S	ENGINEERING CHANGE NOTICE	1. ECN 651732 <hr/> Proj. ECN
	Page 1 of <u>2</u>	

2. ECN Category (mark one) Supplemental ☐ Direct Revision ☒ Change ECN ☐ Temporary ☐ Standby ☐ Supersedeure ☐ Cancel/Void ☐	3. Originator's Name, Organization, MSIN, and Telephone No. TM Greager, TRU Waste Program T4-05, 376-4344	4. USQ Required? ☐ Yes ☒ No	5. Date 08/31/99
	6. Project Title/No./Work Order No. TRU Project	7. Bldg./Sys./Fac. No. N/A	8. Approval Designator D, Q
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13a. Description of Change This ECN revises the Final Hanford Site Transuranic Waste Characterization Quality Assurance Project Plan (QAPjP) in its entirety. This change was to incorporate Carlsbad Area Office (CAO) review of enhanced program definitions.	13b. Design Baseline Document? ☐ Yes ☒ No
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14a. Justification (mark one) Criteria Change ☐ Design Improvement ☐ Environmental ☐ Facility Deactivation ☐ As-Found ☒ Facilitate Const. ☐ Const. Error/Omission ☐ Design Error/Omission ☐	14b. Justification Details Design verification not required Document is being revised in it's entirety No USQ required
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15. Distribution (include name, MSIN, and no. of copies) See attached distribution sheet	<div style="border: 2px solid black; padding: 10px; display: inline-block;"> <div style="text-align: right; font-weight: bold; font-size: small;">RELEASE STAMP</div> <div style="font-size: 2em; font-weight: bold; margin: 10px 0;">SEP 09 1999</div> <div style="display: flex; justify-content: space-between;"> <div> DATE: STA: 5 </div> <div style="border: 1px solid black; padding: 5px; text-align: center;"> HANFORD RELEASE </div> <div style="border: 1px solid black; border-radius: 50%; padding: 10px; text-align: center; width: 40px;"> 12 </div> </div> <div style="text-align: right; font-size: small;">ID:</div> </div>
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ECN 651732

19. **Change Impact Review:** Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 13. Enter the affected document number in Block 20.

20. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

21. Approvals

DEPARTMENT OF ENERGY

Signature or a Control Number that tracks the Approval Signature

See Page 11

ADDITIONAL

DISTRIBUTION SHEET

To Distribution		From TRU Waste Program		Page 1 of 1	
Project Title/Work Order HNF-2599, Hanford Site TRU Waste Characterization QA Project Plan				Date 09/07/99	
				EDT No.	
				ECN No. 651732	
Name	MSIN	Text With All Attach.	Text Only	Attach./Appendix Only	EDT/ECN Only
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Paul Crane	T4-05	x			
Bill Jasen	T4-05	x			
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Final Hanford Site Transuranic Waste Characterization Quality Assurance Project Plan

TM Greager

Waste Management Federal Services of Hanford, Inc., Richland, WA 99352
U.S. Department of Energy Contract DE-AC06-96RL13200

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Abstract: The Transuranic Waste Characterization Quality Assurance Program Plan required each U.S. Department of Energy (DOE) site that characterizes transuranic waste to be sent the Waste Isolation Pilot Plan that addresses applicable requirements specified in the QAPP.

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HNF-2599 Revision 1

**HANFORD SITE TRANSURANIC
WASTE CHARACTERIZATION
QUALITY ASSURANCE PROJECT PLAN**

HNF-2599 / Revision 1

FINAL 08/04/99

Waste Management Federal Services of Hanford, Inc.
P.O. Box 700
Richland, Washington 99352

HNF-2599 Revision 1

HANFORD SITE TRANSURANIC WASTE CHARACTERIZATION QUALITY ASSURANCE PROJECT PLAN

August 4, 1999

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ACRONYMS AND ABBREVIATIONS

%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
AK	acceptable knowledge
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BFB	4-bromofluorobenzene
B.S.	Bachelor of Science
CAO	Carlsbad Area Office
CAR	Corrective Action Report
CAS	Chemical Abstract Service
CCC	calibration check compound
CFR	<i>Code of Federal Regulations</i>
CH TRU	contact-handled transuranic
COC	chain of custody
CV	coefficient of variation
DFTPP	decafluorotriphenylphosphine
DMS	Data Management System
DOE	U.S. Department of Energy
DOE-RL	U.S. Department of Energy-Richland Operations Office
DQO	data quality objective
EICP	Extracted Ion Current Profile
EPA	U.S. Environmental Protection Agency
FDH	Fluor Daniel Hanford
FTIRS	Fourier transform infrared spectroscopy
Gas PDP Plan	<i>Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases for the TRU Waste Characterization Program (DOE 1995c)</i>
GC	gas chromatography
GC/ECD	gas chromatography/electron capture detector
GC/FID	gas chromatography/flame ionization detector
GC/MS	gas chromatography/mass spectrometry
GC/TCD	gas chromatography/thermal conductivity detector
GEA	gamma energy assay
GFAA	graphite furnace atomic absorption spectroscopy
HPLC	high-pressure liquid chromatography
ICP/AES	inductively coupled plasma/atomic emission spectroscopy
ICP/MS	inductively coupled plasma/mass spectrometry
IDL	instrument detection limit
IPAN	imaging passive/active neutron

LDA	linear diode array
LIMS	Laboratory Information Management System
LLW	low-level waste
MDC	minimum detectable concentration
MDL	method detection limit
Methods Manual	<i>Transuranic Waste Characterization Sampling and Analysis Methods Manual</i> (DOE 1996e)
NCR	nonconformance report
NDA PDP Plan	<i>Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program</i> (DOE 1995d)
NDA	nondestructive assay
NDE	nondestructive examination
NEIC	<i>National Enforcement Investigation Center Policies and Procedures</i> (EPA 1991)
NIST	National Institute of Standards and Technology
NQA-1	<i>Quality Assurance Program Requirements for Nuclear Facilities</i> (ASME 1989)
NQA-2a	<i>Quality Assurance Program Requirements of Computer Software for Nuclear Facility Applications</i> (ASME 1990)
NRC	U.S. Nuclear Regulatory Commission
NTP	National TRU Program
OJT	on-the-job training
OVA	organic vapor analyzer
PCB	polychlorinated biphenyl
PDP	Performance Demonstration Program
PFTBA	perfluorotributylamine
PRDL	Program required detection limit
PRQL	Program required quantitation limit
Project	Transuranic Waste Certification Project
Pu	plutonium
QA	quality assurance
QA/QC	quality assurance/quality control
QAO	quality assurance objective
QAPjP	quality assurance project plan
QAPD	<i>Quality Assurance Program Document</i> (DOE 1996a)
QAPP	<i>Transuranic Waste Characterization Quality Assurance Program Plan</i> (DOE 1996d)
QC	quality control
RCRA	Resource Conservation and Recovery Act
RIDS	records inventory and disposition schedule
RPD	relative percent difference
RRT	relative retention time
RT	retention time
RTL	regulatory threshold limit
RTR	real-time radiography

Solid PDP Plan	<i>Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes (DOE 1995e)</i>
SPCC	system performance check compound
SPM	site project manager
SPO	site project office
SQAO	site quality assurance officer
SVOC	semivolatile organic compound
TC	toxicity characteristic
TCO	transportation certification official
TIC	tentatively identified compound
TRAMPAC	<i>TRUPACT-II Authorized Methods for Payload Control (Appendix 1.3.7 of NRC 1997)</i>
TRU	transuranic
TRUCON	<i>TRUPACT-II Content Codes (TRUCON) (DOE 1996b)</i>
TRUPACT-II	Transuranic Package Transporter-II
TWBIR	<i>Transuranic Waste Baseline Inventory Report (DOE 1995g)</i>
UCL ₉₀	upper 90 percent confidence limit
VOC	volatile organic compound
VTSR	validated time of sample receipt
WCO	waste certification official
WG	weapons grade
WIPP	Waste Isolation Pilot Plant
WIPP WAC	<i>Waste Acceptance Criteria for the Waste Isolation Pilot Plant (DOE 1996g)</i>
WSPF	<i>Waste Stream Profile Form</i>
WWIS	WIPP Waste Information System

DEFINITIONS

Accuracy. Degree of agreement between a measured value and an accepted reference or the true value. Accuracy is determined as the percent recovery.

Analysis date/time. Date and military time (24-hr clock) of the introduction of the sample, standard, or blank into the analysis system.

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

Analytical batch. Suite of samples of a similar matrix (i.e., gas or solid) 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 [QC] 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.

Analytical method. Sample preparation and instrumentation procedures or steps that must be performed to estimate the quantity of analyte in a sample.

Analytical sample. Any solution or medium introduced into an instrument on which an analysis is performed, excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank. (The following are defined as analytical samples: TRU waste samples, duplicate samples, spiked samples, laboratory control samples, and field and manifold blanks.)

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

Audit. 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.

Blind audit sample. Sample of known composition provided as a single-blind sample to the analytical laboratory. Used by U.S. Department of Energy (DOE) to evaluate analytical laboratory performance. Blind audit samples are distributed to participating laboratories as part of the Performance Demonstration Program.

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

Calibration blank. Analyte-free matrix used to establish zero-response during calibration.

Chain of custody. Set of procedures established to ensure that sample data integrity is maintained.

Comparability. 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. Percentage of measurements made which are judged to be valid measurements. The completeness goal is to generate a sufficient amount of valid data based on Transuranic (TRU) Waste Certification Project (Project) needs. Valid results for analytical, radioassay, and radiography data are those that were obtained when the laboratory or testing facility demonstrated that the instrumentation and method were in control. That is, all calibration, verification, interference, and non-matrix checks met acceptance criteria. Valid samples are those collected and submitted for analysis that were representative and met all preservation requirements upon arrival at the laboratory.

Condition adverse to quality. All-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, and nonconformances. A significant condition adverse to quality is one that, if uncorrected, could have a serious effect on safety or operability.

Continuing calibration standards. Analytical standards run periodically to verify the calibration of the analytical system.

Control limits. Range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliance data be flagged.

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

Corrective Action Report. The identification, cause, and corrective action for conditions adverse to quality and significant conditions adverse to quality.

Correlation coefficient. Number (r) that indicates the degree of dependence between two variables (e.g., concentration and absorbance). The more dependent they are, the closer the value to one. Determined by least squares analysis.

Data quality objectives. Qualitative and quantitative statements derived from the outputs of the first six steps of the data quality objective (DQO) Process (see DQO Process, 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.

DOE Field Office. Responsible for 1) managing and executing assigned programs, 2) directing contractors who conduct the programs, and 3) ensuring that environment, safety, and health are integral parts of each program.

DQO Process. A strategic planning approach based on the scientific method used to prepare for a data collection activity. It provides a systematic procedure for defining the criteria that a data collection design should satisfy, including when and where to collect samples, the tolerable level of decision errors for the study, and how many samples to collect. By using the DQO Process, DOE will ensure that the type, quantity, and quality of environmental data used in decision making will be appropriate for the intended application. In addition, DOE will guard against committing resources to data collection efforts that do not support a defensible decision. The DQO Process consists of seven steps and is more fully described in U.S. Environmental Protection Agency (EPA) (1994).

Equipment blanks. Samples of high-purity gas or water used to establish cleanliness of sampling equipment. Collected after equipment has been cleaned and prior to sampling, and useful in documenting adequate cleaning of sampling equipment.

Equipment cleaning batch. Number of sampling equipment items cleaned at one time using the same cleaning method.

Field blanks. 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 prior to 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. Used to identify any bias in the sampling process.

Flammable volatile organic compound (VOC). A headspace gas VOC that has a National Fire Protection Association Flammability Hazard Degree of 3 or 4 and a flashpoint of less than 100°F; or that is considered by the EPA to be a significant fire hazard under Waste Isolation Pilot Plant (WIPP) repository conditions. Flammable headspace gas VOCs that are evaluated for the Project are listed in Table 1-3 of the *Transuranic Waste Characterization Quality Assurance Program Plan* (DOE 1996d) (QAPP).

Gases. Hydrogen, methane, and VOCs listed in Table 12-1 of the QAPP.

Gauge pressure. Pressure that is measured by the canister pressure gauge. Zero gauge pressure is equal to ambient barometric pressure.

Guidance material. Recommended practices to complete a given task and maintain reasonable assurance that the goals for that task will have been attained at completion. This type of material provides a means of accomplishing a task that has been found acceptable to the responsible agency. The word "should" is used to denote guidance material.

