet the standardized training requirements for RTR operators. These documents are based upon existing industry standard training requirements and comply with the training and qualification requirements of ASME NQA-1, Element 2 (except for Supplement 2S-2.

The RWMC training program provides RTR operators with both formal and on-the-job training (OJT). RTR operators are instructed in the specific waste generating practices associated with the waste and typical packaging configurations expected to be found in each MPC at the INEEL. An experienced, qualified RTR operator conducts the OJT and apprenticeship prior to qualification of the training candidate. The RWMC training program contains the following required elements based on ASME NQA-1 requirements:

Formal Training

- Project Requirements
- State and Federal Regulations
- Basic Principles of Radiography
- Radiographic Image Quality
- Radiographic Scanning Techniques
- Application Techniques
- Radiography of Waste Forms
- Standards, Codes, and Procedures for Radiography
- Site-Specific Instruction

On-the-Job Training

- System Operation
- Identification of Packaging Configurations
- Identification of Waste Material Parameters
- Weight and Volume Estimation
- Identification of Prohibited Items

RWMC personnel have assembled radiography test drums that include items common to the IDCs stored at the INEEL. The test drums may be divided into layers with varying packing densities or different drums may be used to represent different situations that may occur during RTR examination at the site. The RWMC implementation plan provides further details. One test drum contains the following required elements:

- Aerosol can with puncture
- Horsetail bag
- Pair of coveralls
- Empty bottle
- Irregular shaped pieces of wood

- Empty one gallon paint can
- Full container
- Aerosol can with fluid
- One gallon bottle with three tablespoons of fluid
- One gallon bottle with one cup of fluid (upside down)
- Leaded glove or leaded apron
- Wrench

These items must be successfully identified by the operator as part of the qualification process. To be qualified, RTR operators:

- Successfully pass a comprehensive exam based upon training enabling objectives
- Perform practical capability demonstration in the presence of an appointed site RTR subject matter expert (the RTR subject matter expert is an experienced RTR operator who is qualified as an OJT trainer)

Requalification of operators is based upon evidence of continued satisfactory performance (primarily audio/videotape reviews) and is performed at least every two years. Unsatisfactory performance results in disqualification. Unsatisfactory performance is defined as the misidentification of a prohibited item in a training drum or a score of less than 80% on the comprehensive exam. Retraining and demonstration of satisfactory performance are required before an operator is again allowed to operate the RTR system.

Each operator periodically scans a training drum with various container sizes. A supervisor reviews the videotape to ensure that operators' interpretations remain consistent and accurate. Imaging system characteristics of the monitoring system are verified on a routine basis.

RTR operators perform independent replicate scans and replicate observations of the video output of the RTR process under uniform conditions and procedures. RTR operators perform independent replicate scans on one waste container per day or one per testing batch (see Definitions section), whichever is less frequent. An independent observation of one scan (not the replicate scan) is also performed once per day or once per testing batch, whichever is less frequent.

RTR-qualified personnel other than the operator who dispositioned the waste container perform the oversight functions identified above. The results of this verification are available to the RTR operator. RWMC personnel are responsible for monitoring the quality of the RTR data and calling for corrective action, when necessary.

10.3.2 Visual Examination

The visual examination process at the INEEL consists of a combination of activities performed by three key job functions: the SPO Visual Examination Expert (VEE), the ANL-W VEE and ANL-W Visual Examination Technicians (VETs).

- The SPO visual examination expert is responsible for the overall management and implementation of visual examination program at the INEEL.

- The ANL-W VEE is responsible for the overall management and implementation of the waste visual examination process at ANL-W.

- ANL-W visual examination technicians perform the actual waste examination; weighing the waste and reporting the data, including waste material parameter weights and a brief description of the drum contents.

The SPO VEE is familiar with the waste generating processes, selects drums to undergo visual examination, provides visual exam guidance to the ANL-W VEE on a drum by drum basis per the SPO FIP and evaluates the visual examination data. The visual examination data evaluation includes verification of the MPC and comparison of the RTR and visual examination results.

The ANL-W VEE reviews and approves the visual exam guidance (see SPO FIP) and ANL-W VETs visually examine a statistically determined portion of waste containers and supply data to the SPO. SPO personnel verify the RTR results. The ANL-W implementation plan and referenced procedures describe visual examination activities; this examination includes the waste material parameter weights. SPO personnel compare RTR and visual examination results and verify MPCs as described in the SPO implementation plan. SPO personnel transmit visual examination results to the RWMC.

The SPO VEE randomly selects drums for visual examination based on the miscertification rate observed in waste containers visually examined over a minimum 12-month period (Section 5). The SPO VEE determines a new miscertification rate each year and this rate is used to calculate the number of waste containers randomly selected for visual examination in the coming 12-month period. The SQAO performs the RTR/visual examination data comparison.

For visual examination, ANL-W personnel perform a semi-quantitative and/or qualitative evaluation of the waste container contents, and record the examination on audio/videotape. The ANL-W implementation plan describes ANL-W standardized training for visual including both formal classroom and OJT. The SPO and ANL-W visual examination experts are instructed in the specific waste generating processes associated with the waste, typical packaging configurations, and waste material parameters expected to be found in each matrix parameter category at the INEEL. An ANL-W visual exam technician experienced and qualified in visual examination conducts the OJT and apprenticeship prior to qualification of the candidate. Visual examination personnel are requalified once every two years. The ANL-W HFEF and SPO training programs contain the applicable required elements based on ASME NQA-1 requirements as shown in Table 10-2.

Table 10-2. Visual examination training elements.

Training	Visual Exam Technician	Visual Exam Expert	ANL-W VEE
Formal			
• Project requirements	N/A	✔	✔
• State and federal regulations	N/A	✔	
• Application techniques	✔	✔	✔
• Site-specific instruction	✔		
On-the-job			
• Identification of packaging configurations	✔	✔	✔
• Identification of waste material parameters	✔	✔	✔
• Weight and volume estimation	✔	✔	✔
• Identification of prohibited items	✔	✔	✔

The SPO visual examination expert selection, qualification, and training requirements are identified in the SPO implementation plan and include education, experience and, familiarity with INEEL TRU waste processes.

The SPO visual examination expert decides the extent of waste segregation that is required to achieve TWCP objectives and provides written visual exam guidance to the ANL-W VEE. The visual examination expert's decision-making criteria are described in the SPO implementation plan and include acceptable knowledge documentation, IDCs, and other pertinent waste description information.

ANL-W VETs record a description of the waste container contents on a data form (or electronic equivalent) and summarize and report waste material parameter weights. In cases where bags are not opened, ANL-W VETs provide a brief written description of the contents of the bags and an estimate of the amount of each waste type in the bags. They supplement the written records of visual examination with the audio/video recording.

10.4 Instrument Testing, Inspection, and Maintenance Requirements

RWMC personnel procure all RTR equipment in accordance with the LMITCO Quality Assurance Program Procurement Requirements, and test and maintain it in accordance with manufacturer instructions. The RWMC implementation plan identifies the testing and inspection procedures. RWMC site document control personnel maintain records of testing and maintenance as described in the RWMC implementation plan.

10.5 Instrument Calibration and Frequency

RTR operators ensure equipment is calibrated and maintained in accordance with controls and performance criteria established and described in the RWMC implementation plan and referenced procedures that address performance criteria in the QAPP. When RTR equipment is in use, RTR operators conduct operational checks that include observation of a test pattern to verify video quality at the beginning of each work shift, as described in the RWMC implementation plan.

10.6 Data Management

RWMC personnel submit RTR batch data reports for each testing batch to the SPO on approved standard forms. The RWMC implementation plan or referenced procedures include example forms that will be used for data reporting. RTR batch data reports consist of the following:

- RWMC identifier, testing batch number, waste drum numbers included in the testing batch, and signature releases of facility personnel as described in Section 3.1.1
- Table of Contents
- Data review checklists for each testing batch verifying the data generation level review, validation, and verification (Section 3.1.1) has taken place; checklists may contain tables showing the results of testing batch-related QC samples (i.e., replicate scans, independent observations)
- Separate testing report sheet(s) for each waste drum in the testing batch that includes
  - Title "Radiography Data Sheet"
  - Date of radiography examination
  - Waste drum number
  - TRUCON Code, Item Description Code, and MPC, as applicable
  - Any changes made to MPC
  - Estimate of each applicable waste material parameter weight
  - Presence/absence of waste drum liner (yes/no)
  - Description of contents packaging materials, including the number of layers of packaging
  - QC replicate scan (yes/no); if yes, brief description of comparison results
  - Audio/videotape or CD Rom identification number
  - Operator signature/date
  - Reviewer signature/date
- NCRs, if applicable

- For waste drums undergoing visual examination, ANL-W personnel report the following additional data:
  - MPC determined by visual examination
  - Waste material parameter weights
  - Audio/video tapes

NOTE 1: *The RTR and RA data (see Section 9) may be combined into a single RWMC data report.*

NOTE 2: *TRIPS electronic reporting includes all of the above information. The data is validated using electronic checklists. TRIPS utilizes a password protected electronic signature process.*

RWMC and ANL-W maintains the following appropriate items in Facility files, documented and retrievable by testing batch number:

- Audio/videotapes or CD Rom
- All raw data, including instrument readouts, calculation records, and radiography QC results
- All instrument calibration reports, as applicable

10.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 10-3 lists the major technical implementing procedures pertinent to this section of the QAPjP. The RWMC, ANL-W, and SPO implementation plans identify additional procedures.

Table 10-3. Section 10 implementing procedures.

Document Number	Title
TPR-1726	*TRU Waste Characterization Examination*
TPR-1572	*Operating the RTR System*
HFEF OI 6890	*Waste Characterization*

11. HYDROGEN AND METHANE ANALYSIS

ECL personnel receive samples in SUMMA® canisters resulting from the RWMC headspace gas sampling operations described in Section 7 of this QAPjP. ECL personnel analyze the samples for H_2 and CH_4 in accordance with Section 11.0 of the QAPP, as described in the ECL implementation plan and referenced procedures. RWMC personnel perform analyses for H_2 using an on-line RGA as described in the RWMC implementation plan. RWMC personnel also analyze headspace gas samples for CH_4 using FTIRS. The FTIRS requirements for CH_4 are included in Section 12 of this QAPjP.

11.1 Quality Assurance Objectives

Table 11-1 lists the QAOs for hydrogen and methane analysis. Key data quality indicators for H_2 and CH_4 measurements are defined below, and the methods to assess compliance with these indicators are presented in Section 3.2 of this QAPjP.

Table 11-1. Hydrogen and methane analysis quality assurance objectives.

Analyte	CAS Number	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	MDL (vol%)	PRQL (vol%)	Completeness (percent)
Hydrogen	1333-74-0	≤25	70-130	0.05	0.1	90
Methane	74-82-8	≤25	70-130	0.05	0.1	90

%RSD	=	Percent relative standard deviation
RPD	=	Relative percent difference
%R	=	Percent recovery
MDL	=	Method detection limit
PRQL	=	Program required quantitation limit

a. Criteria apply to PRQL concentrations

To demonstrate compliance with the QAOs, ECL and RWMC personnel:

- Measure precision by analyzing laboratory or on-line duplicates, replicate analyses of laboratory control samples, and PDP blind audit samples and calculate %RSD or RPD based on the results.
- Measure accuracy by analyzing laboratory or on-line control samples and PDP blind audit samples and calculate %R based on the results.
- Determine MDLs, expressed in units of volume percent for H_2 and CH_4 and ensure MDLs are less than or equal to those listed in Table 11-1.
- Demonstrate the ability to quantitate at or below the PRQLs given in Table 11-1 by setting the concentration of at least one calibration standard below the PRQL.
- Measure adherence to the 90% completeness criterion in Table 11-1 by calculating the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis.

- Achieve comparability by using standardized procedures and traceable standards and participating in the PDP for headspace gas analysis in accordance with the QAPP requirements

- At ECL, assure representativeness through cleaning and certification of SUMMA® canisters in accordance with Section 7.4 of the QAPP.

- At RWMC, assure representativeness through the use of the standardized headspace gas sampling methods described in Section 7 of this QAPjP and the Methods Manual.

The facility managers and the FQAOs are responsible for monitoring the results from these measurements and determining whether the precision, accuracy, and completeness criteria listed in Table 11-1 are met. They evaluate performance and decide whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

11.2 Methods Requirements

ECL personnel analyze headspace gas samples for H_2 and CH_4 using gas chromatography with thermal conductivity detection (GC/TCD). RWMC personnel analyze headspace gas samples for H_2 using on-line RGA. The GC/TCD method is based on Procedure 520.1 in the Methods Manual, and the RGA mass spectrometric method is based on Procedure 510.1 in the Methods Manual. Specific procedures for the analysis of headspace gas samples are described and identified in Section 11.2 of the ECL and RWMC implementation plans.

11.2.1 Standards

ECL and RWMC personnel purchase manufacturer certified primary H_2 and CH_4 standards from commercial sources (e.g., Scott Specialty Gases or equivalent). The ECL and RWMC implementation plans detail the requirements and identify procedures for the preparation of all standards. Purchased standards are traceable to NIST, EPA, or other nationally recognized sources.

11.2.2 Qualitative and Quantitative Analysis

All instruments used to analyze TWCP samples for H_2 and CH_4 meet the requirements of the Methods Manual. ECL and RWMC personnel quantitate H_2 and CH_4 within the calibration range of the analytical instrument being used. They dilute samples with concentrations greater than the calibration range of the analytical instrument or the calibration curve.

For GC/TCD, ECL personnel identify H_2 and CH_4 by retention time (RT). ECL personnel establish RT windows as a fixed percentage to either side of the mean RT of at least three calibration standards from the most recent valid initial calibration. ECL personnel ensure RT windows are established so the occurrence of both false positive and false negative results are minimized. The ECL implementation plan details RT window determination. ECL personnel quantitate H_2 and CH_4 based on the area response and report concentrations as volume percent.