Headspace. For any volume contained by a drum or 55-gal. poly bag, 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.

High-purity gas. Gas certified by the manufacturer to contain less than 1 ppm total VOCs.

Holding time. Maximum time allowed between time of sample collection and time of preparation or analysis.

Homogeneous waste. Solid waste materials, excluding soil/gravel, that do not meet the EPA Land Disposal Restrictions criteria for classification as debris. Homogeneous solids may include water or other residual or absorbed liquids. Examples of homogeneous solids are sludges and particulate-type materials.

Independent assessment. A quality assurance (QA) program assessment conducted by an independent group or organization with the authority and freedom from the line organization to evaluate the scope, status, adequacy, programmatic compliance, and implementation effectiveness of the QA program.

Independent standard. Standard solution that is composed of analytes from a different source than those

used in the standards for the initial calibration.

Instrument calibration. Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Instrument detection limit. Minimum signal an instrument can detect with 99-percent confidence that the analyte concentration is greater than zero.

Interferants. Substances that affect the analysis for the element or compound of interest.

Item description code. Site-specific, internal numerical code applied to individual waste forms to provide identification that is used for physical segregation and computerized record keeping and tracking.

Laboratory blank. Analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample analysis. Used to document contamination resulting from the laboratory sample preparation and analytical process.

Laboratory control sample. Standard of known composition used to indicate method accuracy. Analyzed using the same analytical methods employed for the Project samples received.

Laboratory duplicate. Second aliquot of a sample that is treated the same as the original sample to determine the precision of the method.

Linear range, linear dynamic range. Concentration range over which the analytical curve remains linear.

Management assessment. Determination of managerial effectiveness in establishing and implementing QA program plans that conforms to DOE policy requirements. Based on an analysis of functional appraisals, internal audits, and other information, and on the application of appropriate criteria. A review and evaluation of management performance covering all QA and management responsibilities to ensure proper QA program balance.

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

Matrix parameter category. 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).

Method blank. Analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. Must be carried through the complete sample preparation and analytical procedure. Used to document contamination resulting from the analytical process.

Method detection limit (MDL). Minimum concentration of a substance that can be measured and reported for a given method with 99-percent confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of a sample in a given matrix type containing the analyte of interest.

Minimum Detectable Concentration (MDC) - The MDC is an a priori estimate of the detection capabilities of a given measurement system and method. This concept corresponds to a level of activity that is practically achievable with a given instrument, analytical method, and analyte/matrix combination. The MDC considers not only the instrument characteristics (background and efficiency), but all other factors and conditions that influence the measurement.

Miscertification. A waste container that is certified for disposal based on radiography, but that visual examination determines cannot be certified as meeting the WIPP WAC requirements for prohibited items.

Mixed waste. Waste regulated by both the Atomic Energy Act and the Resource Conservation and Recovery Act.

Narrative. Portion of the data package that includes descriptive documentation of any problems encountered in processing the samples, corrective actions taken, and problem resolution.

Newly generated waste. Waste generated after the development and implementation of a TRU waste characterization Project that meets QAPP requirements.

Nonconformance. Deficiency in Project requirements that renders the quality of an item or sample as unacceptable or indeterminate. Nonconforming Project data only apply to final, reported data that do not meet QA objectives.

On-line batch. Number of headspace gas samples collected and analyzed within a 12-hr period using the same on-line integrated sampling/analysis system.

Out of control. One or more of several conditions relating to the plotting of control data and indicating unacceptable results.

Packaging material. Flexible containment materials (e.g., plastic bags).

Percent difference. Difference between an initial measurement and a subsequent one; expressed as a percentage of the initial measurement.

Performance assessment. Determination of the long-term performance of the WIPP disposal system in accordance with the requirements of the EPA Standard, 40 *Code of Federal Regulations* Part 191, Subparts B and C.

Precision. 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.

Procedure. Detailed, step-by-step description of the sequence of actions to be followed in order to perform a given task. If followed in sequence, a procedure provides enough information that a trained person could complete the covered task without additional information.

Process batch. Amount of material subjected to a particular unit chemical process, unit physical mixing process, or other short-term operation that results in a substantially uniform final product and/or waste stream.

Program required detection limit. Maximum values for instrument detection limits permissible for the Program.

Program required quantitation limit. Minimum level of analyte quantitation acceptable under the QAPP.

Purge and trap. Analytical technique used to isolate volatile (purgeable) organics by stripping the compounds from water or soil with a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Quality assurance. All planned and systematic actions necessary to provide adequate confidence that a facility, structure, system, or component will perform satisfactorily and safely in service. QA goals are to ensure that research, development, demonstration, scientific investigations, and production activities are performed in a controlled manner; 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 resulting technology data are valid, defensible, and retrievable. QA includes QC 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. Characteristics of data associated with its ability to satisfy a given purpose or objective. Characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

Quality control. 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 laboratory.

Radioassay. Assay methods used to identify and quantify radionuclides in TRU waste.

Radiography. Nondestructive testing method used to inspect and determine the physical form of waste.

Recovery. 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. Usually expressed as a percent.

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

Residual material. Anything not characterized as a waste item or packaging material.

Retrievably stored waste. Waste that has been generated before the development and implementation of a TRU waste characterization Project that meets QAPP requirements.

Review/data review. Process used to ensure that the proper collection and reduction of raw data has been accomplished.

Run. Continuous analytical sequence consisting of prepared samples and all associated QA measurements as required by the QAPP.

Sample. Portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Sample number. Unique identification number designated for each sample; appears on all sample reports which document information on that sample.

Sampling batch. Suite of samples of a similar matrix (i.e., gas or solid) collected consecutively using the same sampling equipment within a specific time period. 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.

Standard deviation. Square root of the variance of a set of values.

SUMMA[®] canister, or equivalent canister. 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.

Target analytes. Those headspace gases, VOCs, semivolatile organic compounds, and metals identified by the Project as analytes of interest.

Tentatively identified compounds. Non-target compounds identified using gas chromatography/mass spectrometry. These reported concentrations will have a higher uncertainty associated with them than the reported target analyte concentrations.

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

Toxicity characteristic. A category used in defining hazardous waste based on the danger posed by the waste to plant and/or animal life. Described at 40 CFR §261.24.

Transuranic wastes. Laboratory and process wastes that contain alpha-emitting radionuclides of 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 nCi/g of waste.

Validation. Activity that demonstrates or confirms that a process, item, data set, or service satisfies user-defined requirements.

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

Variance. Measure of the dispersion of a series of results around their average; the sum of the squares of the individual deviations from the average of the results, divided by the number of results minus one.

Verification. 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.

VOCs. For the purposes of the Project, those gas VOCs listed in Table 12-1, the target VOCs listed in Table 13-1, and any additional compounds tentatively identified by the VOC analytical procedures used to satisfy Project requirements.

Waste container. Disposable containment vessel for waste materials, including integral liner or shielding materials intended for emplacement at the WIPP (i.e., 55-gal. drums or waste boxes).

Waste items. Easily identifiable discrete pieces/chunks of waste (e.g., Raschig rings).

Waste material parameter. Physical forms of waste that may impact long-term repository performance.

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

Waste stream lot. Portion of a waste stream identified for the purpose of facilitating random sampling.

**FINAL
HANFORD SITE TRANSURANIC
WASTE CHARACTERIZATION
QUALITY ASSURANCE PROJECT PLAN**

1. PROGRAM MANAGEMENT

The *Transuranic Waste Characterization Quality Assurance Program Plan* (DOE 1996d) (QAPP) requires each U.S. Department of Energy (DOE) site that characterizes transuranic (TRU) waste to be sent to the Waste Isolation Pilot Plant (WIPP) to develop and implement a quality assurance project plan (QAPjP) that addresses applicable requirements specified in the QAPP. This QAPjP describes the implementation of QAPP requirements and complies with quality assurance (QA) requirements specified in the DOE Carlsbad Area Office (CAO) *Quality Assurance Program Document* (DOE 1996a) (QAPD). Requirements contained in the QAPD are based on requirements and criteria outlined in 10 Code of Federal Regulations (CFR) Part 830. The QAPP satisfies applicable requirements of 10 CFR Part 830.120. Table 1-1 provides a cross-reference of QA requirements of this QAPjP to those of CAO QAPD.

This QAPjP establishes requirements to be met by Hanford Site organizations in characterizing waste for shipment to WIPP. These requirements are implemented by the various Hanford Site facilities supporting the Hanford Site TRU Waste Project (Project) in the implementing procedures for each facility. This document does not eliminate applicable Fluor Daniel Hanford (FDH) QA requirements as applicable to non-quality affecting activities associated with the characterization, certification, and transportation of TRU wastes. It provides QA requirements applicable only to the TRU Project activities as defined in this document.

1.1 PROJECT ORGANIZATION

Figure 1-1 is a functional organization chart for TRU waste characterization activities at the Hanford Site performed as part of the Project. The following subsections describe specific Project participants and responsibilities.

1.1.1 National TRU Program Team Leader

The CAO manager delegates the responsibility of overseeing and providing guidance on TRU waste testing, sampling, analysis, sample handling and custody, and associated data management at the Hanford Site to the National TRU Program (NTP) team leader. The NTP team leader is responsible for overseeing TRU waste characterization activities at participating sites, ensuring that NTP requirements are met, and reviewing and concurring with this QAPjP. The NTP team leader, in conjunction with the CAO QA manager, is responsible for conducting audits of these activities as described in the QAPP and QAPD and oversees the Performance Demonstration Program (PDP) required for testing and analytical facilities at the Hanford Site. As part of these activities, the NTP team leader is responsible for reviewing and approving this QAPjP.

Table 1-1. Cross-Reference of Quality Assurance Requirements.

CAO QAPD Requirements	QAPjP Section
Section 1. Project Management	
Organization and Quality Assurance Program Documents	Program Organization
Organization and Quality Assurance Programs	Program Documents
Organization and Quality Assurance Program	Problem Definition and Background
Organization and Quality Assurance Program	Program Description
Planning Scientific Investigations	DQOs
Personnel Qualification and Training	Special Training Requirements and Certifications
Records	Records Management
Data Documentation, Control, and Validation	
Procurement	Procurement
Work Processes	Work Processes
Software Requirements	
Section 2. Assessment and Oversight	
Quality Improvement Management Assessment Independent Assessment	Assessment and Response Actions
Quality Improvement Independent Assessment	Reports to Management
Inspection and Testing	Performance Demonstration Program
Section 3. Data Validation, Usability, and Reporting	
Data Documentation, Control, and Validation	Data Review, Validation, and Verification Requirements
Data Documentation, Control, and Validation	Validation Methods
Data Documentation, Control, and Validation	Reconciliation with DQOs
Records	Data Reporting
Data Documentation, Control, and Validation	
Section 4. Acceptable Knowledge	
Planning Scientific Investigations	QAOs
Work Processes Performing Scientific Investigations	Procedural Requirements
Work Processes	Acceptable Knowledge Information
Data Documentation, Control, and Validation	Acceptable Knowledge Program Control
Independent Assessment	Audits

Section 5. Sampling Process Design

Planning Scientific Investigations

Sampling Process Design

Section 6. Waste Container and Sample Handling and Custody Requirements

Sample Identification

Field documentation

Handling, Storing, and Shipping Samples

Labeling

Disposition of Nonconforming Samples

Chain of custody

Work Processes

Waste container and sample handling

Sections 7 through 15. Techniques

Planning Scientific Investigations

QAOs

Work Processes

Methods Requirements

Performing Scientific Investigations

Quality Control

Work Processes

Equipment Testing, Inspection, and Maintenance Requirements

Inspection and Testing

Work Processes

Equipment Calibration and Frequency

Inspection and Testing

Records

Data Management

Data Documentation, Control, and Validation

CAO	=	Carlsbad Area Office
DQO	=	data quality objective
QAO	=	quality assurance objective
QAPD	=	<i>Quality Assurance Program Document</i> (DOE 1996a)
QAPjP	=	Quality Assurance Project Plan

Figure 1-1. Hanford Site Project Functional Organizational Chart

1.1.2 DOE-Richland Waste Programs Division

The DOE-Richland Operations Office (RL) Waste Programs Division is responsible for reviewing and approving this QAPjP and all revisions affecting compliance with QA/quality control (QC) requirements. The RL TRU waste program manager ensures that characterization activities are conducted in compliance with DOE orders, the QAPP, the QAPD, this QAPjP, and all applicable federal and state regulations. The RL TRU waste program manager provides an interface between the operating contractors at the Hanford Site and the CAO, other DOE operations offices, and DOE Headquarters to resolve any problems that could affect project quality.

1.1.3 FDH TRU Program Integration Manager

FDH is the management and integration contractor that interfaces between RL and the Hanford Site TRU Project. The FDH TRU program integration manager interfaces between the TRU site project manager (SPM) and RL TRU waste program manager and ensures that Project plans and operations are coordinated, integrated, and consistent with Waste Isolation Pilot Plant (WIPP) programs, policies, and guidance.

1.1.4 Program Manager – Waste Management Operations

The Program manager of Waste Management Operations ensures the Hanford site-specific safety, quality, and environmental compliance requirements are maintained. The Program manager interfaces with the FDH TRU program integration manager to ensure proper and consistent coordination between TRU Project and site implementation strategies.

1.1.5 Site Project Manager

The SPM is responsible for Project planning and TRU waste operations at the Hanford Site. The SPM reviews and approves this QAPjP and annual revisions, prepares progress reports as requested by RL, oversees TRU waste activities, and ensures that Project personnel receive appropriate indoctrination and training in Project objectives and requirements. The SPM coordinates with the Hanford Site waste certification official (WCO) and transportation certification official (TCO) to ensure that Hanford Site TRU waste is characterized and certified compliant with WIPP requirements. The TCO and WCO report to the SPM. The SPM is responsible for managing the following Project activities in compliance with the QAPP, the QAPD, and this QAPjP:

- Selecting and tracking waste
- Validating and verifying project-level testing and analytical data
- Reconciling testing and analytical data with data quality objectives (DQOs)
- Assigning U.S. Environmental Protection Agency (EPA) hazardous waste numbers
- Submitting QA/QC reports to the FDH TRU program integration manager for forwarding to RL
- Transmitting testing, sampling, and analytical data to CAO.

The SPM may delegate these activities to another individual.

1.1.6 Site Quality Assurance Officer

The site quality assurance officer (SQAQO) ensures that all Project QA requirements are implemented in accordance with this QAPjP. This includes verification and assessments of the QA program. The SQAQO reviews and approves this QAPjP and provides day-to-day guidance to Project staff on quality-related matters. The SQAQO identifies and reports quality problems to the SPM and initiates, recommends, and tracks corrective actions to closure. The SQAQO has the authority to stop TRU waste characterization activities at any participating facility if quality is not assured or controlled. The SQAQO, supported by QA personnel at each facility, is responsible for the following activities:

- Coordinating internal and external audits and assessments to verify compliance
- Tracking and evaluating trends in compliance with QA objectives (QAOs) established in the QAPjP by performing the following:
 - Ensuring that laboratory/testing facilities are assessed
 - Ensuring that nonconformance reports (NCRs) and corrective action reports (CARs) are prepared, when appropriate
 - Tracking and trending nonconformances
 - Tracking and verifying corrective actions
 - Validating and verifying project-level testing and analytical data
 - Submitting QA/QC reports to the SPM and FDH TRU program integration manager for forwarding to RL

1.2 PROJECT DOCUMENTS

Figure 1-2 shows the hierarchy of documents by which the Project is implemented. For purposes of the Project, the term "procedure" refers to any site- or facility-specific implementing document (e.g., standard operating procedures and operating manuals).

1.2.1 Carlsbad Area Office Documents

Carlsbad Area Office (CAO) documents that specify the requirements for TRU waste characterization include the *Waste Acceptance Criteria for the Waste Isolation Pilot Plant* (DOE 1996g) (WIPP WAC); QAPD; QAPP; and *TRUPACT-II Authorized Methods for Payload Control* (Appendix 1.3.7 of U.S. Nuclear Regulatory Commission [NRC] 1997) (TRAMPAC). The WIPP WAC specifies the parameters for waste acceptance and the conditions or limits, including transportation requirements that must be met for each criterion for waste to be disposed at the WIPP. The QAPD describes and establishes the CAO QA program and specifies the QA requirements for all participants in WIPP-related activities. The QAPP specifies the waste testing, sampling, and analysis requirements established in the *WIPP RCRA Part B Permit Application*, "Waste Analysis Plan" (DOE 1996f) and the Title 40 CFR 191 Compliance Certification Application (DOE 1996c). The TRAMPAC specifies the payload requirements to transport TRU waste to WIPP using the TRUPACT-II.

1.2.2 Quality Assurance Project Plan

This QAPjP follows the document format specified in the QAPP and is implemented by Hanford Site and facility procedures that address TRU waste characterization activities. A table at the end of each section lists procedures that implement QAPP requirements. The SPM reviews this QAPjP at least annually and coordinates the review and approval of the revised document. Distribution of this QAPjP and control of changes are described in "TRU Document Control" (WMH-400, Section 1.4.1). Section 1.2.4 of the QAPjP and Table 1-2 specify minimum review and approvals required before implementation of the QAPjP.

Procedures are developed for all activities affecting Project quality according to "TRU Operating Procedure Preparation and Approval" (WMH-400, Section 2.1.2), "TRU Administrative Procedure Preparation and Approval" (WMH-400, Section 2.1.3), and "TRU Analytical Procedure Process" (WMH-400, Section 2.1.6), which describe the organization and format for Project procedures. The most recent revision of these procedures is available electronically on a network drive. Personnel ensure that they are working to the most up-to-date version of the applicable procedure by accessing the electronic version via the Hanford site shared drive document system or by comparing their hard copy of the procedure to the electronic version.

1.2.3 Facility QA Plans and Procedures

Facility personnel perform Project activities in accordance with written procedures that comply with the QAPP and QAPD. Procedures are written using the graded approach to meet Project QA requirements as described in WMH-400, Sections 2.1.2, 2.1.3, and 2.1.6. Procedures include examples of data (e.g., reports, forms, and data validation checklists), as appropriate. Data forms used in the TRU Project are available via the shared drive document system. Facility procedures are approved at the facility level. Each procedure specifies internal review and approval requirements. Facility QA procedures for TRU Project activities (e.g., records management) must be equivalent to Project QA plans and procedures.

All Project personnel are responsible for reporting any obsolete or superseded information to the SPM. All Hanford Site-specific information is evaluated and approved by the SPM and SQAQ before implementation. The SPM will notify appropriate personnel and the DOE field office of the changes. Changes that affect performance criteria or data quality will be approved by the DOE field office and the NTP team leader prior to implementation. Minor changes to the QAPjP and procedures that do not affect performance criteria or data quality may be made without notifying the DOE field office and the NTP team leader. WMH-400, Sections 2.1.2, 2.1.3, and 2.1.6, specify the process for initiating, revising, modifying, reviewing, and distributing Project documents and changes to Project documents. As potential changes to Project information are identified by the SPM, documents will be revised as necessary and distributed to affected organizations in accordance with this procedure.

1.2.4 Document Review, Approval, and Control

Hanford Site personnel implement the document control system in accordance with WMH-400, Section 1.4.1. QAPjP revisions (other than editorial changes) that affect performance criteria or data quality (e.g., sample handling and custody requirements, sampling and/or analytical methods, QAOs, calibration requirements, or QC sample acceptance criteria) are reviewed and approved by the same level of authority as the original QAPjP. The requirements for review and approval of the QAPjP are shown in Table 1-2. All QA documents are identified with a unique number, revision number, date, and page number. Revised QA document submittals also identify the changes, the reason for the changes, and the justification for concluding that the revised contents continue to satisfy the Project requirements.

Figure 1-2. Hanford Site TRU Waste Certification Project Document Hierarchy

Table 1-2. Review, Approval, and Control Requirements for the Hanford Site QAPjP

Organization/Reviewer	Review	Review and approval	Change approval	Change control	Implement-ation
National TRU Program Team Leader	—	X	X ^a	—	—
Carlsbad Area Office Quality Assurance Manager	—	X	X	—	—
RL TRU Waste Program Manager	—	X	X ^a	—	—
FDH TRU Program Integration Manager	X	—	—	—	—
Site Project Manager	—	X	X	X	X
Site Quality Assurance Officer	—	X	X	—	X

Note: — = not required.

^a Changes affecting performance criteria or data quality only.

1.3 PROBLEM DEFINITION AND BACKGROUND

This QAPjP applies to all waste at the Hanford Site that is defined as retrievably stored and newly generated contact-handled TRU (CH TRU) waste. Characterization requirements for remote-handled (RH) TRU waste are not fully implemented but will be addressed in future revisions of this QAPjP. Waste generated at the Hanford Site will be placed in 55-gal. (208-L) drums, standard waste boxes, or other containers acceptable for shipment to WIPP and will typically be managed as retrievably stored waste for characterization purposes. Newly packaged or repackaged waste may also be managed as newly generated waste at the Hanford Site.

1.4 PROJECT DESCRIPTION

The Project certifies (which includes characterization) newly generated and retrievably stored TRU waste for shipment to the WIPP. The Hanford Site characterizes TRU waste using acceptable knowledge (AK); testing, and sampling; and analytical techniques described in this QAPjP. Project facilities implement the QAPP requirements as described in this QAPjP and facility procedures.

The Project is designed to characterize and certify 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. Hanford Site waste streams are described in the *Transuranic Waste Baseline Inventory Report* (DOE 1995e) (TWBIR). The Project has adopted the waste description nomenclature outlined in the *DOE Waste Treatability Group Guidance* (DOE 1995a). The Project 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's physical form and determine characterization requirements for the Project.

The SPM provides overall management and coordination for the characterization of TRU waste at the Hanford Site. The SPM statistically selects waste containers for solidified waste sampling and visual examination (VE); validates and verifies all sampling, testing, and analytical data; and transmits data to CAO. The SQAQO ensures that independent assessments are conducted at Hanford Site facilities involved with the Project. The SPM coordinates with RL and CAO on NTP issues that affect the Hanford Site.

The Hanford Site conducts TRU waste characterization activities following the process flows depicted on Figures 1-3a through 1-3f. Sections 9 through 15 of this QAPjP and facility procedures identify waste parameters, target analytes, characterization techniques, and QAOs for each characterization technique used. Facility operations include waste storage, retrieval, nondestructive assay (NDA), nondestructive examination (NDE), VE, waste repackaging, and headspace gas and homogeneous solids and soil/gravel sampling and analysis.

1.5 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) are qualitative and quantitative statements that clarify Project technical and QAOs, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. The DQOs established for the Project ensure that regulatory requirements are met and address the specific waste characterization parameters that are evaluated. The DQOs established for each characterization activity of the Project are summarized below:

Radioassay (Section 9):

- To classify waste by activity as low-level waste (LLW) versus TRU waste by demonstrating with a 95 percent probability that the total TRU activity is less than or equal to 100 nCi/g of waste. The 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 LLW.
- To confirm the radionuclide inventory on which the 40 CFR Part 194 Certification Decision; Final Rule (May 18, 1998) is based.
- 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 (Section 10):

- To classify/verify the TRU waste inventory by matrix parameter category and waste material parameter as specified in the TWBIR, on which the 40 CFR Part 194 Certification Decision; Final Rule (May 18, 1998) is based.
- To verify the TRU waste streams by matrix parameter category for purposes of physical waste form identification and determination of sampling and analytical requirements (Section 5).

Figure 1.3a. Transuranic Waste Processing at the Hanford Site

Figure 1.3b. Transuranic Waste Processing at the Hanford Site (continued)

Figure 1.3c. Transuranic Waste Processing at the Hanford Site (continued)

Figure 1.3d. Transuranic Waste Processing at the Hanford Site (continued)

Figure 1.3e. Transuranic Waste Processing at the Hanford Site (continued)

Figure 1.3f. Transuranic Waste Processing at the Hanford Site (concluded)

Gas Sampling and Analysis (Sections 7, 11, and 12):

- To quantify the concentration of hydrogen, methane, and flammable volatile organic compounds (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 hydrogen and methane headspace concentrations in waste containers to support revision of the thermal power restrictions for shipment of TRU waste in the TRUPACT-II.

Homogeneous Solid and Soil/Gravel Sampling and Analysis (Sections 8, 13, 14, and 15):

- To compare the UCL₉₀ values for the mean measured contaminant concentrations in a waste stream to the specified regulatory threshold limit values (i.e., to determine whether a waste stream exhibits a toxicity characteristic)
- To report the average concentrations, standard deviation, UCL₉₀, and number of samples collected for hazardous constituents in a waste stream, as specified in 40 CFR Part 261, Appendix VIII.