For RGA, RWMC personnel identify and quantitate H_2 based on the positive ions formed by electron bombardment and determine the abundance of each mass present from the signal at the

corresponding mass-to-charge ratio. RWMC personnel calculate H_2 concentrations from the partial pressures of H_2 in a sample and report concentrations as volume percent.

11.3 Quality Control

The facility manager (or designated supervisor or technical leader) and FQAOs are responsible for monitoring and documenting procedure performance, including analyzing QC samples, and for implementing corrective action when acceptable procedure performance is not met. The ECL and RWMC implementation plans describe the actions to ensure the daily quality of data for H_2 and CH_4 analysis. ECL and RWMC personnel operate formal QC programs as described in the FIPs and maintain records to document the quality of the data generated. The FIPs and referenced analytical methods describe QC sample requirements and acceptance criteria, summarized in Table 11-2.

ECL and RWMC personnel demonstrate acceptable performance through the analysis of method performance samples as described in FIPs before analyzing any headspace gas samples. ECL and RWMC personnel purchase or prepare method performance samples that contain H_2 and/or CH_4 at concentrations appropriate to verify all QAOs in Table 11-1 are met. Initially, personnel analyze seven method performance samples to demonstrate acceptable precision and accuracy and determine MDLs for H_2 and/or CH_4. They demonstrate acceptable procedure performance semiannually by analyzing four method performance samples.

ECL personnel analyze headspace gas samples for H_2 and CH_4 in analytical batches (see Definitions section). Specific QC samples for each analytical batch include a laboratory blank, a laboratory duplicate, and a laboratory control sample. RWMC personnel analyze headspace gas samples for H_2 in on-line batches; specific QC samples for each on-line batch include an on-line blank, duplicate, and control sample.

ECL and RWMC personnel analyze field or on-line samples in duplicate at a frequency of one per analytical or on-line batch and prepare blanks using the same procedure used to prepare field samples for analysis. ECL personnel prepare laboratory blanks from high-purity nitrogen (99.999% pure). RWMC personnel analyze blanks on-line and use the same procedures used to prepare, introduce, and analyze samples in the RGA system to prepare, introduce, and analyze blanks.

ECL and RWMC personnel prepare laboratory or on-line control samples with commercially purchased gas standards independent of those used for instrument calibration and ensure control samples contain H_2 and/or CH_4 at concentrations in the calibration range of the analytical instrument. They also analyze PDP blind audit samples biannually to determine acceptable laboratory performance.

When QC sample acceptance criteria are not met, the facility manager or designee and the FQAO implement corrective action as described in the ECL and RWMC implementation plans. They ensure QC sample results are flagged as appropriate or an NCR is initiated if QC results associated with final reported sample data do not meet acceptance criteria.

11.4 Instrument Testing, Inspection, and Maintenance Requirements

ECL and RWMC personnel ensure analytical equipment are tested, inspected, and maintained. Maintenance programs ensuring the QAOs in Table 11-1 are met are summarized in the ECL and RWMC implementation plans.

Table 11-2. Summary of laboratory quality control samples and frequencies for hydrogen and methane analysis.

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 11-1 QAOs	Repeat until acceptable
Laboratory duplicates or on-line duplicates	One (1) per analytical or on-line batch	RPD ≤ 25[a]	Specified in FIPs[b]
Laboratory blanks or on-line blanks	One (1) per analytical or on-line batch	Analyte concentrations < PRQL	Specified in FIPs[b]
Laboratory control samples or on-line control samples	One (1) per analytical or on-line batch	70-130 %R	Specified in FIPs[b]
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan

PDP = Performance Demonstration Program
PRQL = Program required quantitation limit
QAO = Quality assurance objective
%R = Percent recovery
RPD = Relative percent difference

a. Applies only to concentrations greater than the PRQL listed in Table 11-1.

b. A nonconformance report, per Section 2.1.2 of the QAPjP, is required when quality control samples associated with final reported sample data do not meet Table 11-2 acceptance criteria.

11.5 Instrument Calibration and Frequency

ECL and RWMC personnel calibrate all instruments before use and verify calibration at routine intervals during analysis. ECL and RWMC personnel ensure all instruments are calibrated in accordance with the Methods Manual procedures, manufacturers' recommendations, and the QAPD, and maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency. The FIPs describe and identify calibration procedures and records. Table 11-3 summarizes calibration requirements for H_2 and CH_4 analysis.

ECL personnel obtain an initial GC/TCD multipoint calibration curve for H_2 and CH_4 that consists of a minimum of three standards. They ensure the low standard is at a concentration less than the PRQL listed in Table 11-1 and the high standard is at a concentration such that it brackets the expected sample concentrations, yet remains within the linear range of the instrument. ECL personnel use linear regression equations (plotting area response versus concentration) or average response factors to construct the calibration plot and ensure initial calibrations meet the criteria listed in Table 11-3.

ECL personnel analyze a GC/TCD continuing calibration verification standard at the beginning of each 12-hour shift, prior to sample analysis. They compare the known concentration of the continuing calibration verification standard to the corresponding concentration determined from the most recent valid initial calibration. If the Table 11-3 acceptance criteria are not met, ECL personnel either run another continuing calibration or generate a new calibration plot. They do not continue sample analysis until the requirements in Table 11-3 are met.

RWMC personnel calibrate the RGA instrument prior to the analysis of any samples to establish a mass pattern and sensitivity for H_2. They ensure the initial calibration meets the criteria listed in Table 11-3. RWMC personnel perform continuing mass and sensitivity calibration at the beginning and end of each analytical batch run and ensure the continuing calibration meets the criteria listed in Table 11-3. If the partial pressure sum differs from the total sample pressure, RWMC personnel assess the problem and take and document appropriate corrective action.

ECL and RWMC personnel may use the laboratory or on-line control sample for a GC/TCD or RGA continuing calibration; however, if they do not use the control sample for continuing calibration, they run the control sample during the analytical sequence. ECL and RWMC personnel ensure the continuing calibration gas standard is from a separate source than that used for the initial instrument calibration, and that it contains all target analytes for the method at concentrations within the calibration range of the analytical instrument.

11.6 Data Management

ECL and RWMC personnel do not blank-correct H_2 and CH_4 results, but report results from blank analysis separately from field sample results. ECL and RWMC personnel review and approve all analytical data, as defined in Section 3.1.1 of this QAPjP, and report all H_2 and CH_4 results in units of volume percent.

The FIPs describe ECL and RWMC data reduction, validation, and reporting processes, and include or reference example forms or describe electronic systems to be used to record and report data. ECL and RWMC personnel reduce raw data to reportable results in compliance with the Methods Manual.

ECL and RWMC personnel submit analytical batch data reports and on-line batch reports, respectively, to the SDCO and maintain analytical records as specified in Section 3.4.1 of this QAPjP.

Table 11-3. Summary of MS (RGA) and GC calibration requirements for hydrogen and methane analysis.

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
RGA	Mass alignment	Initially and as needed	Base peak of 2 for hydrogen
RGA	Initial instrument calibration	Beginning of each on-line batch	$70 \geq \%R \leq 130$ for each analyte from the continuing calibration/on-line control standard
RGA	Continuing calibration	Beginning of each on-line batch	$\leq 10\%$
RGA	Continuing calibration duplicate	End of each on-line batch	$\leq 10\%$
GC	3-pt initial calibration (3 standards)	Initially and as needed	%RSD of response factor for each analyte ≤ 35 -or- Linear regression plot yields straight line and %R for each analyte is 70-130
	Continuing calibration	Every 12 hours	$\%D \leq 30$ for each analyte

RGA	=	Residual gas analysis
%D	=	Percent difference
%R	=	Percent recovery
%RSD	=	Percent relative standard deviation

These records are subject to assessment by representatives from the SPO on a regular basis as described in Section 3.1.2. Analytical batch data report requirements include the following:

- Table of Contents
- Cross reference to field sample numbers
- A COC form showing the date and time of sample transfer and names of individuals handling the samples from the time of sampling through receipt at the laboratory
- Signature releases as specified in the QAPP
- Copies of sample tags
- Data review checklists for each batch verifying that data generation level review, validation, and verification have taken place

- QC sample results (e.g., laboratory duplicates, laboratory control samples) if not included in data review checklists

- A separate analytical report sheet titled "Hydrogen and Methane Analysis Data Sheet," for each sample in the batch, including facility name, program name, analytical batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date analyzed, method number, listing of program analytes, and analytical results in volume percent. The following data qualifying flags are used:

  - B analyte detected in blank
  - E Analyte exceeds the calibration curve
  - H Holding time exceeded
  - J Analyte is less than PRQL but greater than or equal to MDL
  - U Analyte was undetected (report MDL)
  - D Analyte was quantitated from a secondary dilution
  - Z One or more QC samples do not meet acceptance criteria

- Nonconformance reports, if applicable

ECL and RWMC personnel maintain the following records in their files, documented and retrievable by analytical batch numbers and data report numbers:

- Original COC records for analytical batches (not applicable to on-line samples)

- All raw data, including original instrument readouts and/or bench reports, calculation records, and QC sample results. Duplicate results are recorded along with the original sample results, and the RPD between the two results are calculated. Control sample results are entered with the accepted value and the %R.

- Instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the laboratory name, initial and continuing calibration verification source, method identification, and calibration date and time.

- QC result summary, which includes true and found values for all QC samples plus associated result calculations. At a minimum, the QC data include blanks, control samples, duplicates, initial calibration data, initial and continuing calibration verifications, and all other method-specific QC listed in Section 11. The QC result summary includes the facility name, the batch number (if applicable), and method names.

- Original field sample canister tags (or equivalent documentation) for headspace gas samples

NOTE: *TRIPS electronic data reporting includes the same information identified above. The TRIPS data is validated using electronic checklists. TRIPS utilizes a password protected electronic signature process.*

11.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 11-4 lists the major technical implementing procedures pertinent to this section of the QAPjP. The ECL and RWMC implementation plans identify additional procedures.

Table 11-4. Section 11 implementing procedures.

Document number	Title
ACLP-4.10	*Determination of MDLs for Gas Analysis*
ACMM-9920	*Analysis of Gas Samples for Hydrogen and Methane by GC/TCD*
RWMC TPR - 1584	*Headspace sampling/RGA/FTIRS System Operation*
RWMC MCP-1815	*On-Line HSS/RGA/FTIRS Level 1 Data Validation*
RWMC TPR-1612	*RGA Analysis for Hydrogen*

12. GAS VOLATILE ORGANIC COMPOUND ANALYSIS

ECL personnel receive samples in SUMMA® canisters resulting from the RWMC headspace gas sampling operations described in Section 7 of this QAPjP. ECL personnel analyze the samples for VOCs using gas chromatography/mass spectrometric detection (GC/MS) and gas chromatography/flame ionization detection (GC/FID) in accordance with Section 12.0 of the QAPP as described in the ECL implementation plan and referenced procedures.

RWMC personnel analyze headspace gas samples for CH_4 and VOCs using FTIRS as part of an on-line integrated sampling/analysis system. The system uses the manifold sample collection system described in Section 7 of this QAPjP and directs gas samples directly to an FTIRS analyzer. RWMC personnel sample and analyze headspace gas samples for CH_4 and VOCs as described in the RWMC implementation plan.

12.1 Quality Assurance Objectives

Table 12-1 lists the QAOs for headspace gas VOC analysis. The following text defines key data quality indicators for ECL and RWMC measurements and Section 3.2 of this QAPjP presents the methods to assess compliance with these indicators. To demonstrate compliance with the QAOs, ECL and RWMC personnel:

- Measure precision by analyzing laboratory or on-line duplicates, replicate analyses of laboratory control samples, and PDP blind audit samples and calculate %RSD or RPD based on the results.

- Measure accuracy by analyzing laboratory or on-line control samples and PDP blind audit samples and calculate %R based on the results.

- ECL personnel determine GC/MS and GC/FID MDLs, expressed in nanograms for VOCs and ensure MDLs are less than or equal to those listed in Table 12-1.

- RWMC personnel determine FTIRS MDLs for CH_4 expressed in volume % and for VOCs expressed in ppmv*m and ensure MDLs are less than or equal to the FTIRS MDLs listed in Table 12-1.

- Demonstrate the ability to quantitate at or below the PRQLs given in Table 12-1 by setting the concentration of at least one calibration standard below the PRQL.

- Measure adherence to the 90% completeness criterion in Table 12-1 by calculating the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis.

- Achieve comparability by using standardized procedures and traceable standards and participating in the PDP for headspace gas analysis in compliance with QAPP requirements.

- At ECL, assure representativeness through cleaning and certification of SUMMA® canisters in accordance with Section 7.4 of the QAPP.

Table 12-1. Gas volatile organic compounds target analyte list and quality assurance objectives.