1.6 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATIONS

Training records are maintained in the SPO files, as described in Section 1.7. The SPM and facility, laboratory, and other support managers ensure that all Project personnel receive indoctrination into the scope, purpose, and objectives of the Project and the specific QAOs of the task being performed. Project personnel receive initial and continuing training requisite with their activities and level of responsibility, and maintain minimum qualifications as described in "TRU Training and Qualification Plan" (WMH-400, Section 1.2.1). Before beginning work under the Project, personnel qualifications will be evaluated for compliance with Project training and qualification requirements. Personnel who are found to be deficient with regard to the requirements for their assigned position will receive the appropriate training to ensure that the qualification requirements are met before participating in Project-related activities. Facility personnel performing activities affecting quality are trained according to facility training plans to ensure that they achieve and maintain suitable proficiency. It may be necessary to complete initial qualification of specific project personnel prior to establishing a formal on-the-job training (OJT) program.

These personnel will be evaluated by the training manager for qualification and will perform OJT of subsequent personnel. Table 1-3 specifies the minimum qualifications for radioassay, radiography, and analytical laboratory personnel. Facility procedures identify the facility-specific job titles that correspond to the positions listed in Table 1-3. Job performance is evaluated, where appropriate, and documented at periodic intervals not to exceed two years.

1.7 RECORDS MANAGEMENT

Quality records at the Hanford Site are maintained consistent with the requirements of the QAPP and the QAPD to ensure that objective evidence of quality is retrievable. Site project office (SPO) personnel maintain Project files in accordance with "TRU Records Management" (WMH-400, Section 1.5.1).

Table 1-3. Minimum Training and Qualifications Requirements.^a

Personnel	Requirements ^a
NDA Operators ^b	Training based on ASME NQA-1, Element 2 (except Supplement 2S-2) (ASME 1989); requalification every 2 years
Radiography Operators ^b	Training based on ASME NQA-1, Element 2 (except Supplement 2S-2) (ASME 1989 and SNT-TC-1A (ASNT); matrix parameter categories and waste material parameters; requalification every 2 years
GC Technical Supervisors ^c GC Operators ^b	B.S. degree or equivalent experience and 6 months of previous applicable experience
GC/MS Operators ^b MS Operators ^b	B.S. degree or equivalent experience and 1 year of independent spectral interpretation or demonstrated expertise
GC/MS Technical Supervisors ^c MS Technical Supervisors ^c	B.S. degree or equivalent experience and 1 year of applicable experience
Atomic Absorption Spectroscopy Technical Supervisors ^c Atomic Absorption Spectroscopy Operators ^b Atomic Mass Spectrometry Operators ^b Atomic Emission Spectroscopy Operators ^b	
Atomic Mass Spectrometry Technical Supervisors ^c	B.S. degree and specialized training in Atomic Mass Spectrometry and 2 years of applicable experience
Atomic Emission Spectroscopy Technical Supervisors ^c	B.S. degree and specialized training in Atomic Emission Spectroscopy and 2 years of applicable experience

^a Education and experience levels for analytical personnel based on requirements contained in *USEPA Contract Laboratory Program Statement of Work for Organics Analysis* (Document No. OLM 03.0) (EPA 1993) and *Statement of Work for Inorganics Analysis* (Document No. ILM 04.0) (EPA undated).

^b Operators are those persons responsible for the actual operation of analytical equipment. Facility procedures include the facility-specific title for this position.

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

ASME = American Society of Mechanical Engineers
 B.S. = Bachelor of Science
 GC = gas chromatography
 MS = mass spectrometry
 NDA = nondestructive assay

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. Project-related characterization records are generated by various facilities at the Hanford Site. Calibration services may be obtained from offsite vendors. Records documenting characterization, certification, and transportation activities will be provided to the SPO as working or in-process files. The SPO shall complete any validation or reviews necessary, designate which records are QA records, and assign record holding times as detailed in WMH-400, Section 1.5.1. The Project records custodian shall ensure that records designated as QA records are stored and maintained in either dual storage or in 2-hour-rated fireproof safes. Temporary storage in 1-hour-rated fireproof safe is authorized for a period not to exceed 1 year.

Table 1-4 lists the Project records that are maintained as lifetime and nonpermanent. Lifetime records are maintained at the SPO for the life of the Hanford Site Project plus 6 years and then offered to CAO or transferred to the appropriate Federal Records Center. Nonpermanent records are maintained at the Site project office for 10 years from the date of record generation and dispositioned according to the approved Records Inventory and Disposition Schedule (RIDS). SPO personnel or facility managers maintain records necessary to resolve an enforcement action, regardless of the disposition, until WIPP enforcement actions are completed.

1.8 PROCUREMENT

All Hanford Site TRU Project facilities implement procedures to ensure that 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 are implemented according to "TRU Procurement Planning" (WMH-400, Section 2.3.1) and the procedures specified in the following subsections.

1.8.1 Procurement Document Control

The SPM and facility managers ensure that Project personnel control procurement documents in accordance with "TRU Procurement Document Control" (WMH-400, Section 2.3.2). Procurement documents supporting waste management activities must include required specifications and acceptance criteria. Procurement documents are reviewed by appropriate organizations and engineering disciplines to ensure that they contain adequate scope of work, technical requirements, supplier QA program requirements, and provisions for acceptance. The procurement document control system is also defined in WMH-400, Section 2.3.2.

1.8.2 Control of Purchased Items and Services

The SPM and facility managers ensure that Project personnel control items and services purchased (including supplier evaluations and inspections) in accordance with "TRU Control of Purchased Items and Services" (WMH-400, Section 2.3.3) and "TRU Inspection Control" (WMH-400, Section 2.4.1). Potential suppliers of goods and services to the Hanford TRU Waste Project will have their own QA program or will comply with the requirements contained in this QAPjP.

Table 1-4. Classification for Waste Characterization Quality Assurance Records.

Records		
Lifetime		
•	Field sampling data forms	
•	Field and laboratory COC forms	
•	Test facility and laboratory analytical data reports	
•	Summary data packages	
•	Sampling plans	
•	Data reduction, validation, and reporting documentation	
•	Acceptable Knowledge (AK) documentation	
•	Data reconciliation report	
•	Waste Stream Profile Form (WSPF)	
Nonpermanent		
•	Nonconformance documentation	
•	Variance documentation	
•	Assessment documentation	
•	Gas canister tags (or equivalent documentation)	
•	Methods performance documentation	
•	PDP documentation	
•	Sampling equipment certifications	
•	Calculations and related software documentation	
•	Training/qualification documentation	
•	QAPjP (Hanford Site) documentation (all revisions)	
•	Calibration documentation	
•	Analytical raw data	
•	Procurement documentation	
•	QA procedures (all revisions)	
•	Technical implementing procedures (all revisions)	
•	Audio/video recording (radiography, visual, etc.)	
<hr/>		
COC	=	chain of custody
PDP	=	Performance Demonstration Program
QA	=	quality assurance
QAPjP	=	Quality Assurance Project Plan
WSPF	=	Waste Stream Profile Form

1.8.3 Control of Subcontractors

Section 1.8.2 also applies to subcontractors who perform work that directly affects the quality of characterization data. The procedure WMH-400, Section 2.3.3, describes how Project personnel control subcontractor services. Potential subcontractors will be informed of the need to perform operations in compliance with QAPP requirements, including the requirements for training and qualification of personnel. Assessments and audits of subcontractors will be performed and documented to ensure compliance with QAPP and QAPjP requirements. Subcontractor personnel must meet applicable Project training and qualification requirements. Subcontractors shall submit copies of all Project-related documents to the SPM.

1.9 WORK PROCESSES

Project personnel conduct TRU waste characterization operations according to approved procedures that comply with the QAPjP and QAPD requirements. Each section of this QAPjP lists TRU waste characterization procedures applicable to specific work processes.

1.9.1 Control of Processes

The SPM and facility managers ensure that Project activities are controlled and conducted in accordance with "TRU Process Control" (WMH-400, Section 2.1.1) and facility-specific procedures that describe and control work processes applicable to TRU waste characterization. If equipment is designed for waste characterization, site personnel comply with QAPD design control requirements, as described in "TRU Design Control" (WMH-400, Section 2.2.1). Equipment is tested, inspected, and maintained according to QAPD requirements, as described in Sections 7 through 15.

1.9.2 Identification and Control of Items

Project personnel identify and control items (e.g., items with limited shelf or operating lives, materials, equipment, samples) and ensure that only correct and accepted items are used according to "TRU Inspection, Test, and Operating Status" (WMH-400, Section 2.4.3). Section 6 addresses identification and control of waste containers and samples.

1.9.3 Computer Hardware and Software

Computer software and hardware/software configurations used in Project activities are developed, documented, verified, validated, and tested prior to use in compliance with requirements contained in the QAPD, QAPjP, and NQA-1, Subpart 2.7, "Quality Assurance Requirements of Computer Software for Nuclear Facility Applications" (ASME 1989). "TRU Software Quality Assurance" (WMH-400, Section 6.1.1) describes the processes for computer software development, validation, and verification.

1.10 PROCEDURES PERTINENT TO THIS SECTION

Table 1-5 lists the procedures pertinent to this section of the QAPjP.

Table 1-5. Section 1 Implementing Procedures.

Document number	Title
WMH-400, Section 1.2.1	<i>TRU Training and Qualification Plan</i>
WMH-400, Section 1.4.1	<i>TRU Document Control</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 2.1.1	<i>TRU Process Control</i>
WMH-400, Section 2.1.2	<i>TRU Operating Procedure Preparation and Approval</i>
WMH-400, Section 2.1.3	<i>TRU Administrative Procedure Preparation and Approval</i>
WMH-400, Section 2.1.6	<i>TRU Analytical Procedure Process</i>
WMH-400, Section 2.2.1	<i>TRU Design Control</i>
WMH-400, Section 2.3.1	<i>TRU Procurement Planning</i>
WMH-400, Section 2.3.2	<i>TRU Procurement Document Control</i>
WMH-400, Section 2.3.3	<i>TRU Control of Purchased Items and Services</i>
WMH-400, Section 2.4.1	<i>TRU Inspection Control</i>
WMH-400, Section 2.4.3	<i>TRU Inspection, Test, and Operating Status</i>
WMH-400, Section 6.1.1	<i>TRU Software Quality Assurance</i>

2. ASSESSMENT AND OVERSIGHT

Project participants implement an assessment and oversight program to meet QAPP and QAPD requirements. Assessment and oversight actions include independent assessments, management assessments, reports to management, and performance demonstration program (PDP) participation. Implementation of these activities ensures quality improvement of the Project.

2.1 ASSESSMENT AND RESPONSE ACTIONS

The SQAQO has overall responsibility for ensuring that Project personnel conduct assessment and response actions in accordance with QAPP and QAPD requirements. When conditions are identified that are adverse to the requirements, Project personnel initiate and document corrective actions.

2.1.1 Audits

Facilities participating in the Project are subject to CAO audits. A CAO audit of the Project is conducted before waste is shipped to WIPP and annually thereafter. These audits are the responsibility of the CAO QA manager, who coordinates these audits through the SPM or designee and site quality assurance officer (SQAQO). The SQAQO ensures that all conditions adverse to quality are resolved and appropriate corrective actions are implemented in a timely manner. The SQAQO develops a schedule, in association with Project facility managers, that details follow-up activities and final resolution of all corrective actions. The SQAQO tracks corrective actions to completion and monitors the status of the corrective actions to ensure timely closure of nonconforming conditions.

2.1.2 Nonconformances

A nonconformance is a deficiency in a Project requirement that renders the quality of an item or sample as unacceptable or indeterminate. Nonconformances include uncontrolled and unapproved deviations from an approved plan or procedure. Nonconforming items and activities are those that do not meet Project requirements.

All Project participants are responsible for quality improvement, including identifying and reporting nonconforming items and processes adverse to quality. Nonconforming items are marked and segregated as necessary to prevent their inadvertent use and are dispositioned appropriately. The SQAQO and facility managers are responsible for evaluating nonconformances and taking appropriate corrective action. "TRU Nonconforming Item Reporting and Control" (WMH-400, Section 1.3.2) and "TRU Corrective Action Reporting and Control" (WMH-400, Section 1.3.3) identify the process used to control nonconforming items. These procedures identify the person(s) responsible for evaluating, dispositioning, and controlling nonconformances and segregating or otherwise tracking nonconforming items. The individual identifying the nonconformance prepares a nonconformance report (NCR) or corrective action report (CAR). NCRs are normally developed for nonconforming items and CARs are developed to correct a deficient process. Facility personnel report Project-related nonconformances and transmit copies of NCRs and CARs to the SQAQO.

Each NCR or CAR includes the following information:

- Identification of individual(s) identifying or originating the NCR or CAR
- 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 usability of the data, if applicable
- Approval signatures of facility personnel.

2.1.3 Quality Improvement

Project personnel continually evaluate and improve Project activities and implement QAPD quality improvement criteria. Project personnel detect and prevent conditions adverse to quality, ensure quality improvement, and identify trends as described in Sections 2, "Assessment and Oversight," and 3.1, "Data Review, Validation, and Verification Requirements." Project personnel use processes such as initial planning; appropriate training; control of work operations by procedures, audits, control charts, NCRs, CARs, and nonconformance and corrective action tracking; and data review, validation, and verification to detect and prevent quality problems. Information that may be analyzed to identify trends that adversely impact quality include nonconformance information; sampling, testing, and analytical information, such as equipment checks and characterization data; and waste shipment information. "TRU Corrective Action Management" (WMH-400, Section 1.3.1) describes the processes for detecting and preventing quality problems and ensuring quality improvement.

2.1.4 Management Assessment

Project managers at all levels periodically assess the Project and its performance. Management assessments focus on identifying, correcting, and preventing management and technical problems that hinder the achievement of Project objectives. Management assessments evaluate customer requirements and expectations and employee perceptions. Management assessment results are documented in writing and used as input to the quality improvement process. Management assessment activities are described in "TRU Management Assessment" (WMH-400, Section 3.1.1) and facility procedures.

2.1.5 Independent Assessment

Facilities are subject to independent assessments (audits and surveillances) performed by an independent audit team assembled by the SQAQO. The assessments focus on performance of work in accordance with the requirements of this QAPjP and are performed according to "TRU Independent Assessments" (WMH-400, Section 3.2.1) and "TRU Surveillance Program" (WMH-400, Section 3.2.2). The SQAQO ensures that analytical laboratories and characterization facilities are assessed. At a minimum, a quarterly assessment includes a repeat of data-generation-level review, validation, and verification for one randomly selected waste container. The SQAQO determines whether the independent assessment is an audit or process surveillance (as defined in the QAPD). In addition, facility QA officers may perform independent assessments (audits or process surveillances) of Project activities at their facilities.

The independent assessment team is made up of a team leader appointed by the SQAQO and team members and technical specialists selected by the team leader in conjunction with the SQAQO. The team leader provides indoctrination and supervision of the team, organizes and directs the assessment, establishes the scope of the assessment, prepares a plan for conducting the assessment, and prepares and issues an assessment report to the management of the assessed organization and any affected organizations. The assessment team members and technical specialists prepare the assessment checklist, conduct the assessment, brief the management of the assessed organization on a daily basis, and prepare a draft report for presentation at the exit conference for the assessment. Assessments are performed in accordance with WMH-400, Sections 3.2.1 and 3.2.2.

2.2 REPORTS TO MANAGEMENT

The SQAQO provides the QA interface between facilities and the SPM. The SQAQO oversees the NCR/CAR process for Project-related deficiencies and coordinates with facility managers to track nonconformances and verify corrective action completion according to WMH-400, Sections 1.3.2 and 1.3.3. Facility QA officers report the results of their independent assessments to the SQAQO, and together they track assessment results and corrective actions. The SQAQO reports these independent assessment results to the SPM in accordance with "Quality Assurance Reports to Management" (WMH-400, Section 3.1.2). Also, the SQAQO prepares and transmits a semiannual QA report to the SPM and the DOE. The SPM forwards a copy of the report to the FDH TRU program integration manager. The FDH TRU program integration manager forwards a copy of the report to RL. The semiannual QA report includes the following information, as appropriate:

- Any QAPjP changes
- Identification of any significant QA/QC 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 audits, assessments, and surveillances conducted during the period.

2.3 PERFORMANCE DEMONSTRATION PROGRAM

The Hanford Site TRU Project facilities participate in the PDP, as summarized in Table 2-1. PDP samples are processed according to the facility procedures listed in Sections 9 and 11 through 15 of this QAPjP.

Table 2-1. Performance Demonstration Program Participation.

Hanford Site Facility	PDP Plan
NDA facility	<i>Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program (DOE 1995c) (NDA PDP Plan)</i>
Headspace Gas Analytical laboratory	<i>Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases for the TRU Waste Characterization Program (DOE 1995b) (Gas PDP Plan)</i>
Solidified Waste Analytical laboratory	<i>Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes (DOE 1995d) (Solid PDP Plan)</i>

2.4 PROCEDURES PERTINENT TO THIS SECTION

Table 2-2 lists the procedures pertinent to this section of the QAPjP.

Table 2-2. Section 2 Implementing Procedures.

Procedure Number	Title
WMH-400, Section 1.3.1	<i>TRU Corrective Action Management</i>
WMH-400, Section 1.3.2	<i>TRU Nonconforming Item Reporting and Control</i>
WMH 400, Section 1.3.3	<i>TRU Corrective Action Reporting and Control</i>
WMH-400, Section 3.1.1	<i>TRU Management Assessments</i>
WMH-400, Section 3.1.2	<i>Quality Assurance Reports to Management</i>
WMH-400, Section 3.2.1	<i>TRU Independent Assessments</i>
WMH-400, Section 3.2.2	<i>TRU Surveillance Program</i>

3. DATA VALIDATION, USABILITY, AND REPORTING

Data generated during Project testing, sampling, and analytical activities are recorded, reviewed, validated, verified, and reported to the SPO as batch data reports. Sampling personnel collect headspace gas samples and homogeneous solids and soil/gravel samples in sampling batches. NDA, NDE, and VE personnel test waste containers in testing batches. Analytical 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 Sections 11 through 15 include the requirements for preparing analytical batch data reports.

3.1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

To ensure that Project data meet the level of quality required in the QAPP, Project personnel review, validate, verify, and report data at two levels: data generation level and project level. Sections 7 through 15 list procedures that describe the processes for data-generation-level validation, verification, and reporting.

3.1.1 Data Generation-Level Data Validation

Project personnel comply with the following minimum requirements for raw data collection and management, as applicable:

- Sign and date all raw data in permanent, reproducible ink (or equivalent for electronic data).
- Record clearly, legibly, and accurately all data in field and laboratory records (e.g., bench sheets, logbooks, electronic data systems), and include applicable sample identification numbers.
- Line out, initial, and date all changes to original data. When appropriate or not obvious, include justification for changing the original data. Do not obliterate or otherwise disfigure original data so as not to be readable (or equivalent for electronic data management).
- Transfer and reduce from field and laboratory records all data completely and accurately.
- Maintain all field and laboratory records in files according to *National Enforcement Investigation Center Policies and Procedures* (EPA 1991) (NEIC) guidelines.
- Organize data into a standard format for reporting purposes (i.e., testing, sampling, and analytical batch data report), as outlined in Sections 7 through 15, as applicable.
- Store all special processed records (e.g., electronic, optical, magnetic, and microfilm) in accordance with the QAPD requirements to ensure that waste container, sample, and associated QC data are readily retrievable (Section 1.7).

Data-generation-level review, validation, and verification are performed per the facility procedures and include signature release from qualified independent technical reviewer(s), technical supervisor(s), and the facility QA officer, as specified below. Facility personnel review, validate, and verify data using checklists that address all items included in this section. The completed checklists are provided with batch data reports (electronic or hard copy) to the SQAQO.

- One hundred percent 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 independent technical 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 percent check of all hand calculations.
 - The data were reviewed for transcription errors.
 - The testing, sampling, or analytical data QA is complete and includes raw data, reference to 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 (Sections 11 through 15).
 - Sample holding time and preservation requirements were met or exceptions documented.
 - Replicate observation of the radiography tapes were reviewed by a qualified technical reviewer, i.e., radiography operators performed replicate scans on one waste container per day or one per testing batch, whichever is less frequent; independent replicate scans were performed on one waste container per day or one per testing batch, whichever is less frequent; and radiography scans for 10 percent of the waste containers received supervisor review.
 - Field sampling records are complete and include the documentation specified in Section 6.1 of this QAPjP.
- One hundred percent of the data receives technical supervisory signature release for each testing, sampling, and analytical batch. This release ensures that the following:
 - The data are technically reasonable based on the technique used.
 - All data received independent technical review.

- The testing, sampling, or analytical data QA documentation is complete and includes raw data, calculation records, sample COC forms, reference to calibration records, QC sample results, and gas sample canister tags or documentation, 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.
- One hundred percent of data receive facility QA official (FQAO) signature release for each testing, sampling, and analytical batch. This release ensures that the following:
 - Independent technical and technical supervisor reviews were performed as evidenced by the appropriate signature releases.
 - The testing, sampling, or analytical data QA is complete as appropriate for the point of data generation.
 - Testing, 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 data package errors or omissions are identified, the FQAO and facility manager evaluate the nature of the problem and revise the data package as necessary. If they cannot rectify the problem by correcting the data package, they generate an NCR or CAR as described in Section 2.1.2. The facility manager and FQAO ensure the information is available in the data package so the next level data review may be completed. After data packages undergo data-generation-level review, validation, and verification, they are forwarded to the SQAQO along with the required signature releases and checklists.

3.1.2 Project-Level Data Validation

Data validation and verification at the project level involves scrutiny by and signature release from the SPM and SQAQO to ensure that minimum requirements are met for each waste container. If the SPM or SQAQO identify data package errors or omissions, they evaluate the nature of the problem and initiate revision of the data package, as necessary. If the SPM or SQAQO cannot rectify the problem by correcting the data package, they generate an NCR or CAR, as described in Section 2.1.2. "TRU Waste Project Level Data Validation and Verification" (WMH-400, Section 7.1.6) describes the project-level data validation, verification, and reporting process.

One hundred percent of the data receives SPM signature release, which ensures the following:

- Data-generation-level independent technical, technical supervisor, and FQAO review, validation, and verification were performed as evidenced by the appropriate signature releases.
- Testing, sampling, and analytical batch data review checklists are complete.

- Testing, sampling, and analytical 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).

One hundred percent of the data receives SQAQ signature release, which ensures the following:

- Sampling batch QC checks (e.g., equipment blank, field duplicate, field reference standard) were properly performed and meet the established QAQs (Sections 7 and 8).
- Testing batch QC checks (e.g., replicate scans, measurement system checks, replicate counts) were properly performed and meet the established QAQs (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 established QAQs (Sections 11 through 15).
- Proper procedures were followed to ensure that representative samples of headspace gas and homogeneous solids and soil/gravel were taken.
- Radiography data are complete and acceptable based on the video tape review of one waste container per day or one per testing batch (whichever is less frequent). For visually examined waste containers, a comparison with the radiography results has been performed and documented.
- NDA data are complete and acceptable.

In association with project-level data validation and verification, the SPM prepares a Data Validation Summary and the SQAQ prepares a Site Project QA Officer Summary for each batch data package, as described in WMH-400, Section 7.1.6. The Site Project QA Officer Summary includes, on a per-waste-container basis, a validation checklist for each testing, sampling, and analytical batch. Checklists for the Site Project QA Officer Summary are sufficiently detailed to validate all aspects of a testing, sampling, or analytical batch that affect data quality. The Data Validation Summary provides confirmation that, on a per-waste-container basis, all data have been validated in accordance with the QAPjP. The Data Validation Summary lists each testing, sampling, or analytical batch, describes how the validation was performed and whether or not problems were detected, and includes a statement indicating that all data are acceptable. The SPM and SQAQ shall ensure that a repeat of the data-generation-level review, validation, and verification is performed on the data for a minimum of one randomly chosen waste container quarterly (every 3 months).

In the case of analytical laboratory results, the laboratory will not dispose of samples until notification is received from the SPM. If the SPM requests that samples be retained for future use, the laboratory retains the samples under the same sample identification and chain-of-custody and documents the reason for the sample retention. Sample tags are removed and forwarded to the Project records custodian for filing as QA records.

3.2 VALIDATION METHODS

Data-generation-level QA officers validate data (qualitative and quantitative) to ensure that 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 volatile organic compound (VOC) data and total VOC, semivolatile organic compound (SVOC), and metals data. Quantitative determinations are calculated in accordance with Equations 3-1 through 3-8 below. Project personnel compare the quantitative determinations to the QAOs specified in Sections 11 through 15. Facility personnel use the validation methods described in Section 9 of the QAPP to demonstrate compliance with the QAOs for NDA. The SQAQ uses VE results to verify the matrix parameter category and waste material parameter weights determined by radiography, as described in Section 10.