Compound	CAS Number	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	MDL[b] (ng)	FTIRS MDL (ppmv*m)	PRQL[b] (ppmv)	Completeness (percent)
Benzene	71-43-2	≤25	70-130	10	5	10	90
Bromoform	75-25-2	≤25	70-130	10	5	10	90
Carbon tetrachloride	56-23-5	≤25	70-130	10	5	10	90
Chlorobenzene	108-90-7	≤25	70-130	10	5	10	90
Chloroform	67-66-3	≤25	70-130	10	5	10	90
Cyclohexane	110-87-7	≤25	70-130	10	5	10	90
1,1-Dichloroethane	75-34-3	≤25	70-130	10	5	10	90
1,2-Dichloroethane	107-06-2	≤25	70-130	10	5	10	90
1,1-Dichloroethylene	75-35-4	≤25	70-130	10	5	10	90
cis-1,2-Dichloroethylene	156-59-2	≤25	70-130	10	5	10	90
Ethyl benzene	100-41-4	≤25	70-130	10	10	10	90
Ethyl ether	60-29-7	≤25	70-130	10	5	10	90
Methane	74-82-8	≤25	70-130	---	0.05[d]	0.1[d]	90
Methylene chloride	75-09-2	≤25	70-130	10	5	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤25	70-130	10	5	10	90
Tetrachloroethylene	127-18-4	≤25	70-130	10	5	10	90
Toluene	108-88-3	≤25	70-130	10	5	10	90
1,1,1-Trichloroethane	71-55-6	≤25	70-130	10	5	10	90
Trichloroethylene	79-01-6	≤25	70-130	10	5	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1 95-63-6	≤25	70-130	10	5	10	90
1,2,4-Trimethylbenzene	108-67-8	≤25	70-130	10	5	10	90
1,3,5-Trimethylbenzene	108-38-3	≤25	70-130	10	5	10	90
m-Xylene[c]	95-47-6	≤25	70-130	10	5	10	90
o-Xylene	106-42-3	≤25	70-130	10	5	10	90
p-Xylene[c]		≤25	70-130	10	5	10	90
Acetone	67-64-1	≤25	70-130	150	50	100	90
Butanol	71-36-3	≤25	70-130	150	50	100	90
Methanol	67-56-1	≤25	70-130	150	50	100	90
Methyl ethyl ketone	78-93-3	≤25	70-130	150	50	100	90
Methyl isobutyl ketone	108-10-1	≤25	70-130	150	50	100	90

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

MDL = Method detection limit [maximum permissible value, total number of nanograms (ng) delivered to the analytical system per sample]

PRQL = Program required quantitation limit

a. Criteria apply to PRQL concentrations.

b. Values based on delivering 10 mL to the analytical system.

c. These xylene isomers cannot be resolved by the GC/MS analytical methods employed in this program.

d. For methane analyzed only by FTIRS, MDL and PRQL are given in volume percent.

- At RWMC, assure representativeness through the use of the standardized headspace gas sampling methods described in Section 7 of this QAPjP and the Methods Manual.

The facility managers and FQAOs are responsible for monitoring the results of these measurements and determining whether the precision, accuracy, and completeness criteria listed in Table 12-1 have been met. They evaluate ECL and RWMC performance and decide whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

12.2 Methods Requirements

ECL personnel analyze headspace gas samples for the VOCs listed in Table 12-1 using GC/MS and GC/FID as described in Section 12.2 of the ECL implementation plan. The ECL GC/MS method is based on Procedure 430.1 in the Methods Manual. The ECL GC/FID method is based on Procedure 440.1 in the Methods Manual.

ECL personnel use equipment and materials that meet all Methods Manual requirements for both GC/MS and GC/FID methods. They equip gas chromatographs with two capillary columns based on those recommended in the Methods Manual. They operate GC/MS systems in the full scan mode to detect and quantitate target analytes and identify nontarget compounds.

RWMC personnel collect and analyze headspace gas samples for the VOCs listed in Table 12-1 and CH_4 using FTIRS as described in Section 12.2 of the RWMC implementation plan. The RWMC FTIRS methods is based on Procedure 430.7 in the Methods Manual. RWMC personnel use equipment and materials that meet all Methods Manual requirements.

12.2.1 Standards

ECL and RWMC personnel purchase certified primary standards from the best available commercial source. Purchased standards are traceable to NIST, EPA, or other nationally recognized standards. They prepare all secondary and calibration standards according to procedures identified in the ECL and RWMC implementation plans. All standards are analyzed at the same temperature as the samples (±2°C).

12.2.2 GC/MS Qualitative and Quantitative Analysis

ECL personnel qualitatively identify target VOCs as described in the ECL implementation plan by ensuring the analytes elute within ±0.06 relative retention time (RRT) units of the RRT of the continuing calibration check standard and have a mass spectrum that corresponds to the standard mass spectrum. They define RT windows as ±0.5 minutes from the compound's absolute RT based on the analysis of a calibration single standard. They determine RTs for all analytes prior to the analysis of any samples and whenever a new GC column is installed as described in the ECL implementation plan and referenced GC/MS procedures.

ECL personnel use internal standard quantitation for GC/MS quantitative VOC analysis, as described in the ECL implementation plan. They meet the %RSD criteria for all analytes or generate a second- or third-order regression calibration curve. They calculate %RSD as the standard deviation of relative response factors for an analyte divided by the mean of the initial response factors for that analyte. They use the integrated abundance from the Extracted Ion Current Profile (EICP) of the primary

characteristic ion to calculate concentrations. They quantitate analytes within the calibration range of the analytical instrument and dilute samples with concentrations greater than the calibration range of the instrument or calibration curve.

12.2.3 GC/FID Qualitative and Quantitative Analysis

ECL personnel establish RT windows for all analytes for GC/FID qualitative analysis. They positively identify all analytes by RT confirmation on both columns and ensure that sample component peaks fall within the appropriate RT window. They determine RT windows for both columns with each initial calibration. They calculate RT windows as the mean RT of the initial calibration standards plus or minus a percentage of the mean RT. ECL personnel ensure RT windows are established so the occurrence of both false positive and false negative results are minimized.

They determine RT windows for all analytes on each GC column prior to the analysis of any samples, whenever a new initial calibration is performed, and whenever a new GC column is installed. RT window determination is described in the ECL implementation plan and GC/FID procedures.

ECL personnel quantitate analytes using one of the two columns. They ensure that the column used for quantitation is free of interferants in the RT window corresponding to the analyte. They generate average response factors or regression equations for each specified target analyte and quantitate all analytes within the calibration range of the analytical instrument.

12.2.4 FTIRS Qualitative and Quantitative Analysis

RWMC personnel use a multivariate analysis technique for FTIRS qualitative and quantitative analysis. They use partial least squares (PLS) as described in the RWMC implementation plan and FTIRS procedures. They generate a set of factors as a result of the PLS training/calibration step. The factors describe the analyte(s) of interest and any interference(s) included in the calibration set.

12.3 Quality Control

The ECL and RWMC facility managers (or designated supervisor or technical leader) and the FQAOs are responsible for monitoring and documenting procedure performance, including the analysis of QC samples and for implementing corrective action when procedure performance is not acceptable. The ECL and RWMC implementation plans describe the actions to ensure the daily quality of data for headspace gas CH_4 and VOC analysis. ECL and RWMC personnel operate formal QC programs, as described in the FIPs, and maintain records to document the quality of the data generated. The FIPs and referenced analytical procedures describe all QC elements established by the analytical methods, including the analysis of QC samples summarized in Table 12-2.

ECL and RWMC personnel demonstrate acceptable performance prior to the analysis of any headspace gas samples through the analysis of method performance samples in accordance with ECL and RWMC implementation plans. They purchase or prepare method performance samples and ensure the samples contain the analytes listed in Table 12-1 at concentrations appropriate to verify all QAOs are met. Initially, they analyze seven method performance samples to demonstrate acceptable precision and accuracy and determine MDLs for the target analytes of interest. They demonstrate acceptable procedure performance semiannually by analyzing four method performance samples.

ECL personnel analyze samples in analytical batches and RWMC personnel analyze samples in on-line batches (see Definitions section). Specific QC samples for each analytical batch and on-line batch include a blank, a duplicate, and a control sample. RWMC personnel also collect and analyze a comparison sample for analysis by GC/MS at ECL once per on-line batch.

ECL personnel analyze field samples in duplicate at a frequency of one per analytical batch. They prepare laboratory blanks by filling SUMMA® canisters with high-purity humid zero air or nitrogen (99.999% pure) and use the same sample preparation procedures used for field samples.

Table 12-2. Summary of quality control samples and frequencies for gas volatile organic compounds analyses.

QC sample	Minimum frequency	Acceptance criteria	Corrective action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 12-1 QAOs	Repeat until acceptable
Laboratory duplicates or on-line duplicates	One (1) per analytical batch for GC/MS and GC/FID. One (1) per on-line batch for FTIRS	RPD $\leq$25[a]	Specified in FIPs [b]
Laboratory blanks or on-line blanks	Daily prior to sample analysis for GC/MS and GC/FID. Daily prior to sample analysis and one (1) per on-line batch for FTIRS	Analyte amounts <3 * MDLs for GC/MS and GC/FID; <PRQL for FTIRS	Specified in FIPs [b]
Laboratory control samples or on-line control samples	One (1) per analytical batch for GC/MS and GC/FID. One (1) per on-line batch for FTIRS	70-130 %R	Specified in FIPs [b]
GC/MS comparison sample (for FTIRS only)	One (1) per on-line batch	RPD $\leq$25	Nonconformance if RPD >25[b]
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan

MDL = Method detection limit
PDP = Performance Demonstration Program
QAO = Quality assurance objective
%R = Percent recovery
RPD = Relative percent difference

a. Applies only to concentrations greater than the PRQLs listed in Table 12-1.

b. A nonconformance report per Section 2.1.2 of this QAPjP is required when quality control samples associated with final reported data do not meet Table 12-2 acceptance criteria.

RWMC personnel analyze field samples in duplicate at a frequency of one per on-line batch. They use hydrocarbon- and CO_2 -free dry air for blanks. They collect all blanks through the sampling manifold

and use the same procedures used to prepare, introduce and analyze samples in the FTIRS system to prepare, introduce, and analyze blanks. If the field blank meets the acceptance criterion, they may choose to eliminate the on-line blank for that batch.

ECL and RWMC personnel prepare control samples from gas or liquid standards independent of those used for instrument calibration. They ensure GC/MS laboratory control samples contain at least six of the analytes listed in Table 12-1, GC/FID laboratory control samples contain all alcohol and ketone target compounds, and FTIRS on-line control samples contain 10 analytes listed in Table 12-1. They ensure target analytes contained in control samples are present at concentrations in the calibration range of the analytical instrument. They also analyze PDP blind audit samples biannually to determine acceptable laboratory or system performance.

When QC sample acceptance criteria are not met, the facility manager or designee and the FQAOs implement corrective action as described in the ECL and RWMC implementation plans. They ensure QC sample results are flagged as appropriate or an NCR is initiated if final reported QC sample results do not meet acceptance criteria.

12.4 Instrument Testing, Inspection, and Maintenance Requirements

ECL personnel ensure analytical equipment are tested, inspected, and maintained according to manufacturer's specifications, the QAPD, and the Methods Manual. The ECL maintenance program is summarized in the ECL implementation plan.

ECL personnel meet GC/MS instrument performance criteria prior to the analysis of any standards or samples by meeting the 4-bromofluorobenzene (BFB) criteria specified in the Methods Manual. They check the BFB criteria at the beginning of each 12 hours of operation by analyzing 50 ng of BFB.

ECL personnel introduce samples into GC/FID systems by thermostated gas injection valves with sample loops that permit the injection of gas standards directly on column. They equip the system's gas chromatograph with two, dissimilar, wide-bore capillary columns, as specified in the Methods Manual.

RWMC personnel ensure FTIRS equipment is tested, inspected, and maintained according to manufacturers' specifications and the Methods Manual. The RWMC maintenance program is summarized in the RWMC implementation plan. The RWMC uses FTIRS equipment and materials that meet all Methods Manual requirements. Personnel use a sample cell with a path length that allows the MDLs to be met. They use sampling heads and manifolds that meet all requirements found in Section 7.0 of the QAPP and the Methods Manual.

12.5 Instrument Calibration and Frequency

ECL and RWMC personnel calibrate all analytical instruments before use and verify calibration at routine intervals during analysis as described in the FIPs. Analytical procedures comply with calibration requirements summarized in Table 12-3. ECL and RWMC personnel ensure all initial and continuing calibration requirements listed in Table 12-3 are met.

ECL personnel ensure all instruments located at the ECL are calibrated in accordance with the analytical procedures and maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency. ECL calibration procedures and records are described and identified in the ECL

implementation plan. RWMC personnel ensure all instruments located at the RWMC are calibrated in accordance with the analytical procedure and maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency. RWMC calibration procedures and records are described in the RWMC implementation plan.

ECL personnel satisfy GC/MS instrument performance criteria, then use at least five standards to define the calibration range of the instrument for the analytes of interest, setting the concentration of one standard less than the PRQLs listed in Table 12-1. They generate relative response factors for each specified target analyte. They ensure the initial calibration meets all of the acceptance criteria listed in Table 12-3. If linearity is not demonstrated, they use a second- or third-order regression for calibration. They ensure a valid initial calibration exists before any samples are analyzed.

Table 12-3. Summary of calibration requirements for gas volatile organic compounds analyses.

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
GC/MS	BFB Tune	Every 12 hours	Abundance criteria for all key ions are met (see Methods Manual Procedure 430.1)
	5-pt initial calibration (5 standards)	Initially, and as needed	%RSD of response factor for each analyte <35
	Continuing calibration	Every 12 hours	%D for all compounds ≤30 of initial calibration
GC/FID	3-pt initial calibration (3 standards)	Initially, and as needed	%RSD of response factor for each analyte <30 -or- linear regression plot yields straight line and %R is 70-130 for each standard analyte
	Continuing calibration	Every 12 hours	%D for all compounds ≤30 of initial calibration; RTs within most recently established RT window
FTIRS	Initial calibration spectra for analyte components, interferences, and background components	Initially and as needed	Meets PLS requirements
	Continuing calibration	Once per on-line batch	%R of 70-130 for 10 analytes in on-line control sample

BFB = 4-Bromofluorobenzene
%D = Percent difference
PLS = Partial least s
%RSD = Percent relative standard deviation quares
%R = Percent recovery
RT = Retention time

ECL personnel use a midpoint calibration standard to verify the initial GC/MS calibration curve at the beginning of every 12 hours of operation after satisfying the instrument performance criteria using 50 ng of BFB. They determine the %D using continuing calibration response factors and average relative response factors from the most recent initial calibration and ensure the %Ds meet Table 12-3 acceptance

criteria. If second- or third-order regression is used, they ensure the continuing calibration verification standard point falls within 30% of the curve value from the associated initial calibration. They generate a new five-point (minimum) calibration curve if the continuing calibration standard does not satisfy the acceptance criteria and do not proceed with sample analysis until the GC/MS system has satisfied the appropriate daily calibration criteria.