Sampling personnel ensure sample representativeness through proper implementation of sampling procedures listed in Tables 7-4 and 8-1. Representativeness of waste containers from waste streams subject to VE 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 waste containers from within a waste stream were randomly selected (Section 5).

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:

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

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:

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

where s is the standard deviation and y is the mean of the replicate sample analyses.

The standard deviation, s , is calculated as follows:

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

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:

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

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:

$$\%R = \frac{C_m}{C_{srn}} * 100 \quad (3-5)$$

where C_m is the measured concentration value obtained by analyzing the sample and C_{srn} 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:

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

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 percent confidence that the analyte concentration is greater than zero. The MDL for all quantitative measurements is defined as follows:

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

where $t_{(n-1, 1-\alpha=.99)}$ is the t -distribution value appropriate to a 99 percent 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 Fourier Transform Infrared Spectroscopy (FTIRS), MDL is defined as $MDL = 3s$ where s is the standard deviation.

3.2.4 Completeness

Completeness is a measure of the amount of valid data (i.e., data that meet all QA/QC requirements) 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, is calculated as follows:

$$\%C = \frac{V}{n} * 100 \quad (3-8)$$

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 Project are comparable through the use of standardized approved testing, sampling, and analytical techniques, and by meeting the QAOs specified in Sections 7 through 15.

3.3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The SPM assesses whether data of sufficient type, quality, and quantity were collected and 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 waste containers was performed for the purposes of waste stream characterization. In association with the data validation and verification described above, the SPM is responsible for ensuring that all data reported meet the DQOs in Section 1.5.

For each waste stream characterized, the SPM 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 waste container is TRU waste
- Average concentration of hydrogen, methane, and each VOC in the headspace gas of waste containers in the waste stream
- Potential flammability of TRU waste headspace gases
- Mean concentrations, UCL₉₀ for the mean concentrations, and standard deviations for headspace gas VOCs included in the Resource Conservation and Recovery Act (RCRA) descriptions for F001, F002, F003, and F005 listed wastes

- Mean concentrations, UCL₉₀ for the mean concentrations, standard deviations, and number of solid samples collected for VOCs and SVOCs included in the RCRA descriptions for F001, F002, F003, F004, and F005; and the toxicity characteristic (TC) VOCs, SVOCs, and metals in the waste stream (if applicable)
- Whether the waste stream is listed for the presence of spent solvents under 40 CFR Part 261, Subpart D
- Whether the waste stream exhibits a TC under 40 CFR Part 261, Subpart C
- Whether the waste stream can be classified as RCRA hazardous waste or nonhazardous waste at the 90 percent confidence level
- Whether a sufficient number of waste containers were visually examined to determine with a reasonable level of certainty that the UCL₉₀ for the miscertification rate is less than 14 percent

“TRU Waste Characterization Data Quality Objectives Reconciliation and Reporting” (WMH-400, Section 7.1.1) implements the DQO reconciliation process. If the SPM determines that insufficient data were collected to make the determinations listed above, additional data are collected.

The SPM evaluates and reports waste characterization data from the analysis of homogeneous solids, soil/gravel, and debris waste streams following the statistical procedures presented in Section 5.0 of the QAPP. These procedures, which include UCL₉₀ calculations, are followed to assess compliance with the DQOs and are applied to all laboratory analytical data for headspace VOCs, total VOCs, total SVOCs, and total metals in samples of homogeneous solids and soil/gravel waste streams. The SPM verifies the assignment of EPA hazardous waste numbers for the presence of spent solvents by comparing data from analysis of appropriate headspace VOCs, total VOCs, and total SVOCs to the PRQLs in Tables 12-1, 13-1, and 14-1, and 40 CFR Part 261, Subpart D. The SPM determines the assignment of TC EPA hazardous waste numbers (40 CFR Part 261.24) by comparing AK information and data from the analysis of the appropriate metals and organic compounds to the regulatory threshold limit (RTL) values in 40 CFR Part 261, Subpart C and listed in Table 3-1. WMH-400, Section 7.1.1, describes this data evaluation process.

3.4 DATA REPORTING

Data-generation-level data reporting requirements are specific to the testing, sampling, or analytical methods used. Project-level data reporting requirements include waste container and waste stream information. Table 3-2 and Sections 7 through 15 list facility and project-level procedures for data reporting.

Table 3-1. Toxicity Characteristic Levels Expressed as Regulatory Threshold Limit 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
SVOCs^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

^aThe calculations assume 1) the maximum amount of material suggested by the toxicity characteristic leaching procedure is used; 2) wastes are 100 percent solid (no liquid fraction); 3) the maximum amount of extraction fluid is used; and 4) all analytes are 100 percent soluble in the extraction fluid.

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

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

RTL = regulatory threshold limit
SVOC = semivolatile organic compound
VOC = volatile organic compound

3.4.1 Data Generation-Level Reporting

Facility personnel assign identification numbers to samples (i.e., gas samples, homogeneous solids samples, and soil/gravel samples), as described in Section 6.2. Facility personnel assign a unique identification number to every sampling, testing, and analytical batch and every field and laboratory sample.

Facility personnel assign unique serial numbers to batch data reports and ensure that each page is numbered. The serial number used for data reports may be the same as the batch number. Facility personnel transmit an electronic or hard copy of all testing, sampling, and analytical batch data reports and data review checklists to the SPO. Facility 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. Facility procedures listed in Sections 7 through 15 identify the procedures that implement data-generation-level data reporting requirements.

The FQAO submits final sampling batch data reports and testing batch data reports to the Project records custodian within 28 days of sampling or testing the last waste container in the batch. Analytical personnel submit analytical batch data reports to the Project records custodian within 28 days of the validated time of sample receipt (VTSR) of the last sample in an analytical batch. Batch data report requirements for sampling, testing, and analytical methods are identified in Sections 7 and 8, 9 and 10, and 11 through 15, respectively. The batch data reports are maintained in the Project files in accordance with "TRU Records Management" (WMH-400, Section 1.5.1).

3.4.2 Project-Level Data Reporting

The SPM reports data on an individual waste container basis to CAO using the WIPP Waste Information System (WWIS), as specified in the WIPP WAC, and on a waste stream basis using a waste stream profile form (WSPF). The SPM submits the WSPF and the reconciliation with DQOs report to the CAO and the WIPP management and operating contractor.

If CAO requests hard-copy data reports, the SPM ensures that the data are compiled into a data package and submits the reports. The data package includes 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 relating sample numbers to waste container numbers; SQA 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, as applicable.

3.5 PROCEDURES PERTINENT TO THIS SECTION

Table 3-2 lists the project-level procedures pertinent to this section of the QAPjP.

Table 3-2. Section 3 Implementing Procedures.

Procedure number	Title
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 7.1.1	<i>TRU Waste Characterization Data Quality Objectives Reconciliation and Reporting</i>
WMH-400, Section 7.1.6	<i>Transuranic Waste Project Level Data Validation and Verification</i>

4. ACCEPTABLE KNOWLEDGE

Using acceptable knowledge (AK) at the Hanford Site, TRU waste is characterized at the waste stream level to take advantage of the most detailed information available. The methodology for delineating waste streams is described in HNF-2605, *Hanford Site Transuranic Waste Sampling Plan* (current version) (Sampling Plan). Waste streams are delineated based on the facility and process that generated the waste, physical form, and the presence or absence of RCRA-regulated hazardous constituents.

The Hanford Site uses AK information to delineate waste streams, assign matrix parameter categories, make hazardous waste determinations, identify waste material parameters, and determine radionuclides present in waste streams. The SPM is responsible for ensuring that the AK information is accurate, complete, and updated (as necessary) to reflect current waste characterization data. "Acceptable Knowledge Documentation Management" (WMH-400, Section 7.1.9) describes procedures for compliance with requirements for compiling, confirming, and controlling AK information.

Hanford Site personnel implementing these requirements receive training requisite to the AK requirements and associated implementing procedures, as described in "TRU Training and Qualification Plan" (WMH-400, Section 1.2.1).

4.1 QUALITY ASSURANCE OBJECTIVES

The Hanford Site uses AK documentation (e.g., records; management, procedural, and QC documents 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 TRU waste in compliance with the QAPP. This primarily qualitative information is assessed according to the QAOs established by CAO to ensure that DOE sites consistently apply the AK process. To demonstrate compliance with the QAOs (except for precision, which does not apply to AK), Project personnel:

- Measure accuracy based on the percentage of waste containers that require reassignment to a new matrix parameter category and/or designation of different EPA hazardous waste numbers upon reevaluation of AK using characterization data
- Ensure completeness by compiling 100 percent of the required Project information and TRU waste stream information described in Sections 4.3.1 and 4.3.2 for each waste stream
- Ensure comparability of data between sites by meeting training requirements and complying with the minimum standards outlined for procedures used to implement the AK process. The SPM assigns matrix parameter categories and EPA hazardous waste numbers and identifies waste material parameters and radionuclides. As this AK information is developed, it will be made available to other sites that store or generate similar waste streams.
- Ensure representativeness by obtaining, evaluating, and documenting AK information. Project personnel assess and document the limitations of the acceptable knowledge information used to assign matrix parameter categories and EPA hazardous waste numbers and to identify waste material parameters and radionuclides (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]).

The qualitative determinations of acceptable knowledge, such as compiling and assessing acceptable knowledge documentation, do not lend themselves to statistical evaluations of precision.

4.2 PROCEDURAL REQUIREMENTS

Project personnel compile the required AK documentation in an auditable record, confirm AK information using radiography, radioassay, and headspace gas and solidified waste sampling and analysis, and assess AK records for completeness.

4.2.1 Assembling Acceptable Knowledge Documentation

“Acceptable Knowledge Documentation Management” (WMH-400, Section 7.1.9) describes the process for assembling AK documentation. The SPM is responsible for ensuring that AK documentation:

- Is compiled in an auditable record and includes a roadmap for all applicable information
- Correlates specific waste stream information to the overview of the Hanford Site and Project operations in the context of the Hanford Site mission
- Describes correlations between waste streams with regard to the time of generation, waste generating processes, and Hanford Site facilities
- Includes a reference list that identifies documents, databases, QA protocols, and other sources of information that support AK information

The procedure also describes or provides the following:

- The specific methodology used to assemble AK 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 (e.g., 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 matrix parameter category to each waste stream
- The process for identifying the waste material parameters and radionuclides present in each waste stream
- The process for identifying hazardous wastes and assigning the appropriate EPA hazardous waste numbers to each waste stream
- The process used to ensure that unacceptable wastes are identified and segregated and that waste is certified for shipment to the WIPP facility
- The management controls used to ensure that nonconforming items are documented and managed

- A reference to radiography and VE procedures that list prohibited items that the operator verifies are not present in each waste container (i.e., liquids, sealed containers, pressurized containers, incompatible waste)
- The process for confirming AK in accordance with Section 4.4.3
- A cross-reference to the applicable matrix parameter summary category (i.e., S3000, S4000, and S5000) for each waste stream to verify that all required confirmation data have been evaluated and proper EPA hazardous waste numbers have been assigned
- The process for evaluating AK information and resolving any discrepancies in documentation
- For debris waste streams, the process for documenting changes to matrix parameter categories, waste stream assignment, and any associated EPA and state of Washington hazardous waste numbers based on material composition
- For newly generated waste streams, describe how AK information is confirmed VE prior to or during waste packaging. Radiography, rather than VE, may be performed where more practical.
- For all waste streams, the process for reevaluating AK information if radiography or VE results in the reassignment of a different matrix parameter category (e.g., plastic/rubber [S5310] versus paper/cloth [S5330])
- For all waste streams, the process for reassigning waste to a different waste stream and assigning appropriate EPA and state of Washington hazardous waste numbers

For any newly generated waste, the following minimum requirements are addressed in waste management procedures:

- Purpose and scope (i.e., waste streams)
- Responsible organization(s)
- Management controls
- Material inputs to process
- Process controls and range of operation that affect final matrix parameter category, waste material parameter, radionuclide, and hazardous waste determinations
- Rate and quantity of waste generated
- List of applicable operating procedures relevant to waste determinations
- Nonconformance reporting
- AK confirmation sampling (i.e., headspace gas sampling and/or solidified waste annual sample)
- Reporting and records management.