ECL personnel use at least three external standards to define the calibration range of the GC/FID system for the alcohols and ketones, setting the concentration of one standard less than the PRQLs specified in Table 12-1. They generate average response factors or a linear regression equation to construct the calibration plot, plotting area response versus concentration, and ensure the initial calibration meets the acceptance criteria listed in Table 12-3. They verify the initial GC/FID calibration curve with a midpoint calibration standard (continuing calibration verification standard) at the beginning of each 12 hours of operation. They compare known concentrations for the continuing calibration standard to the corresponding concentration determined from the most recent valid three-point calibration. They ensure that the RT of each analyte falls within the applicable RT window. They generate a new three-point initial calibration curve if the continuing calibration standard does not meet the requirements of Table 12-3 and do not proceed with sample analysis until the GC/FID system has satisfied the calibration criteria.

RWMC personnel calibrate the FTIRS using a relatively large set of training/calibration spectra and the PLS algorithm. They ensure the initial set of calibration spectra consists of a minimum of two pure component spectra of the analyte(s) of interest, two pure component spectra of each suspected interference, and additional spectra which demonstrate background components such as water or CO_2. They use independent PLS algorithms for each analyte of interest so the optimal spectral region for each analyte is used to minimize the effects of interferences and widely different sample compositions. They use the on-line control sample as the continuing calibration check (continuing calibration verification standard) and ensure the acceptance criterion listed in Table 12-3 is met.

12.6 Data Management

ECL personnel quantify analyte concentrations using average relative response factors for GC/MS results and average response factors or a linear regression equation derived from the initial calibration for GC/FID results. They do not blank-correct target analyte concentrations but report blank results separately from field sample results.

ECL personnel report nontarget compounds as tentatively identified compounds (TICs) with a higher uncertainty than target analyte concentrations. They perform appropriate search routines of the latest NIST or Wiley mass spectral library on the 20 TICs with the greatest area counts and with total ion current peaks greater than 10% of the nearest (RT) internal standard. The SPM adds the positively identified TICs listed in 40 CFR Part 264, Appendix IX, to the target analyte list if they are detected in 25% of all samples from a given IDC. ECL personnel assume a relative response factor equal to one, using the nearest internal standard, when estimating concentration for TICs.

ECL data reduction, validation, and reporting processes are described in the ECL implementation plan. Analytical procedures include or reference example forms or describe electronic systems to be used to record and report data. ECL personnel reduce raw data to reportable results in compliance with the Methods Manual. They report analytical results in units of ppmv, limited to two significant figures. ECL personnel review, validate, and verify data as described in Section 3.1.1 of this QAPjP.

RWMC personnel assume that an unmodeled interferant or TIC is present in a sample if spectral residuals are still prevalent after dilution. They subtract contributions from compounds found in the sample from the original sample spectrum. They interpret the resulting spectrum for functional groups and compound identifications. They perform a library search to determine the five most likely compounds contributing to the interference. They collect a sample containing the interfering compound(s) in a SUMMA® canister and submit this sample to the ECL for GC/MS analysis to confirm the identity of the unknown compound(s).

RWMC data reduction, validation, and reporting processes are described in the RWMC implementation plan. Analytical procedures include or reference example forms or describe electronic systems to be used to record and report data. RWMC personnel reduce raw data to reportable results in compliance with Methods Manual procedures. They report analytical results for CH_4 in volume percent and for VOCs in units of ppmv, limited to two significant figures. RWMC personnel review, validate, and verify data as described in Section 3.1.1 of this QAPjP.

ECL and RWMC personnel submit batch data reports to the SDCO and maintain analytical records as specified in Section 3.4.1 of this QAPjP. These records are subject to assessment by representatives from the SPO on a regular basis as discussed in Section 3.1.2. Analytical batch data report requirements include the following:

- Laboratory name, analytical or on-line batch number, sample numbers included in that batch, a cross reference to field sample numbers, and the signature releases of laboratory personnel specified in Section 3.1.1 of the QAPP
- Table of Contents
- A COC form showing the date and time of sample transfer and names of individuals handling the samples from the time of sampling through receipt at the laboratory
- Copies of sample tags
- Data review checklists for each batch verifying that data generation level review, validation, and verification have taken place.
- QC sample results (e.g., laboratory duplicates, laboratory control samples) if not included in data review checklists.
- A separate analytical report sheet titled, "Gas VOC Analysis Data Sheet," for each sample in the batch, including facility name, program name, analytical batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date analyzed, method number, listing of program analytes, and analytical results in ppmv. The following data qualifying flags are used:
  - B Analyte detected in blank
  - E Analyte exceeds the calibration curve
  - H Holding time exceeded

- J Analyte is less than PRQL but greater than or equal to MDL
- U Analyte was undetected (report MDL)
- D Analyte was quantitated from a secondary dilution or reduced volume aliquot
- Z One or more QC samples do not meet acceptance criteria

- Nonconformance reports, if applicable

ECL and RWMC personnel maintain the following records in their files, documented and retrievable by analytical batch numbers and data report numbers:

- Original COC records for analytical batches (not applicable to on-line samples)
- All raw data, including original instrument readouts and/or bench reports, calculation records, and QC sample results. Duplicate results are recorded along with the original sample results, and the RPD between the two results are calculated. Control sample results are entered with the accepted value and the %R.
- Instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the laboratory name, initial and continuing calibration verification source, method identification, and calibration date and time.
- QC result summary, which includes true and found values for all QC samples plus associated result calculations. At a minimum, the QC data include blanks, control samples, duplicates, initial calibration data, initial and continuing calibration verifications, and all other method-specific QC listed in Section 12. The QC result summary includes the name, the batch number (if applicable), and method names.
- Original field sample canister tags (or equivalent documentation) for headspace gas samples

NOTE: *TRIPS electronic data reporting includes the same information identified above. The TRIPS data is validated using electronic checklists. TRIPS utilizes a password protected electronic signature process.*

12.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 12-4 lists the major technical implementing procedures pertinent to this section of the QAPjP. The ECL and RWMC implementation plans identify additional procedures.

Table 12-4. Section 12 implementing procedures.

Document Number	Title
ACLP-4.10	*Determination of MDLs for Gas Analysis*

ACMM-9930	*Analysis of Gas Samples for VOCs by GC/MS Using MFC Sample Introduction*
ACMM-9910	*Analysis of Gas Samples for Alcohols and Ketones by GC/FID*
TPR 1584	*Drum Venting System (DVS)*
MCP-1815	*RWMC/SWEPP On-Line HSS Level 1 Validation Procedure*
TPR-1613	*FTIRS analysis for VOCs and Methane*

13. TOTAL VOLATILE ORGANIC COMPOUND ANALYSIS

ACL personnel receive solidified samples resulting from the ANL-W homogeneous solids and soil/gravel sampling operations described in Section 8 of this QAPjP. ACL personnel analyze the samples for total VOCs in accordance with Section 13.0 of the QAPP, as described in the ACL implementation plan.

13.1 Quality Assurance Objectives

Table 13-1 lists the QAOs for total VOC analysis. The following text defines key data quality indicators for ACL measurements and Section 3.2 of this QAPjP presents methods to assess compliance with these indicators. To demonstrate compliance with the QAOs, ACL personnel:

- Measure precision by analyzing matrix spike duplicates, replicate analyses of laboratory control samples, and PDP blind audit samples and calculate %RSD or RPD based on the results.

- Measure accuracy by analyzing laboratory control samples, matrix spikes, surrogate compounds, and PDP blind audit samples and calculate %R based on the results.

- Determine MDLs, expressed in mg/kg, and ensure MDLs are less than or equal to those listed in Table 13-1.

- Demonstrate the ability to quantitate at or below the PRQLs in Table 13-1 by setting the concentration of at least one calibration standard below the PRQL.

- Measure adherence to the 90% completeness criterion by calculating the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis.

- Achieve comparability by using standardized procedures and traceable standards and participating in the PDP for RCRA constituent analysis of solidified wastes in compliance with the QAPP requirements

ANL-W personnel assure representativeness through the use of standardized and approved sampling methods described in Section 8 of this QAPjP and the Methods Manual.

The ALD manager and the ALD FQAO are responsible for monitoring the results from these measurements; determining whether precision, accuracy, and completeness requirements are met. They evaluate ACL performance and decide whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

13.2 Methods Requirements

ACL personnel analyze homogeneous solids and soil/gravel samples for total VOCs using ion trap GC/MS and GC/FID methods as described in Section 13.2 of the ACL implementation plan. The GC/MS method is based on Procedure 430.4 in the Methods Manual. The GC/FID method is based on Procedure 440.2 in the Methods Manual. ACL personnel use GC/FID for the analysis of samples for acetone,

Table 13-1. Total volatile organic compounds target analyte list and quality assurance objectives.

Compound	CAS Number	Precisiona (%RSD or RPD)	Accuracy[a] (%R)	MDL (mg/kg)	PRQL (mg/kg)	Completeness (%)
Benzene	71-43-2	≤45	37-151	1	10	90
Bromoform	75-25-2	≤47	45-169	1	10	90
Carbon disulfide	75-15-0	≤50	60-150	1	10	90
Carbon tetrachloride	56-23-5	≤30	70-140	1	10	90
Chlorobenzene	108-90-7	≤38	37-160	1	10	90
Chloroform	67-66-3	≤44	51-138	1	10	90
1,4-Dichlorobenzene[b]	106-46-7	≤60	18-190	1	10	90
ortho-Dichlorobenzene[b]	95-50-1	≤60	18-190	1	10	90
1,2-Dichloroethane	107-06-2	≤42	49-155	1	10	90
1,1-Dichloroethylene	75-35-4	≤250	D-234[c]	1	10	90
Ethyl benzene	100-41-4	≤43	37-162	1	10	90
Methylene chloride	75-09-2	≤50	D-221[c]	1	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤55	46-157	1	10	90
Tetrachloroethylene	127-18-4	≤29	64-148	1	10	90
Toluene	108-88-3	≤29	47-150	1	10	90
1,1,1-Trichloroethane	71-55-6	≤33	52-162	1	10	90
1,1,2-Trichloroethane	79-00-5	≤38	52-150	1	10	90
Trichloroethylene	79-01-6	≤36	71-157	1	10	90
Trichlorofluoromethane	75-69-4	≤110	17-181	1	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤50	60-150	1	10	90
Vinyl chloride	75-01-4	≤200	D-251[c]	1	4	90
m-xylene[e]	108-38-3	≤50	60-150	1	10	90
o-xylene.	95-47-6	≤50	60-150	1	10	90
p-xylene[e]	106-42-3	≤50	60-150	1	10	90
Acetone	67-64-1	≤50	60-150	10[d]	100	90
Butanol	71-36-3	≤50	60-150	10[d]	100	90
Ethyl ether	60-29-7	≤50	60-150	10[d]	100	90
Isobutanol	78-83-1	≤50	60-150	10[d]	100	90
Methanol	67-56-1	≤50	60-150	10[d]	100	90
Methyl ethyl ketone	78-93-3	≤50	60-150	10[d]	100	90
Pyridine[b]	110-86-1	≤50	60-150	10[d]	100	90

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

MDL = Method detection limit (maximum permissible value)

PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for benzene assuming a 25 g sample, 0.5 L of extraction fluid, and 100% analyte extraction

a. Criteria apply to PRQL concentrations.

b. Can also be analyzed as a semivolatile organic compound.

c. Detected; result must be greater than zero.

d. Estimate, to be determined.

e. These xylene isomers cannot be resolved by the analytical methods employed in this program.

butanol, ethyl ether, isobutanol, methanol, methyl ethyl ketone, and pyridine. They use GC/MS for the analysis of samples for all of the other VOCs listed in Table 13-1. ACL personnel use appropriate sample preparation methods based on those included in Procedures 430.4 and 440.2 in the Methods Manual.

13.2.1 Standards

ACL personnel purchase certified primary standards from the best available source. Commercially-purchased primary standards are certified by the manufacturer and their concentrations are traceable to the NIST, EPA, or other nationally-recognized standards. ACL personnel prepare secondary analytical standards, surrogate standards, calibration standards, and matrix spiking standards as specified in the appropriate Methods Manual procedure. The ACL implementation plan describes and identifies procedures used to prepare standards.

13.2.2 Qualitative and Quantitative Analysis

ACL personnel qualitatively identify analytes using GC/MS by ensuring the analytes elute within an RRT window of plus or minus 0.06 RRT units of the RRT of the continuing calibration standard and have a mass spectrum that corresponds to the standard analyte mass spectrum. They calculate RRT windows from the individual RTs in the associated continuing calibration standard within the same 12 hours as the sample.

ACL personnel use internal standards for quantitating target analytes by GC/MS. ACL personnel meet the criteria for system performance check compounds (SPCCs) and calibration check compounds (CCCs), and calculate response factors and the %RSD for response factors for all analytes. They meet all daily BFB tune and calibration criteria for SPCCs, CCCs, internal standard responses, and RTs. They quantitate each target analyte based on the integrated abundance from the EICP of the primary ion. They quantitate all target analytes within the calibration range of the instrument.

ACL personnel use external standard quantitation for GC/FID analysis and base target analyte identification on RT confirmation on each of two different columns. ACL personnel determine RT windows for both columns with each initial calibration (ICAL) and calculate windows as the mean RT of the ICAL standards plus or minus a fixed percentage for each analyte. ACL personnel ensure RT windows are established so the occurrence of both false positive and false negative results are minimized. ACL personnel determine RT windows per the QAPP as described in the ACL implementation plan. ACL personnel quantitate target analytes against external standards on one of the two columns. They base analyte quantitation on the peak area response, or peak height response, from one of the two columns. They ensure the column used for quantitation is free of interferences in the analyte's RT window and quantitate by a three-point (minimum) calibration curve or response factor.

Refer to the ACL implementation plan (INEEL 1999a) for a more detailed discussion of the qualitative and quantitative analysis.

13.3 Quality Control

The ALD manager and the ALD FQAO are responsible for monitoring and documenting procedure performance, including the analysis of QC samples, and are responsible for implementing corrective actions as described in the ACL implementation plan when procedure performance is not acceptable. The ACL implementation plan and referenced procedures describe the actions to ensure the daily quality of

analytical data for total VOC analysis, specify acceptance criteria for TWCP QC samples, and specify corrective action measures to be taken when these criteria are not satisfied. ACL personnel operate a formal QC program and maintain records to document the quality of the data generated. They implement all QC practices established by the QAPP, SW-846, and the Methods Manual, including QC sample requirements summarized in Table 13-2.

Table 13-2. Summary of laboratory quality control samples and frequencies for total volatile organic compound analyses.

QC sample	Minimum frequency	Acceptance criteria	Corrective action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 13-1 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	Analyte concentrations <3 * MDLs	Specified in the ACL implementation plan[a]
Matrix spikes	One (1) per analytical batch	Meet Table 13-1 %Rs	Specified in the ACL implementation plan[b,a]
Matrix spike duplicates	One (1) per analytical batch	Meet Table 13-1 RPDs and %Rs	Specified in the ACL implementation plan[b,a]
Laboratory control samples	One (1) per analytical batch	80 - 120 %R	Specified in the ACL implementation plan[a]
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples for a given matrix ±3 standard deviations[c]	Specified in the ACL implementation plan[b,a]
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

MDL = Method detection limit
QAO = Quality assurance objective
PDP = Performance Demonstration Program
%R = Percent recovery
RPD = Relative percent difference

a. A nonconformance report per Section 2.1.2 of this QAPjP is required when quality control samples associated with final reported data do not meet Table 14-2 acceptance criteria (See footnote b. exception).

b. Matrix spikes, matrix spike duplicates, and surrogate compounds that do not meet acceptance criteria due to matrix interference effects shall be flagged as "Z" and a nonconformance report is not required.

c. ACL calculates surrogate compound acceptance criteria as 3s of mean recovery per matrix when an accurate estimate of the mean and standard deviation can be derived.

ACL personnel demonstrate acceptable performance prior to the analysis of any solid samples through the analysis of method performance samples. They ensure method performance samples contain all of the analytes listed in Table 13-1 at concentrations appropriate to verify all QAOs are met. Initially, they analyze seven method performance samples to demonstrate acceptable precision and accuracy and to determine MDLs for all analytes. They demonstrate acceptable method performance semiannually by analyzing four method performance samples.

ACL personnel analyze samples in analytical batches (see Definitions section). Specific QC samples for each analytical batch include a laboratory blank, a matrix spike, a matrix spike duplicate, a laboratory control sample, and surrogate compounds. Laboratory procedures describe the preparation and analysis of QC samples.

ACL personnel choose surrogate compounds based on guidance provided in the Methods Manual and add these compounds to each field sample and laboratory QC sample. They analyze matrix spike duplicates in place of laboratory duplicates. They ensure that matrix spikes and matrix spike duplicates contain at least five of the VOCs listed in Table 13-1; spiking amounts are specified in analytical procedures and are generally assigned such that the matrix spike sample concentrations are greater than or equal to midrange on the calibration.

ACL personnel demonstrate ongoing laboratory performance through the analysis of laboratory control samples and meet the acceptance criteria listed in Table 13-2. For GC/MS analysis, they prepare laboratory control samples, containing at least ten of the analytes listed in Table 13-1 at a mid-range concentration from standards independent of those used for instrument calibration. For GC/FID analysis, they prepare laboratory control samples, containing all of the nonhalogenated VOC analytes listed in Table 13-1 at a mid-range concentration from standards independent of those used for instrument calibration. Laboratory control sample concentrations are specified in analytical procedures. Laboratory control samples are not carried through the same preparation procedures as samples because the solid extraction procedures are incompatible with the noninterfering matrix liquid laboratory control sample. ACL personnel also demonstrate acceptable laboratory performance biannually by analysis of PDP blind audit samples.

When QC sample acceptance criteria are not met, the ALD manager and the ALD FQAO initiate corrective action as described in the ACL implementation plan. They ensure data are flagged as appropriate or an NCR is initiated if final reported QC sample results do not meet the acceptance criteria. If matrix spikes, matrix spike duplicates, and surrogate compounds do not meet acceptance criteria due to matrix interference effects, the ALD manager or FQAO ensures the data are flagged as "Z."

13.4 Instrument Testing, Inspection, and Maintenance Requirements

ACL personnel use ion trap GC/MS instruments instead of quadrupole instruments. Although more sensitive, the ion trap GC/MSs meet all performance requirements specified in the Methods Manual and SW-846. All other ACL equipment and materials meet the Methods Manual and SW-846 requirements. They equip gas chromatographs with columns selected from among those recommended by the methods (based on availability). They operate GC/MS systems in the full scan mode and report nontarget compounds as TICs with a higher uncertainty than target analyte compounds.

ACL personnel meet GC/MS instrument performance criteria prior to the analysis of any standards or samples by meeting the BFB criteria specified in the appropriate Methods Manual procedure. They check the BFB criteria at the beginning of each 12 hours of operation by analyzing 50 ng of BFB.

13.5 Instrument Calibration and Frequency

ACL personnel ensure all analytical instruments are calibrated before use and verify calibration at routine intervals in compliance with Section 13.5 of the QAPP and Table 13-3. ACL personnel ensure the initial and continuing calibration requirements listed in Table 13-3 are met and maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency. ACL calibration and records management processes are described and identified in the ACL implementation plan and referenced procedures.

Table 13-3. Summary of calibration requirements for total volatile organic compounds analyses.

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
GC/MS	BFB Tune	Every 12 hours	Abundance criteria for all key ions are met (see SW-846 Method 8260A)
	5-pt initial calibration (5 standards)	Initially, and as needed	Response factor %RSD for CCCs ≤30; response factor for SPCCs ≥0.30[a]; average relative response factor is used if %RSD ≤15; regression equation is generated if %RSD >15
	Continuing calibration	Every 12 hours	Response factor or conc. %D for CCCs ≤20; response factor for SPCCs ≥0.30[a]; RT for internal standards must be ±30 seconds from last daily calibration check; internal standard area count must be >50 or <200% of the area counts from the last daily calibration check.
GC/FID	3-pt initial calibration (3 standards)	Initially, and as needed	Correlation coefficient ≥0.93 (calibration curves) or %RSD for response factors <35 for all analytes
	Continuing calibration	Every 12 hours	Response factor or measured concentration %D for all analytes ≤15 of initial calibration; RT □3 standard deviations from initial calibration

BFB = 4-Bromofluorobenzene
CCC = Calibration check compounds
%D = Percent difference
%RSD = Percent relative standard deviation
RT = Retention time
SPCC = System performance check compounds

a. Bromoform ≥0.10.

ACL personnel satisfy GC/MS instrument performance criteria, then use at least five standards to define the calibration range of the instrument for all target analytes and set the concentration of one

standard less than the PRQLs listed in Table 13-1. They use average relative response factors for each analyte if the %RSD is less than or equal to 15 and use a linear or quadratic regression equation if the %RSD is greater than 15. They use a midpoint calibration standard (the continuing calibration standard) to verify the initial GC/MS calibration curve at the beginning of every 12 hours of operation after satisfying the instrument performance criteria using 50 ng of BFB. They ensure the continuing calibration standard meets all of the daily calibration criteria, SPCCs, CCCs, internal standard area count criteria, and RTs, as specified in Table 13-3. They choose SPCCs and CCCs common to Table 13-1 and appropriate Methods Manual procedures. They generate a new five-point calibration curve if the continuing calibration standard does not satisfy the Table 13-3 acceptance criteria and do not proceed with sample analysis until the GC/MS system has satisfied the appropriate daily calibration criteria.

ACL personnel use at least three standards to define the calibration range of the GC/FID system for the appropriate nonhalogenated VOC analytes listed in Table 13-1. They set the concentration of one standard less than the PRQLs specified in Table 13-1. They generate a linear regression plot of peak area versus concentration or use average response factors to quantitate analytes. They verify the initial GC/FID calibration curve with a midpoint calibration standard (continuing calibration standard) at the beginning of each 12 hours of operation. They ensure the continuing calibration standard meets the daily calibration criteria, as specified in Table 13-3. They prepare a new calibration curve or response factor for any analyte in the continuing calibration standard that does not satisfy the Table 13-3 acceptance criteria and do not proceed with sample analysis until the GC/FID system has satisfied the appropriate daily calibration criteria.

13.6 Data Management

The ACL implementation plan describes data reduction, validation, and reporting processes. Laboratory procedures include or reference example forms to be used to record and report data. ACL personnel reduce raw data to reportable results in compliance with the Methods Manual procedures. ACL personnel review, validate, and verify data as described in Section 3.1.1 of this QAPjP.

ACL personnel quantify analytes by GC/MS using average relative response factors (or regressions as specified in Section 13.5) and do not blank-correct these results. They quantify analytes by GC/FID using a linear regression equation or response factors, and do not blank-correct these results. They report results of blanks run in association with samples separately. They report all VOC results in mg/kg on a weight/wet-weight basis, limited to two significant figures.

ACL personnel report nontarget compounds as TICs with a higher uncertainty than target analyte concentrations. They perform appropriate search routines of the latest NIST mass spectral library on the 20 TICs with the greatest area counts and with total ion current peaks greater than 10% of the nearest (RT) internal standard. The SPM adds the positively identified TICs listed in 40 CFR Part 264, Appendix IX, to the target analyte list if they are detected in 25% of all samples from a given IDC. ACL personnel assume a relative response factor equal to one, using the nearest internal standard, when calculating concentration for TICs.

ACL personnel submit analytical batch data reports to the SDCO and maintain analytical records as specified in Section 3.4.1 of this QAPjP. These records are subject to assessment by SPO representatives on a regular basis as discussed in Section 3.1.2. Analytical batch data report requirements include the following:

- Laboratory name, analytical batch number, sample numbers included in that analytical batch, a cross reference to field sample numbers, and the signature releases of laboratory personnel specified in Section 3.1.1 of the QAPP

- Table of Contents

- COC form showing the date and time of sample transfer and names of individuals handling the samples from the time of sampling through receipt at the laboratory

- Data review checklists for each analytical batch verifying that data generation level review, validation, and verification have taken place.

- QC sample results (e.g., matrix spikes, matrix spike duplicates, laboratory control samples) if not included in data review checklists.

- A separate analytical report sheet titled, "Total VOCs Analysis Data Sheet," for each sample in the analytical batch, including laboratory name, program name, analytical batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date analyzed, method number, listing of program analytes, and analytical results in mg/kg wet weight basis. The following data qualifying flags are used:

  - B analyte detected in blank
  - E Analyte exceeds the calibration curve
  - H Holding time exceeded
  - J Analyte is less than PRQL but greater than or equal to MDL
  - U Analyte was undetected (report MDL)
  - D Analyte was quantitated from a secondary dilution
  - Z One or more QC samples do not meet acceptance criteria

- Nonconformance reports, if applicable

ACL personnel maintain the following records in their files, documented and retrievable by analytical batch numbers and data report numbers:

- Original COC records

- All raw data, including original instrument readouts and/or bench reports, calculation records, and laboratory QC sample results. Matrix spike/matrix spike duplicate results are recorded along with the spiked amounts, and the RPD between the two results are calculated. Laboratory control sample results are entered with the accepted value and the %R.

- Instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the laboratory name, initial and continuing calibration verification source, method identification, and calibration date and time.

- QC result summary, which includes true and found values for all QC samples plus associated result calculations. At a minimum, the QC data include blanks, matrix spikes, matrix spike duplicates, laboratory control samples, initial calibration data, initial and continuing calibration verifications, and all other method-specific QC listed in Section 13. The QC result summary includes the laboratory name, the analytical batch number (if applicable), and method names.

13.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 13-4 lists the major technical implementing procedures pertinent to this section of the QAPjP. The ACL implementation plan identifies additional facility procedures.

Table 13-4. Section 13 implementing procedures.

Document Number	Title
ACMM 9260	*Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS): Capillary Column Technique*
ACMM 9261	*Determination of Total Volatile Organic Compounds in Homogeneous Solids and Soil/Gravel by Gas Chromatography/Mass Spectrometry (GC/MS)*
ACMM 9441	*Determination of Nonhalogenated Volatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID)*
ACMM 9501	*Sample Preparation of TRU Waste Characterization Samples for Organic Analysis.*

14. TOTAL SEMIVOLATILE ORGANIC COMPOUND ANALYSIS

ACL personnel receive solid samples resulting from the ANL-W homogeneous solids and soil/gravel sampling operations described in Section 8 of this QAPjP. ACL personnel analyze the samples for total SVOCs in accordance with Section 14.0 of the QAPP as described in the ACL implementation plan and referenced procedures.