The SPM ensures that documentation is developed to demonstrate consistency in (a) assigning matrix parameter categories and EPA and state of Washington hazardous waste numbers, and (b) in determining waste material parameters and radionuclides; and to defend these assignments and determinations to independent auditors. WMH-400, Section 7.1.9, describes the following processes:

- Compiling all required information, including any AK information regarding the materials and processes that generate a specific waste stream, in an auditable record
- Reviewing the required information to determine the appropriate matrix parameter category
- 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 assigning all applicable listed EPA hazardous waste numbers
- Reviewing the required information to determine whether the waste may contain hazardous constituents that exhibit toxicity characteristics specified in 40 CFR Part 261, Subpart C. If an identified TC constituent is not included as a listed waste, the appropriate TC EPA hazardous waste number is assigned to the waste stream if AK documentation indicates that the concentration of the TC constituent exceeds the RTL listed in 40 CFR Part 261, Subpart C. Unless sufficient data are available to demonstrate that the concentration of the constituent in the waste is less than the TC RTL, the TC EPA hazardous waste number for the identified hazardous constituent is applied to the waste stream.
- Making no judgment regarding the quality of information in the case of discrepancies in information, ensuring that all applicable EPA hazardous waste numbers are assigned to the waste stream
- Identifying the specific waste containers of retrievably stored waste in each waste stream and correlating the waste stream to the mandatory AK information (Sections 4.3.1 and 4.3.2)

Currently, the Hanford Site does not generate any TRU waste that meets the definition as newly generated. In the future, however, some TRU waste may be newly generated during remediation activities. Samples of this waste may be collected as necessary (i.e., for homogeneous solid and soil/gravel waste) during its generation and the waste will be characterized in accordance with the applicable requirements. Repackaged retrievably stored waste may be managed as newly generated waste and characterized accordingly. The AK documentation for this waste will be developed in conjunction with the physical sampling. TRU Waste generated prior to obtaining certification will be managed as retrievably stored.

4.2.2 Confirmatory Testing

The SPM uses waste characterization results to confirm AK information in accordance with WMH-400, Section 7.1.9. Waste characterization methods used to confirm AK information are summarized below and described in Sections 7 through 15.

4.2.2.1 Radiography and Visual Examination

Radiography is performed on retrievably stored wastes to confirm the matrix parameter category. The SPM uses radiography and VE results to confirm the matrix parameter category and waste material parameters assigned using AK. The waste is repackaged if a prohibited item is found during radiography. VE of the waste may be performed during repackaging. If VE is performed, the SPM uses these results (rather than radiography) to confirm AK.

4.2.2.2 Sampling and Analysis

The SPM uses headspace gas data to confirm AK concerning the presence of hazardous constituents that may be F-listed solvents in all TRU waste. The SPM confirms the assignment of TC constituents and spent solvent EPA hazardous waste numbers (40 CFR Part 261, Subparts C and D, respectively) by evaluating the mean concentrations of each VOC detected in container headspace gas and/or the solidified waste matrix. The UCL₉₀ for the mean constituent concentration is compared to the PRQL for the constituent. If the UCL₉₀ for the mean constituent concentration is greater than or equal to the PRQL, the SPM reevaluates AK information and determines the potential source of the constituent.

If the source of the constituent is identified as a 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, the SPM either (a) assigns the applicable listed spent solvent EPA hazardous waste number to the entire waste stream, or (b) segregates the waste containers containing detectable concentrations of the solvent into a separate waste stream and assigns applicable EPA hazardous waste numbers.

The SPM confirms the EPA hazardous waste numbers associated with homogeneous solids and soil/gravel waste streams based on the results of the total VOC, SVOC, and metals analyses. If discrepancies between the results obtained from the solidified waste sampling and headspace gas sampling and analysis exist (e.g., a VOC is detected in the solidified waste but not in the headspace), Project personnel use the solidified waste data to confirm AK and assign applicable EPA hazardous waste numbers. Project personnel assign applicable TC EPA hazardous waste numbers (to debris wastes) based on the presence of hazardous constituents if sufficient information does not exist to demonstrate that the concentration of the TC constituent is less than the PRQL.

To determine the mean concentrations of solvent VOCs, Project personnel use all data of a given medium for a waste stream or waste stream 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, Project personnel use one-half the MDL to calculate the mean concentration. However, they omit the "U"-flagged concentrations that result from dilutions to calculate the mean for the purpose of assigning F-listed EPA hazardous waste numbers. They do not remove EPA hazardous waste numbers assigned using AK if hazardous waste constituents are not detected in the headspace.

Hanford TRU waste streams will be defined and characterized in accordance with WIPP requirements. The information developed in characterizing these waste streams will be used to ensure that it complies with both federal and state of Washington regulations. State of Washington and EPA hazardous waste numbers will be conservatively assigned to the wastes based on sampling and analysis and AK to ensure compliance with all applicable storage, handling, and labeling requirements. Only EPA hazardous waste numbers will be used to assess and ensure compliance to transportation and disposal requirements. For each facility participating in the Project, the RCRA permit status will be reviewed, as appropriate, to ensure that the designated operations are performed in compliance with the appropriate requirements.

4.3 ACCEPTABLE KNOWLEDGE INFORMATION

The SPM ensures that the required AK information listed in Sections 4.3.1 and 4.3.2 is included or referenced in the AK record. The AK record (i.e., auditable record) is compiled in accordance with WMH-400, Section 7.1.9, in such a way as to progress from general site information to facility-specific information, and includes supplemental information as necessary to complete the auditable record. The auditable record includes clearly defined waste characterization schemes and terminology and a breakdown of the types and quantities of TRU waste stored at Hanford. It also contains a reference list of all information sources and a road map identifying the specific physical location where the AK information is located. If mandatory information is not available for a particular waste, that waste will not be submitted for disposal at WIPP. Additional sampling and analytical information may be submitted in those cases to provide the required waste characterization information.

4.3.1 TRU Waste Management Project Information

WMH-400, Section 7.1.9, describes the compilation, updating, and management of AK documentation, which is maintained in an auditable record. The auditable record developed using this procedure provides an overall perspective of TRU waste management operations and serves as a guide to waste stream-specific information (Section 4.3.2). The TRU waste management Project information included in the auditable record clearly defines waste categorization schemes and terminology and provides a breakdown of the types and quantities of TRU waste stored at the Hanford Site. The auditable record includes 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 Hanford Site
- Waste identification or characterization schemes used at the facility(ies)
- Types and quantities of TRU waste generated
- Correlation of waste streams generated from the same building and process, as appropriate
- Waste certification procedures for wastes to be sent to the WIPP facility.

4.3.2 TRU Waste Stream Information

For each waste stream, the auditable record includes all process information and data that support the AK used to characterize that waste stream. At a minimum, the waste process information includes:

- Area(s) and building(s) from which the waste stream was generated
- Waste stream 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 waste stream and the physical waste form.

The auditable record includes a summary traceable to referenced documents to identify all information sources and the basis and rationale for defining each waste stream based on the parameters of interest. The auditable record also identifies and justifies any assumptions made in defining each waste stream.

4.3.3 Supplemental Acceptable Knowledge Documentation

The auditable record includes supplemental AK documentation for particular waste streams. This supplemental documentation may include, but is not 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
- Hanford Site databases
- Information from Hanford Site personnel
- Industry documents
- Previous analytical data relevant to the waste stream, including results from fingerprint analyses, spot-checks, or routine verification sampling
- Material Safety Data Sheets (MSDS), product labels, or other product package information
- Sampling and analysis data from comparable or surrogate waste streams
- Laboratory notebooks that detail the research processes and raw materials used in an experiment.

If supplemental documentation is included for a particular waste stream, the specific, relevant information is identified and justification is provided for its use (e.g., identification of a TC). If discrepancies exist between supplemental information and the required documentation, Project personnel assign applicable EPA hazardous waste numbers to the subject waste stream pending resolution of the discrepancy. Project personnel prioritize 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) is considered the least defensible. The pages from large documents (e.g., safety analysis reports) are flagged, or otherwise identified with the relevant information provided.

4.4 ACCEPTABLE KNOWLEDGE PROGRAM CONTROL

Project personnel responsible for assembling, evaluating, and assessing AK receive training and follow the procedure WMH-400, Section 7.1.9. Project personnel coordinate activities to segregate any waste container determined to be unacceptable and resolve any discrepancies in AK before the waste is shipped.

4.4.1 Training

Project personnel responsible for assessing information and resolving discrepancies associated with AK 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
- Matrix parameter category and waste material parameter designations
- The nonconformance process, including discrepancy resolution and reporting (see Section 2.1.2)
- Procedures related to waste characterization using AK.

4.4.2 Management Controls of Nonconforming Items

Nonconformances are managed as described in Section 2.1.2. Nonconformance procedures listed in Table 2-2 describe management controls used to ensure that nonconforming items or waste are segregated from certifiable populations. These procedures 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 (OJT) specific to management control procedures
- A stop work process in the event of noncompliance with management controls
- A nonconformance process (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

The SPM confirms AK through sampling, testing, and analytical results. WMH-400, Section 7.1.9, describes the procedures for confirming AK; maintaining additional records generated during confirmation; managing discrepancies identified during confirmation (e.g., how AK information is reevaluated, waste is reassigned to a different waste stream, appropriate EPA hazardous waste numbers assigned); and how additional information is incorporated into the auditable record. Project personnel track and document changes to waste determinations.

4.4.4 Resolution of Discrepancies

In the event of discrepancies in AK information, all relevant information will be included in the auditable record. If conflicting sources of information indicate that different hazardous wastes are present, Project personnel conservatively assign all appropriate hazardous waste numbers. The SPM ensures that waste with discrepancies in AK is segregated from certifiable populations. Project personnel resolve AK discrepancies prior to shipping waste by compiling and assessing additional information that represents a reasonable characterization of the waste and accurately describes the waste constituents and/or confirming AK through testing, sampling, and analysis of data.

4.4.5 Reevaluation of Acceptable Knowledge

Project personnel reevaluate AK, as described in WMH-400, Section 7.1.9. Project personnel take the following minimum steps if a waste must be assigned to a different matrix parameter category based on radiography or VE results:

- Review existing information based on the waste container identification number, and document all differences in EPA hazardous waste number assignments.
- Reassess and document all required AK information associated with the new designation if differences exist in the EPA hazardous waste numbers that were assigned.
- Reassess and document all sampling and analytical data associated with the waste.
- Reevaluate waste material parameter determinations, and document any changes.
- Reevaluate radionuclide content, and document any changes.
- Verify and document that the reassigned matrix parameter category 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 VE.
- Record all changes to AK records.
- If discrepancies exist in the AK information for the reassigned matrix parameter category, Project personnel complete an NCR, document the segregation of the waste container, and define the corrective actions necessary to fully characterize the waste.

4.5 AUDITS

The SQAQO evaluates the AK process and waste stream documentation through independent assessments (audits or process surveillances). For these assessments, the SQAQO assembles a team knowledgeable of RCRA regulations and EPA guidance regarding the use of AK for waste characterization, hazardous waste determinations, matrix parameter categories, waste material parameters, and QAPP requirements. Team members are independent of the TRU waste management operations being assessed. Independent assessments of AK include the following elements:

- Availability and implementation of documentation of the process used to compile, evaluate, and record AK
- Documentation of personnel training and qualifications

- Compilation of all required AK documentation in an auditable record
- Existence of a procedure and records for assigning EPA hazardous waste numbers to waste streams
- Existence of a procedure and records for assigning a matrix parameter category to a waste stream
- Existence of a procedure for determining waste material parameters present in a waste stream
- Existence of a procedure for determining the radionuclides present in a waste stream
- Existence of a procedure for resolving inconsistencies in AK documentation
- Existence of a procedure for confirming AK information through: (a) radiography or VE, (b) headspace gas sampling and analysis, and (c) solidified waste sampling and analysis
- Availability of results of other audits or assessments of the TRU waste characterization Project at the facility.

Team members will evaluate all AK documentation associated with at least one debris and at least one solidified waste stream and will evaluate compliance with written site procedures for developing the AK. Team members will evaluate the logic and defensibility of the AK 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 waste streams and the basis for making waste determinations and changes in waste stream information. They will verify that sites include all required information and conservatively include all potential EPA hazardous waste numbers indicated by the AK documentation. They will report all deficiencies found in the AK documentation to the SQAQ.

4.6 PROCEDURES PERTINENT TO THIS SECTION

Table 4-1 lists the procedures pertinent to this section of the QAPjP.