14.1 Quality Assurance Objectives

Table 14-1 lists the QAOs for total SVOC analysis. The following text defines key data quality indicators for ACL measurements and Section 3.2 of this QAPjP presents the methods to assess compliance with these indicators. To demonstrate compliance with the QAOs, ACL personnel:

- Measure precision by analyzing matrix spike duplicates, replicate analyses of laboratory control samples, and PDP blind audit samples and calculate %RSD or RPD based on the results.
- Measure accuracy by analyzing laboratory control samples, matrix spikes, surrogate compounds, and PDP blind audit samples and calculate %R based on the results.
- Determine MDLs, expressed in mg/kg, and ensure the MDLs are less than or equal to those listed in Table 14-1.
- Demonstrate the ability to quantitate at or below the PRQL given in Table 14-1 by setting the concentration of at least one calibration standard below the PRQL.
- Measure adherence to the 90% completeness criterion in Table 14-1 by calculating the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis.
- Achieve comparability by using standardized procedures and traceable standards and participating in the PDP for RCRA constituent analysis of solidified wastes in compliance with the QAPP requirements.

ANL-W personnel assure representativeness through the use of standardized and approved sampling methods described in Section 8 of this QAPjP and the Methods Manual.

The ALD manager and the ALD FQAO are responsible for monitoring the results from these measurements and determining whether precision, accuracy, and completeness requirements are met. They evaluate ACL performance and decide whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

14.2 Methods Requirements

ACL personnel use ultrasonic extraction and ion trap GC/MS to prepare and analyze homogeneous solids and soil/gravel samples for all total SVOC analytes except polychlorinated biphenyls (PCBs). The ultrasonic extraction method is based on SW-846 Method 3550A and the GC/MS analysis method is based on Methods Manual Procedure 430.6. GC/MS SVOC analysis is described in Section 14.2 of the ACL implementation plan.

Table 14-1. SVOC target analyte list and quality assurance objectives.

Compound	CAS Number	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	MDL (mg/kg)	PRQL (mg/kg)	Completeness (percent)
Cresols	1319-77-3	≤50	60-150	5	40	90
1,4-Dichlorobenzene[b]	106-46-7	≤86	20-124	5	40	90
ortho-Dichlorobenzene[b]	95-50-1	≤64	32-129	5	40	90
2,4-Dinitrophenol	51-28-5	≤119	D-172[c]	5	40	90
2,4-Dinitrotoluene	121-14-2	≤46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤319	D-152[c]	0.3	2.6	90
Hexachloroethane	67-72-1	≤44	40-113	5	40	90
Nitrobenzene	98-95-3	≤72	35-180	5	40	90
Pentachlorophenol	87-86-5	≤128	14-176	5	40	90
Pyridine[b]	110-86-1	≤50	60-150	5	40	90
Polychlorinated Biphenyls (PCBs)						
Aroclor 1016[d]	12674-11-2	≤33	50-114	5	40	90
Aroclor 1221[d]	11104-28	≤110	15-178	5	40	90
Aroclor 1232[d]	11141-16-5	≤128	10-215	5	40	90
Aroclor 1242[d]	53469-21-9	≤49	39-150	5	40	90
Aroclor 1248[d]	12672-29-6	≤55	38-158	5	40	90
Aroclor 1254[d]	11097-69-1	≤62	29-131	5	40	90
Aroclor 1260[d]	11096-82-5	≤56	8-127	5	40	90

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

MDL = Method detection limit (maximum permissible value)

PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for nitrobenzene assuming a 100 g sample, 2 L of extraction fluid, and 100 % analyte extraction

a. Criteria apply to PRQL concentrations

b. Can also be analyzed as a volatile organic compound

c. Detected; result must be greater than zero

d. PCBs; required only for matrix parameter category S3220 (organic sludges)

ACL personnel use ultrasonic extraction with sulfuric acid/permanganate cleanup and gas chromatography with electron capture detection (ECD) to prepare and analyze homogeneous solids and soil/gravel samples for PCBs. The extraction and cleanup methods are based on SW-846 Method 3550A and Method 3665A, respectively. The GC/ECD analysis method is based on Methods Manual Procedure 440.3. PCB analysis is described in Section 14.2 of the ACL implementation plan.

14.2.1 Standards

ACL personnel purchase certified primary standards from the best available source. Commercially-purchased primary standards are certified by the manufacturer and their concentrations are traceable to the NIST, EPA, or other nationally-recognized standards. ACL personnel prepare standards in accordance with SW-846 methods and Methods Manual Procedure cited in Section 14.2 of this QAPjP. The ACL implementation plan describes and identifies procedures used to prepare standards.

14.2.2 Qualitative and Quantitative Analysis

ACL personnel qualitatively identify analytes using GC/MS by ensuring the analytes elute within an RRT window of plus or minus 0.06 RRT units of the RRT of the continuing calibration standard and have a mass spectrum that corresponds to the standard analyte mass spectrum. They calculate RRT windows from the individual RTs in the associated continuing calibration standard within the same 12 hours as the sample. ACL personnel determine RT windows per the QAPP and the ACL implementation describes RT window determination.

ACL personnel qualitatively identify PCB target analytes based on pattern recognition and sample component elution within established RT windows. Although RT windows are used as a tool for qualitative identification, ACL personnel rely primarily on pattern recognition for PCB Aroclor identification. RT windows are calculated as fixed percentages of mean ICAL RTs, as described in the ACL implementation plan.

ACL personnel use external standard quantitation, using a minimum of three standards, for GC/ECD analysis of PCBs. Analyte quantitation is based on peak area response measured under three to five major peaks for each Aroclor. All analytes are quantitated within the calibration range of GC.

ACL personnel use internal standards for quantitating GC/MS target analytes. They meet the criteria for SPCCs and CCCs and calculate response factors and %RSD for all analytes. They meet all daily DFTPP tune and calibration criteria for SPCCs, CCCs, internal standard responses, and RTs. They quantitate each target analyte based on the integrated abundance from the EICP of the primary ion. They quantitate all target analytes within the calibration range of the GC/MS instrument.

Refer to the ACL implementation plan for a more detailed discussion of qualitative and quantitative analysis.

14.3 Quality Control

The ALD manager and the ALD FQAO are responsible for monitoring and documenting procedure performance, including the analysis of QC samples, and are responsible for implementing corrective actions as described in the ACL implementation plan when procedure performance is not acceptable. The ACL implementation plan and referenced procedures describe the actions to ensure the daily quality of

analytical data for total SVOC analysis, specify acceptance criteria for TWCP QC samples, and specify corrective action measures to be taken when these criteria are not satisfied. ACL personnel operate a formal QC program and maintain records to document the quality of the data generated. They implement all QC practices established by the QAPP, SW-846, and Methods Manual, including QC sample requirements summarized in Table 14-2.

ACL personnel demonstrate acceptable performance prior to the analysis of any solid samples through the analysis of method performance samples as described in the ACL implementation plan. They ensure method performance samples contain all analytes listed in Table 14-1 at concentrations appropriate to verify all QAOs are met. Initially, they analyze seven method performance samples to demonstrate acceptable precision and accuracy and to determine MDLs for all analytes. They demonstrate acceptable procedure performance semiannually by analyzing four method performance samples.

ACL personnel analyze samples in analytical batches (see Definitions section). Specific QC samples for each analytical batch include a laboratory blank, a matrix spike, a matrix spike duplicate, a laboratory control sample, and surrogate compounds. Laboratory procedures detail the preparation and analysis of QC samples.

ACL personnel choose surrogate compounds based on guidance provided in the Methods Manual and add these compounds to each field sample and laboratory QC sample. They analyze matrix spike duplicates in place of laboratory duplicates. They ensure matrix spikes and matrix spike duplicates contain at least three of the target analytes listed in Table 14-1 for GC/MS SVOC analysis and one Aroclor from Table 14-1 for PCB analysis. Spiking concentrations are specified in the analytical procedure and are generally assigned such that the matrix spike sample concentrations are greater than or equal to midrange on the calibration.

ACL personnel demonstrate ongoing laboratory performance through the analysis of laboratory control samples to meet the acceptance criteria listed in Table 14-2. They prepare laboratory control samples (LCSs) for GC/MS analysis in methylene chloride to contain, at a minimum, 1,4-dichlorobenzene, 2,4-dinitrotoluene, hexachloroethane, and nitrobenzene. They prepare LCSs for PCBs in hexane or isooctane to contain the most representative Aroclor (usually Aroclor-1254 or Aroclor-1260) from Table 14-1. ACL personnel prepare LCSs at mid-range concentrations from standards independent of those used for instrument calibration. Laboratory control sample concentrations are specified in analytical procedures. Laboratory control samples are not carried through the same preparation procedures as samples because the solid extraction procedures are incompatible with the noninterfering matrix liquid laboratory control sample. ACL personnel also demonstrate acceptable laboratory performance biannually by analysis of PDP blind audit samples.

When QC sample acceptance criteria are not met, the ALD manager and the ALD FQAO initiate corrective action as described in the ACL implementation plan. They ensure data are flagged as appropriate or an NCR is initiated if final reported QC sample results do not meet the acceptance criteria. If matrix spikes, matrix spike duplicates, and surrogate compounds do not meet acceptance criteria due to matrix interference effects, the ALD manager or the FQAO ensures the data are flagged as "Z."

Table 14-2. Summary of laboratory quality control samples and frequencies for total SVOC analyses.

QC Sample	Minimum frequency	Acceptance criteria	Corrective action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 14-1 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	Analyte concentrations <3 * MDLs	Specified in the ACL implementation plan[a]
Matrix spikes	One (1) per analytical batch	Meet Table 14-1 %Rs	Specified in the ACL implementation plan[b,a]
Matrix spike duplicates	One (1) per analytical batch	Meet Table 14-1 RPDs and %Rs	Specified in the ACL implementation plan[b,a]
Laboratory control samples	One (1) per analytical batch	80-120 %Rs	Specified in the ACL implementation plan[a]
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples from a given matrix ±3 standard deviations[c]	Specified in the ACL implementation plan[b,a]
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

MDL = Method detection limit

QAO = Quality assurance objective

PDP = Performance Demonstration Program

%R = Percent recovery

RPD = Relative percent difference

a. A nonconformance report per Section 2.1.2 of this QAPjP is required when quality control samples associated with final reported sample data do not meet Table 14-2 acceptance criteria.

b. Matrix spikes, matrix spike duplicates, and surrogate compounds that do not meet acceptance criteria due to matrix interference effects shall be flagged as "Z" and a nonconformance report is not required.

c. ACL personnel calculate surrogate compound acceptance criteria as ± 3s of mean recovery per matrix when an accurate estimate of the mean and standard deviation can be derived.

14.4 Instrument Testing, Inspection, and Maintenance Requirements

ACL personnel use ion trap GC/MS instruments instead of quadrupole instruments. Although more sensitive, the ion trap GC/MSs meet all performance requirements specified in the Methods Manual and SW-846. All other equipment and materials meet the Methods Manual and SW-846 requirements. They equip GC/MSs with columns selected from among those recommended by the methods (based on availability). They operate GC/MS systems in the full scan mode and report nontarget compounds as TICs with a higher uncertainty than target analyte compounds.

ACL personnel meet GC/MS instrument performance criteria prior to the analysis of any standards or samples by meeting the decafluorotriphenylphosphine (DFTPP) criteria specified in the appropriate Methods Manual procedure. Due to ion trap GC/MS sensitivity, they check the DFTPP criteria at the beginning of each 12 hours of operation by analyzing 5 ng of DFTPP instead of 50 ng.

ACL personnel use G/ECD systems equipped with a single analytical column selected from those recommended by SW-846 Method 8081 and Methods Manual Procedure 440.3. They prime the system daily with a solution containing an Aroclor at a concentration approximately twenty times the midpoint standard order to deactivate the GC column.

14.5 Instrument Calibration and Frequency

ACL personnel ensure all analytical instruments are calibrated before use and verify calibration at routine intervals in compliance with Section 14.5 of the QAPP, as described in the ACL implementation plan. ACL personnel ensure all the initial and continuing calibration requirements listed in Table 14-3 are met and maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency. ACL calibration procedures and records are described and identified in the ACL implementation plan.

ACL personnel satisfy GC/MS instrument performance criteria, then use at least five standards to define the calibration range of the instrument for all target analytes and set the concentration of one standard less than the PRQLs listed in Table 14-1. They use average relative response factors for each analyte if the %RSD is less than or equal to 15 and use a linear or quadratic regression equation if the %RSD is greater than 15. They use a midpoint calibration standard (continuing calibration standard) to verify the initial GC/MS calibration curve at the beginning of every 12 hours of operation after satisfying the instrument performance criteria using 5 ng of DFTPP. They ensure the continuing calibration standard meets all daily calibration criteria for %D, SPCCs, internal standard area count criteria, and RTs, as specified in Table 14-3. They choose SPCCs and CCCs common to Table 14-1 and the appropriate Methods Manual procedures. They generate a new five-point calibration curve if the continuing calibration standard does not satisfy the Table 14-3 acceptance criteria and do not proceed with sample analysis until the GC/MS system has satisfied the appropriate daily calibration criteria.

Table 14-3. Summary of calibration requirements for total SVOC analyses.

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
GC/MS	DFTPP Tune	Every 12 hours	Abundance criteria for all key ions are met (see SW-846 Method 8270B)
	5-pt initial calibration (5 standards)	Initially and as needed	Response factor %RSD for CCCs ≤30; response factor for SPCCs ≥0.05; average relative response factor used if %RSD ≤15; regression equation generated if %RSD >15
	Continuing calibration	Every 12 hours	%D ≤20 for all analytes; response factor for SPCCs ≥0.05; RT for internal standards must be □30 seconds from last daily calibration check; internal standards area count must be >50 or <200% of the area count from daily calibration check.
GC/ECD	3-pt initial calibration (3 standards)	Initially and as needed	Response factor or measured %D for all analytes ≤ 20
	Continuing calibration	Every 12 hours as needed	%D ≤ 15 for all analytes compared to ICAL

CCC	=	Calibration check compounds
%D	=	Percent difference
DFTPP	=	Decafluorotriphenylphosphine
%RSD	=	Percent relative standard deviation
RT	=	Retention time
SPCC	=	System performance check compounds

ACL personnel prime the GC/ECD system, then use at least three standards to define the calibration range of the instrument for each target analyte and set the concentration of at least one standard less than the PRGLs listed in Table 14-1. In accordance with the QAPP and SW-846 Method 8081, they use linear regressions plotting peak area against concentration to quantitate each analyte. They ensure that calibration criteria in Table 14-3 are met before quantitating any sample. ACL personnel ensure that the continuing calibration standard meets Table 14-3 criteria for those Aroclors detected in samples within that 12-hour shift, and that the RTs of each analyte are within the determined RT windows. They perform corrective actions, including generating new initial calibrations, if continuing calibration criteria are not met, and do not continue with sample analysis until the GC/ECD system has satisfied the daily calibration criteria.

14.6 Data Management

The ACL implementation plan describes data reduction, validation, and reporting processes. Laboratory procedures include or reference example forms to be used to record and report data. ACL personnel reduce raw data to reportable results in compliance with the Methods Manual procedures. ACL personnel review, validate, and verify data as described in Section 3.1.1 of this QAPjP.

ACL personnel quantify GC/MS analytes using average relative response factors (or regressions as specified in Section 14.5) and quantify GC/ECD analytes (i.e., PCBs) using linear regressions. They report all total SVOC results (GC/MS and GC/ECD) in mg/kg on a weight/wet weight basis to two significant figures. They do not blank-correct results and report separately the results of blanks run in association with samples.

ACL personnel report nontarget compounds as TICs with a higher uncertainty than target analyte concentrations. They perform appropriate search routines of the latest NIST mass spectral library on the 20 TICs with the greatest area counts and with total ion current peaks greater than 10% of the nearest (RT) internal standard. The SPM adds the positively identified TICs listed in 40 CFR Part 264, Appendix IX, to the target analyte list if they are detected in 25% of all samples from a given IDC. ACL personnel assume a relative response factor equal to one, using the nearest internal standard, when calculating concentrations for TICs.

ACL personnel submit analytical batch data reports to the SDCO and maintain analytical records as specified in Section 3.4.1 of this QAPjP. These records are subject to assessment by representatives from the SPO on a regular basis, as discussed in Section 3.1.2. Analytical batch data report requirements include the following:

- Laboratory name, analytical batch number, sample numbers included in that analytical batch, a cross reference to field sample numbers, and the signature releases of laboratory personnel specified in Section 3.1.1 of the QAPP
- Table of Contents
- COC form showing the date and time of sample transfer and names of individuals handling the samples from the time of sampling through receipt at the laboratory
- Data review checklists for each analytical batch verifying that data generation level review, validation, and verification have taken place

- QC sample results (e.g., matrix spikes, matrix spike duplicates, laboratory control samples) if not included in data review checklists

- A separate analytical report sheet titled, "Total SVOCs Analysis Data Sheet," and/or "PCB Analysis Data Sheet,"for each sample in the analytical batch, including laboratory name, program name, analytical batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date analyzed, method number, listing of program analytes, and analytical results in mg/kg wet weight basis. The following data qualifying flags are used:

  - B analyte detected in blank
  - E Analyte exceeds the calibration curve
  - H Holding time exceeded
  - J Analyte is less than PRQL but greater than or equal to MDL
  - U Analyte was undetected (report MDL)
  - D Analyte was quantitated from a secondary dilution
  - Z One or more QC samples do not meet acceptance criteria

- Nonconformance reports, if applicable

ACL personnel maintain the following records in their files, documented and retrievable by analytical batch numbers and data report numbers:

- Original COC records

- All raw data, including original instrument readouts and/or bench reports, calculation records, and laboratory QC sample results. Matrix spike and matrix spike duplicate results are recorded along with the spiked amounts, and the RPD between the two results are calculated. Laboratory control sample results are entered with the accepted value and the %R.

- Instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the laboratory name, initial and continuing calibration verification source, method identification, and calibration date and time.

- QC result summary, which includes true and found values for all QC samples plus associated result calculations. At a minimum, the QC data include blanks, matrix spikes, matrix spike duplicates, laboratory control samples, initial calibration data, initial and continuing calibration verifications, and all other method-specific QC listed in Section 14. The QC result summary includes the laboratory name, the analytical batch number (if applicable), and method names.

14.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 14-4 lists the major technical implementing procedures pertinent to this section of the QAPjP. The ACL implementation plan identifies additional facility procedures.

Table 14-4. Section 14 implementing procedures.

Document Number	Title
ACMM 9271	*Determination of Semivolatile Organic Compounds in TRU Waste Characterization Samples*
ACMM 9501	*Sample Preparation of TRU Waste Characterization Samples for Organic Analysis*
ACMM 9081	*Determination of PCBs in Radioactive Organic Sludge by Gas Chromatography/Electron Capture Detection*

15. TOTAL METAL ANALYSIS

ACL personnel receive solid samples resulting from the ANL-W homogeneous solids and soil/gravel sampling operations described in Section 8 of this QAPjP. ACL personnel analyze the samples for total metals in accordance with Section 15.0 of the QAPP, as described in the ACL implementation plan and referenced procedures.

15.1 Quality Assurance Objectives

Table 15-1 lists the QAOs for total metals analysis. The following text defines key data quality indicators for ACL measurements and Section 3.2 of this QAPjP presents the methods to assess compliance with these indicators. To demonstrate compliance with the QAOs, ACL personnel:

- Measure precision by analyzing laboratory matrix spike duplicates, replicate analyses of laboratory control samples, and PDP blind audit samples and calculate %RSD or RPD based on the results.
- Measure accuracy by analyzing laboratory matrix spikes, PDP blind audit samples, and laboratory control samples, and calculate %R based on the results.
- Determine IDLs, expressed in µg/L, and ensure IDLs are less than or equal to the program-required detection limits (PRDLs) listed in Table 15-1.
- Demonstrate the ability to quantitate at or below the PRQLs given in Table 15-1 by setting the concentration of at least one calibration standard below the solution-equivalent of the PRQL.
- Measure adherence to the 90% completeness criterion by calculating the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis.
- Achieve comparability by using standardized procedures and traceable standards and participating in the PDP for RCRA constituent analysis of solidified wastes in compliance with the QAPP requirements.

ANL-W personnel assure representativeness through the use of standardized and approved sampling methods described in Section 8 of this QAPjP and the Methods Manual.

The ALD manager and the ALD FQAO are responsible for monitoring the results from these measurements and determining whether precision, accuracy, and completeness requirements are met. They evaluate ACL performance and decide whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

15.2 Methods Requirements

ACL personnel analyze homogeneous solids and soil/gravel samples for total metals using acid digestion followed by spectrometric analyses as described in Section 15.2 of the ACL implementation plan.

The acid digestion method used for all analytes except mercury is a microwave-assisted hot acid procedure based on Procedure 610.1 in the Methods Manual. They analyze samples for antimony, arsenic, barium, beryllium, cadmium, chromium, lead, nickel, selenium, silver, thallium, vanadium, and zinc by inductively coupled

Table 15-1. Total metals target analyte list and quality assurance objectives.

Analyte	CAS Number	Precision (%RSD or RPD)[a]	Accuracy (%R)[b]	PRDL[c] (µg/L)	PRQL (mg/kg)	Completeness (%)
Antimony	7440-36-0	≤30	80-120	100	100	90
Arsenic	7440-38-2	≤30	80-120	100	100	90
Barium	7440-39-3	≤30	80-120	2000	2000	90
Beryllium	7440-41-7	≤30	80-120	100	100	90
Cadmium	7440-43-9	≤30	80-120	20	20	90
Chromium	7440-47-3	≤30	80-120	100	100	90
Lead	7439-92-1	≤30	80-120	100	100	90
Mercury	7439-97-6	≤30	80-120	4.0	4.0	90
Nickel	7440-02-0	≤30	80-120	100	100	90
Selenium	7782-49-2	≤30	80-120	20	20	90
Silver	7440-22-4	≤30	80-120	100	100	90
Thallium	7440-28-0	≤30	80-120	100	100	90
Vanadium	7440-62-2	≤30	80-120	100	100	90
Zinc	7440-66-6	≤30	80-120	100	100	90

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

PRDL = Program required detection limit (i.e., maximum permissible value for IDL)

PRQL = Program required quantitation limit

a. ≤30% control limits apply when sample and duplicate concentrations are ≥10 x IDL for ICP-AES and AA techniques. If less than these limits, the absolute difference between the two values shall be less than or equal to the PRDL.

b. Applies to laboratory control samples. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

c. PRDL set such that it is a factor of 10 below the PRQL for 100% solid samples, assuming a 100X dilution during digestion.

plasma-atomic emission spectroscopy (ICP-AES) based on Procedure 640.1 in the Methods Manual. They prepare and analyze samples for mercury by cold vapor atomic fluorescence spectroscopy (CVAF) based on Procedure 650.3 in the Methods Manual.

15.2.1 Standards

ACL personnel purchase stock standard solutions or prepare these solutions from ultra-high-purity grade chemicals or metals (99.99 to 99.999% pure). All stock standard solutions have a known valid relationship to a nationally recognized standard material (e.g., NIST). They maintain a certificate of analysis on file from the manufacturer documenting traceability if commercial stock solutions are used. They label and track commercial stock solutions to ensure they are not used beyond their manufacturer-specified shelf life.

ACL personnel prepare working calibration and QC standards by diluting stock standard solutions using volumetric glassware and calibrated pipettors. They prepare working calibration standards, blanks, and QC standards using the same types and concentrations of acids as used in prepared samples. They verify working calibration standards with each use through comparison with a freshly prepared (daily) initial calibration verification standard from an independent source.

ACL personnel prepare working calibration and QC standards for ICP-AES at least weekly. They prepare working calibration and QC standards for CVAF daily in accordance with the Methods Manual. Laboratory procedures identified in the ACL implementation plan describe metals standards preparation.

15.2.2 Quantitative Analysis

All analytical instruments used to quantitate metal analytes meet the requirements of the Methods Manual and SW-846. Operating requirements are described in the ACL implementation plan and referenced procedures. ACL personnel establish and document instrument settings for each analyte on each applicable instrument. They quantitate all analytes within the calibration range of the analytical instruments. They dilute samples with concentrations greater than the calibration range of the instrument and use two integrations to quantitate all analytes (with the exception of CVAF) and report the average. They use a flow-through system with a single 60- second integration for CVAF quantitation.

Analytical procedures specify reagent purity, instrument operating conditions, background correction procedures, and interference detection and evaluation if applicable. For ICP-AES, ACL personnel determine interelement interference correction factors annually and apply them manually after data generation for samples having interfering element concentrations sufficient to cause an interference effect of a magnitude exceeding 5 times the instrument IDL. They use the method of standard additions when appropriate.

15.3 Quality Control

The ALD manager and the ALD FQAO are responsible for monitoring and documenting procedure performance, including the analysis of QC samples and are responsible for implementing corrective actions as described in the ACL implementation plan when procedure performance is not acceptable. The ACL implementation plan and referenced procedures describe the actions to ensure the daily quality of analytical data for total metals analysis, specify acceptance criteria for TWCP QC samples, and specify

corrective action measures to be taken when these criteria are not satisfied. ACL personnel operate a formal QC program and maintain records to document the quality of the data generated. They implement all QC practices established by SW-846 and the Methods Manual, including QC sample requirements listed in Section 15.3 of the QAPP, and summarized in Table 15-2.

ACL personnel demonstrate acceptable performance prior to the analysis of any solid samples through the analysis of method performance samples. They ensure method performance samples contain all analytes listed in Table 15-1 at concentrations appropriate to verify all QAOs are met. Initially, they analyze a minimum of seven method performance samples to demonstrate acceptable precision and accuracy and determine IDLs for all analytes. They demonstrate acceptable procedure performance semiannually by analyzing at least four method performance samples.

ACL personnel analyze samples in analytical batches (see the Definitions section). Specific QC samples for each analytical batch include a laboratory blank, a matrix spike, a matrix spike duplicate, and a laboratory control sample. Matrix spike duplicates are analyzed in lieu of laboratory duplicates.

ACL personnel digest and analyze laboratory blanks according to the same procedures used for solid samples. They prepare matrix spikes and matrix spike duplicates to contain the metal(s) being analyzed. Spiking levels are specified in ACL procedures and are generally assigned such that the matrix spike sample concentrations are greater than or equal to midrange on the calibration. They demonstrate ongoing laboratory performance through the analysis of laboratory control samples to meet the acceptance criteria listed in Table 15-2. They prepare solid laboratory control samples to contain the metal(s) being analyzed and quantitate these samples within the calibration range of the instruments. They use a solid matrix laboratory control sample that is independent of calibration standards and matches the expected sample matrix as closely as possible. They employ all the sample preparation procedures performed on field samples for laboratory control samples. They also demonstrate acceptable laboratory performance biannually by analyzing PDP blind audit samples.

When QC sample acceptance criteria are not met, the ALD manager and the FQAO initiate corrective action as described in the ACL implementation plan and referenced procedures. They ensure data are flagged as appropriate or an NCR is initiated if final reported QC sample results do not meet the acceptance criteria. If matrix spikes and matrix spike duplicates do not meet acceptance criteria due to matrix interference effects, the ALD manager or the FQAO ensures the data are flagged as "Z."

15.4 Instrument Testing, Inspection, and Maintenance Requirements

ACL personnel use equipment and materials that meet SW-846 and Methods Manual requirements. ACL personnel test, inspect, and maintain instruments as recommended by the manufacturers to meet SW-846 requirements and ensure all the QAOs listed in Table 15-1 can be met. They ensure the precision QAO can be met at the PRQL concentrations listed in Table 15-1. Specific instrument testing, inspection, and maintenance schedules and actions are described in the ACL implementation plan and referenced procedures.

15.5 Instrument Calibration and Frequency

ACL personnel ensure all analytical instruments are calibrated before use and verify calibration at routine intervals as described in the ACL implementation plan. ACL personnel ensure all the initial and continuing calibration requirements listed in Table 15-3 are met and maintain instrument run logs that

permit the reconstruction of the calibration sequence and frequency. The ACL implementation plan describes and identifies procedures for instrument calibration and documentation.

Table 15-2. Summary of laboratory quality control samples and frequencies for total metals analyses.

QC Samples	Minimum Frequency	Acceptance Criteria	Corrective Actions
Method performance samples	Seven (7) samples initially, and four (4) semiannually	Meet Table 15-1 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	≤3 * IDL	Redigest and reanalyze any samples with analyte concentrations which are ≤10 * blank value and ≥0.5 * PRQL[b]
Matrix spikes	One (1) per analytical batch	80-120 %R	Specified in the ACL implementation plan[a,b]
Matrix spike duplicates	One (1) per analytical batch	RPD ≤30 80-120 %R	Specified in the ACL implementation plan[a,b]
Laboratory control samples	One (1) per analytical batch	80-120 %R([c])	Redigest and reanalyze for affected analytes[b]
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

IDL = Instrument detection limit
PDP = Performance Demonstration Program
PRQL = Program required detection limit
%R = Percent recovery
RPD = Relative percent difference

a. Matrix spikes and matrix spike duplicates that do not meet acceptance criteria due to matrix interference effects shall be flagged as "Z" and a nonconformance report is not required.

b. A nonconformance report per Section 2.1.2 of the QAPjP is required when quality control samples associated with final reported data do not meet Table 15-2 acceptance criteria.

c. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements

Table 15-3. Summary of calibration requirements and analysis for QC for total metals analyses.

Technique	Procedure	Frequency of procedure	Acceptance criteria	Corrective action
ICP-AES	1-pt. Initial calibration (1 standard and a blank)	Daily	90-110%R for independent initial calibration verification solution 95-105%R for highest calibration standard	Correct problem and repeat initial calibration
	Continuing calibration	every 10 samples plus beginning and end of run	90-110% for check standard; blank must measure ≤3 * IDL	Recalibrate and rerun last 10 samples
	Interference correction verification	Beginning and end of run or twice per 8 hours, whichever is more frequent	Solution containing interferants only must measure ≤3 * IDL for analytes; solution continuing interferants plus analytes must be 80-120%R for all analytes	Correct problem and recalibrate
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	5x dilution of sample which is >50*IDL must be ≤10%D initial value	Define in Laboratory SOPs
	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch if serial dilution, matrix spike, or matrix spike duplicate does not meet acceptance criteria	75-125 %R	Define in laboratory SOPs
CVAF	5-pt. Initial calibration (5 standards and a blank)	Daily	90-110 %R for independent initial calibration verification solution 95-105 %R for highest calibration standard r^2 must be ≥0.995	Correct problem and repeat initial calibration
	Continuing calibration	Every 10 samples plus beginning and end of run	80-120 %R for check standard (mandatory); blank (optional) should measure ≤3*IDL	Recalibrate and rerun last 10 samples
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	5x dilution of sample which is >25*IDL must be ≤10 %D of initial value	Use MSA to quantitative samples of like matrix
	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch	85-115 %R	use MSA to quantitative samples of like matrix

%D = Percent difference

IDL = Instrument detection limit

%R = Percent recovery

15.6 Data Management

The ACL implementation plan describes data reduction, validation, and reporting processes. Laboratory procedures include or reference example forms to be used to record and report data. ACL personnel reduce raw data to reportable results in compliance with the Methods Manual. ACL personnel review, validate, and verify data as described in Section 3.1.1 of this QAPjP.

ACL personnel report total metals quantitative values in mg/kg wet weight basis, limited to two significant figures.

ACL personnel submit analytical batch data reports to the SDCO and maintain analytical records as specified in Section 3.4.1 of this QAPjP. These records are subject to assessment by SPO representatives on a regular basis, as discussed in Section 3.1.2. Analytical batch data report requirements include the following:

- Laboratory name, analytical batch number, sample numbers included in that analytical batch, a cross reference to field sample numbers, and the signature releases of laboratory personnel specified in Section 3.1.1 of the QAPP
- Table of Contents
- COC form showing the date and time of sample transfer and names of individuals handling the samples from the time of sampling through receipt at the laboratory
- Data review checklists for each analytical batch verifying that data generation level review, validation, and verification have taken place.
- QC sample results (e.g., matrix spikes, matrix spike duplicates, laboratory control samples) if not included in data review checklists.
- A separate analytical report sheet titled, "Total Metal Analysis Data Sheet," for each sample in the analytical batch, including laboratory name, program name, analytical batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date extracted, date analyzed, method number, listing of program analytes, and analytical results in mg/kg wet weight basis. The following data qualifying flags are used:
  - B analyte blank concentration (laboratory or calibration verification) greater than or equal to 20% of the sample concentration prior to dilution correction
  - H Holding time exceeded
  - J Analyte is greater than or equal to IDL but less than five times the IDL before dilution correction
  - U Analyte was undetected (report IDL corrected for dilution)
  - Z One or more QC samples do not meet acceptance criteria

- Nonconformance reports, if applicable

ACL personnel maintain the following records in their files, documented and retrievable by analytical batch numbers and data report numbers:

- Original COC records
- All raw data, including original instrument readouts and/or bench reports, calculation records, and laboratory QC sample results. Matrix spike and matrix spike duplicate results are recorded along with the spiked amounts, and the RPD between the two results are calculated. Laboratory control sample results are entered with the accepted value and the %R.
- Instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the laboratory name, initial and continuing calibration verification source, method identification, and calibration date and time.
- QC result summary, which includes true and found values for all QC samples plus associated result calculations. At a minimum, the QC data include blanks, matrix spikes, matrix spike duplicates, laboratory control samples, initial calibration data, initial and continuing calibration verifications, and all other method-specific QC listed in Section 15. The QC result summary includes the laboratory name, the analytical batch number (if applicable), and method names.

15.7 Procedures Pertinent to this Section

The QAPD Procedures Matrix identifies quality procedures that implement quality requirements of the QAPD. Table 15-4 lists the major technical implementing procedures pertinent to this section of the QAPjP. The ACL implementation plan identifies additional facility procedures.

Table 15-4. Section 15 implementing procedures.

Document Number	Title
ACMM 8909	*Microwave Assisted Digestion of Homogeneous Solids and Soil Gravel*
ACMM 2900	*Determination of Trace Metals in Environmental Samples by ICP Emission Spectrometry*
ACMM 7802	*Determination of Mercury by Cold-Vapor Fluorescence Spectrophotometry*
ACMM 8969	*Determination of Percent Solids*

16. REFERENCES

10 CFR Part 71, September 1994, "Packaging and Transportation of Radioactive Material," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

10 CFR Part 830, April 1994, "Nuclear Safety Management," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

40 CFR Part 191, December 1993, "Environmental Radiation Protection Standards for the Management and Disposal of Spent Nuclear Fuel, High-Level and Transuranic Radioactive Wastes," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

40 CFR Part 194, February 1996, "Criteria for the Certification and Re-Certification of the Waste Isolation Pilot Plant's Compliance with the 40 CFR Part 191 Disposal Regulations," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

40 CFR Part 261, October 1994, "Identification and Listing of Hazardous Waste," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

40 CFR Part 268, January 1995, "Land Disposal Restrictions," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

40 CFR Part 270, December 1994, "EPA Administered Permit Programs: The Hazardous Waste Permit Program," *Code of Federal Regulations*, Washington, D.C., Office of the Federal Register National Archives and Records Administration.

ANL-W, 1998, *Argonne National Laboratory-West Quality Assurance Plan*, W0001-0929-QM-06, Argonne National Laboratory-West.

ANL-W, 1999, *Argonne National Laboratory-West Implementation Plan for the Transuranic Waste Characterization Program*, W0096-0481-ES, Argonne National Laboratory-West.

ANSI, 1987, *Standard for Software User Documentation,* New York, New York, American National Standards Institute.

ASME, 1989, *Quality Assurance Program Requirements for Nuclear Facility Applications*, ASME NQA-1, 1989 Edition, New York, New York, American Society of Mechanical Engineers.

ASME, 1990, *Quality Assurance Program Requirements of Computer Software for Nuclear Facility Applications,* ASME NQA-2a-1990, Part 2.7, Addenda to NQA-2, New York, New York, American Society of Mechanical Engineers.

Clements, T. L., Jr., 1993, *Program Plan for Certification of INEL Contact Handled Stored Transuranic Waste*, WM-PD-88-011-6, EG&G Idaho, Inc., Idaho National Engineering Laboratory.

DOE, 1992, *TRUPACT-II Content Codes (TRUCON)*, DOE/WIPP 89-004, Revision 6, Waste Isolation Pilot Plant, U.S. Department of Energy, Carlsbad, New Mexico.

DOE, 1995a, *DOE Waste Treatability Group Guidance*, DOE/LLW-217, Revision 0, Idaho Falls, Idaho, INEL-Lockheed Idaho Technologies Company, U.S. Department of Energy.

DOE, 1995b, *Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases for the TRU Waste Characterization Program*, CAO-95-1076, Revision 0, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE, 1995c, *Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program*, CAO-94-1045, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE, 1995d, *Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes,* CAO-95-1077, Revision 1, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE, 1995e, *Waste Isolation Pilot Plant Transuranic Waste Baseline Inventory Report*, CAO-94-1005, current revision, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, New Mexico.

DOE, 1998a, *Quality Assurance Program Document*, CAO-94-1012, Revision 2, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, New Mexico.

DOE, 1996b, *Transuranic Waste Characterization Quality Assurance Program Plan*, CAO-94-1010, Revision 0 with interim changes dated February 1996 and November 15, 1996, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, New Mexico.

DOE, 1996c, *Transuranic Waste Characterization Sampling and Analysis Methods Manual*, DOE/WIPP-91-043, Revision 1.0, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE, 1996d, *Waste Acceptance Criteria for the Waste Isolation Pilot Plant,* DOE/WIPP-069, Current Revision, Waste Isolation Pilot Plant, U.S. Department of Energy, Carlsbad, New Mexico.

INEEL, 1998a, *Description of the SWEPP Certified Waste Sampling Program*, EDF-RWMC-363, INEL-96/104 Lockheed Martin Idaho Technologies Company Idaho National Engineering Laboratory.

EPA, 1991, *National Enforcement Investigations Center Policies and Procedures,* EPA-330/9-78-001-R, Denver, Colorado, National Enforcement Investigations Center, U.S. Environmental Protection Agency.

EPA, 1995, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, Final Update I and Final Update II, Washington, D.C., Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency.

INEL, 1995, *Matrix Parameter Category Groups (MPCG),* INEL-95/029, EDF-RWMC-805, Lockheed Martin Idaho Technologies Company, Idaho National Engineering Laboratory.

INEL, 1998, *Transuranic Waste Sampling Plan for the INEEL*, INEL-96/295, EDF-RWMC-909, Lockheed Martin Idaho Technologies Company, Idaho National Engineering Laboratory.

INEEL, 1999a, *Analytical Chemistry Laboratory Implementation Plan for the Transuranic Waste Characterization Program*, PLN-187, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL, 1999b, *Environmental Chemistry Laboratory Implementation Plan for the Transuranic Waste Characterization Program*, PLN-186, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL 1999c, *INEEL Transuranic Waste Characterization, Transportation, and Certification Quality Program Plan*, INEEL PLN-182, March 1997 or most current revision, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL 1999d, *INEEL Transuranic Waste Program Procedures Matrix for the DOE-CAO QAPD*, PRD-198, March 1999 or most current revision, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL 1997e, *Program Description for LMITCO Quality Assurance Program*, PDP-1, January 1997 or most current revision, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL 1999f, *Program Plan for Certification of INEEL Contact-Handled Stored Transuranic Waste*, WM-PD-88-011-5, March 1999 or most current revision, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL, 1999g, *Radioactive Waste Management Complex Implementation Plan for the Transuranic Waste Characterization Program*, PLN-185, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL 1999h, *RWMC Compliance Plan for TRUPACT-II Authorized Methods for Payload Control (TRAMPAC)*, WM-PD-88-012, March 1997 or most current revision, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

INEEL, 1999i, *Site Project Office Implementation Plan for the Transuranic Waste Characterization Program*, PLN-188, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

LITCO, 1995, *Container Selection Process for SWEPP Operations*, INEL-95/016, EDF-RWMC-798, Lockheed Idaho Technologies Company, Idaho National Engineering Laboratory.

INEEL, 1997j, Project Plan for Shipment of 3,100 m^3 of Certified Transuranic Waste to WIPP, PLN-129, Current Revision, Lockheed Martin Idaho Technologies Company.

RWMC, RWMC HWMA/RCRA Permit, Permit Condition IIK-4 Report, Current Revision.

INEEL, 1997k Identification of Transuranic Waste Streams, INEL-96/349, EDF-RWMC-922, Lockheed Martin Idaho Technologies Company, Idaho National Engineering and Environmental Laboratory.

LMITCO 1998, Acceptable Knowledge Document for INEL Stored Transuranic Waste – Rocky Flats Plant Waste (INEL-96/0280)