Table 4-1. Section 4 Implementing Procedures

Document number	Title
WMH-400, Section 1.2.1	<i>TRU Training and Qualification Plan</i>
WMH-400, Section 1.3.2	<i>TRU Nonconforming Item Reporting and Control</i>
WMH 400, Section 1.3.3	<i>TRU Corrective Action Reporting and Control</i>
WMH-400, Section 7.1.9	<i>Acceptable Knowledge Documentation Management</i>

5. SAMPLING PROCESS DESIGN

Project personnel implement the sampling process design described in Section 5.0 of the QAPP for retrievably stored wastes, and define waste streams based on AK for purposes of making RCRA hazardous waste determinations. Project personnel assess all waste containers in all matrix parameter categories (summary categories S3000, S4000, and S5000) for the presence of spent solvents based on headspace gas analytical results. A randomly selected portion of waste containers in homogeneous solids and soil/gravel waste streams (summary categories S3000 and S4000, respectively) are sampled and analyzed for total VOCs, SVOCs, and metals for RCRA characterization. Waste containers are randomly selected from all matrix parameter categories annually for VE to confirm the results of radiography. These processes are described in the following sections.

5.1 DESCRIPTION OF ACCEPTABLE MATRIX PARAMETER CATEGORIES

Using AK, Project personnel define waste streams and assign waste containers to waste streams. They assign each waste stream a matrix parameter category based on the physical and chemical composition of the waste consistent with waste stream designations in the TWBIR and matrix parameter categories in the *DOE Waste Treatability Group Guidance* (DOE 1995a). The matrix parameter categories are divided into three broad groups: homogeneous solids (summary category S3000), soil/gravel (summary category S4000), and debris wastes (summary category S5000). The appropriate matrix parameter category will be entered on all applicable waste characterization data forms (e.g., analytical batch data reports, NDE and VE testing reports). Sampling and analysis activities are based on the waste stream summary category, as described in Section 5.2. The Sampling Plan describes the process for defining and documenting waste streams and associated matrix parameter categories using AK and the characterization strategy using testing, sampling and analysis, and AK documentation.

5.2 PARAMETERS, RATIONALE, AND TEST METHODS

Once a waste stream is identified, Project personnel obtain the required waste characterization information. Project personnel characterize all waste by NDA (Section 9), radiography and/or VE (Section 10), and headspace gas sampling and analysis (Sections 7, 11, and 12). Project personnel select waste containers of homogeneous solids and soil/gravel for sampling and analysis (Sections 8 and 13 through 15), as described in Section 5.4.1, and for VE (Section 10), as described in Section 5.4.2. All existing wastes will be characterized as retrievably stored, except for repackaged debris waste, which may be characterized as newly generated (i.e., not required to undergo radiography).

5.2.1 Homogeneous Solids and Soil/Gravel

Sampling personnel obtain samples of homogeneous solids (summary category S3000) and soil/gravel (summary category S4000), as described in Section 8. Waste containers to be sampled and sampling locations within those containers will be identified on a statistically random basis, as described in Section 5.4.1. These samples are analyzed for total RCRA-regulated VOCs, SVOCs, and metals, as described in Sections 13, 14, and 15. Transformer oils containing polychlorinated biphenyls (PCBs) have been identified in a limited number of waste streams included in organic sludges (matrix parameter category S3220). Therefore, only waste streams included in the organic sludges matrix parameter category must be analyzed for PCBs.

5.2.2 Debris Wastes

Project personnel characterize debris wastes for RCRA-regulated VOCs, SVOCs, and metals based on AK. Section 4 describes how Project personnel document and verify AK information for waste streams.

5.3 CONFIRMATION OF EPA HAZARDOUS WASTE NUMBERS FOR SPENT SOLVENTS

The SPM evaluates headspace gas VOC analytical results from all TRU waste drums for each waste stream to confirm and update EPA hazardous waste numbers for spent solvents (i.e., F001, F002, F003, F005). A statistical method is used to determine whether spent solvent EPA hazardous waste numbers should be added to a waste stream. The UCL_{90} (equations 5-1 through 5-3 of the QAPP) for the mean concentration of each spent solvent constituent is compared to the PRQL (listed in Table 12-1) for the evaluation. If the UCL_{90} for the mean is equal to or greater than the PRQL, the SPM assigns the appropriate EPA hazardous waste number to the waste stream if it has not already been assigned based on AK. The spent solvent hazardous waste number is not assigned if definitive AK information indicates that the listed constituent was not used for its solvent properties and does not otherwise meet the criteria for hazardous wastes from nonspecific sources (40 CFR Part 261, Subpart D). If the UCL_{90} for the mean is less than the PRQL, the assignment of spent solvent EPA hazardous waste numbers remains unchanged. "Sampling Design and Data Analysis for RCRA Characterization and Visual Examination of Retrievably Stored Transuranic Waste" (WMH-400, Section 7.1.4) describes the process used to evaluate and document waste stream RCRA characterization.

The statistical tests described above are based on the assumption that the measured concentrations of each contaminant are normally distributed. Data are assessed for normality using the Shapiro-Wilk (or *W*) test or equivalent for goodness of fit. The Shapiro-Wilk test is a least squares regression of the data. This assumption of normal distribution is best verified by comparing the fit of the untransformed data to the fit after certain transformations. Data transformations may include, but are not limited to, square, cube, square root, logarithmic, natural logarithmic, reciprocal root, reciprocal, reciprocal square, or reciprocal cube. If a transformation is required to achieve normality, the transformed PRQL will also be calculated. The tests will then be performed the same as before, with the transformed data and PRQL being substituted into the equations. If 30 or more samples have been collected from the waste stream or waste stream lot, the Central Limit Theorem (CLT) may be applied, the distribution assumed to be approximately normal, and a transformation unnecessary. WMH-400, Section 7.1.4, addresses testing for normal distribution, transformation of data to normality (if needed), and treatment of less-than-detectable analytical results.

5.4 SAMPLING PLAN

Representativeness expresses the degree to which a sample or group of samples represents the population being studied and, for the Project, is based on the random selection of waste containers 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 the population of interest. The SPM is also responsible for reviewing and approving HNF-2605, *Hanford Site Transuranic Waste Sampling Plan* (Sampling Plan).

The sampling plan describes the statistical approach used to determine the number of waste containers to select for characterization. The sampling plan describes the strategies for obtaining a preliminary estimate of the coefficient of variation (CV) and randomly selecting waste containers of homogeneous solids and soil/gravel for final waste stream characterization. The sampling plan includes the following information:

- Identification of site name as Hanford Site
- Waste to which the sampling plan applies (i.e., newly generated TRU waste and retrievably stored 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 VE)
- 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 the 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 whether 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 VE, as described in Section 5.4.2
- Description or citation of procedures for interfacing with operations personnel regarding retrieval of selected containers

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 of a waste stream may be characterized as a waste stream lot.

Statistical process control techniques and verification sampling will be used to support AK in characterizing newly generated homogeneous solids and soil/gravel wastes. Section 5.4.3 describes sampling and analysis of newly generated wastes.

5.4.1 Resource Conservation and Recovery Act Characterization of Homogeneous Solids and Soil/Gravel

The Project approach for RCRA characterization relies on AK to initially categorize waste containers of homogeneous solids and soil/gravel into waste streams and characterization on a waste stream rather than individual container basis. Once segregated by waste stream, Project personnel randomly select and sample waste containers for RCRA characterization. The SPM is responsible for verifying that the samples collected from within a waste stream are randomly selected. Project personnel sample and analyze

a minimum of five containers in each waste stream. Where fewer than five containers of waste exist for sampling, multiple samples will be randomly selected from the containers, as necessary, to ensure that a minimum of five samples are obtained to characterize the waste. Preliminary estimates of the mean concentration and variance for each RCRA-regulated TC contaminant in the waste are determined from preliminary sampling and analysis of the waste stream or from previous sampling and analysis of the waste stream. Preliminary estimates of the CV for each contaminant are calculated as the ratio of the standard deviation to the mean, as described in Section 5.4.1 of the QAPP, and the highest CV is used to determine the number of samples to collect and analyze for RCRA characterization for 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 waste containers sampled is dependent on defined levels of acceptable error for the hazardous versus nonhazardous determination and is calculated using equations 5-6 through 5-9 in Section 5.4.1 of the QAPP. WMH-400, Section 7.1.4, describes the process for determining the estimates and number of containers to sample.

The statistical tests described above are based on the assumption that the measured concentrations of each contaminant are normally distributed. This assumption is best verified by comparing the fit of the untransformed data to the fit after certain transformations using the Shapiro-Wilk or equivalent statistical test, as described in Section 5.4.1 of the QAPP. If a transformation is required to achieve normality, the transformed PRQL will also be calculated. The tests will then be performed the same as before, with the transformed data and PRQL being substituted into the equations. If 30 or more samples have been collected from the waste stream or waste stream lot, the Central Limit Theorem may be applied, the distribution assumed to be approximately normal, and a transformation unnecessary. Actual numeric values for each analyte will be reported, if possible, whether or not the measurement is above or below the MDL. For those analyte concentrations reported as less-than-detectable, a suitable substitution (e.g. one-half the MDL) will be made in accordance with Section 5.4.1 of the QAPP. WMH-400, Section 7.1.4 addresses transformation of data to normality (if needed) and treatment of less-than-detectable analytical results.

Project personnel randomly select waste containers from each waste stream as described in WMH-400, Section 7.1.4. The SPM is responsible for verifying that the samples collected within a waste stream were selected randomly. Upon completion of the required sampling and analysis, the final mean, variance, and CV for each contaminant are determined. The observed CV is then compared to 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, the required number of samples is recomputed using the observed CV. Additional waste containers are randomly selected for sampling if the recomputed number of required samples is greater by 20 percent than the number of samples collected.

When sampling and analysis is completed for a waste stream, the UCL_{90} for the mean concentration of each contaminant is calculated using equation 5-10 in Section 5.4.1 of the QAPP. WMH-400, Section 7.1.4, references the QAPP for descriptions of applicable calculations used for the RCRA characterization of waste streams. The procedure also describes the treatment of data transformations (if necessary) and less-than-detectable analytical results.

First, the SPM uses the results to confirm AK concerning the presence of hazardous constituents that may be F-listed solvents. The SPM assigns appropriate spent solvent EPA hazardous waste numbers (F001, F002, F003, F004, F005) to the waste stream if the UCL_{90} for the mean concentration of a constituent used as a solvent is greater than or equal to the PRQLs in Tables 13-1 and 14-1 for spent solvents. The spent solvent hazardous waste number is not assigned if definitive AK information indicates that the listed constituent was not used for its solvent properties and does not otherwise meet the criteria for hazardous wastes from nonspecific sources (40 CFR Part 261, Subpart D). If the UCL_{90} for the mean concentration is less than the PRQLs for the spent solvents, the SPM makes no changes to the EPA hazardous waste number assignment.

Second, the SPM uses the results to confirm AK concerning the presence of hazardous constituents that may be TC wastes. The SPM assigns applicable TC EPA hazardous waste numbers to the waste stream if the UCL_{90} for the mean contaminant concentration is greater than or equal to the RTLs (Table 3-1). For constituents used only as solvents, the SPM assigns a TC EPA hazardous waste number only if a spent solvent EPA hazardous waste number has not already been assigned for this same contaminant. If the UCL_{90} for the mean contaminant concentration is less than the RTL, no TC hazardous waste codes for the constituents need be applied, and no additional information needs to be supplied in the AK record.

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

As a QC check on radiography and matrix parameter category assignments and material parameter weight estimates made during radiography, facility personnel visually examine a statistically selected portion of the waste containers that are expected to be certified in a 12-month period. WMH-400, Section 7.1.4, describes how waste containers are selected for VE. Each year, the number of waste containers to be visually examined is determined in accordance with the sampling plan (Hanford 1998) based on the number of waste containers that are expected to be certified and the previous year's miscertification rate.

Miscertified containers are those that radiography indicates meet the WIPP WAC radiography-determined and *TRUPACT-II Authorized Methods for Payload Control* (Appendix 1.3.7 of NRC 1997) requirements, but VE indicates do not meet these requirements. During the first year of Project activities, a miscertification rate of 2 percent will be assumed based on historical evidence from the Idaho National Engineering and Environmental Laboratory. In succeeding years, the miscertification rate will be determined each year based on the results of certification activities over the preceding 12 months. The following assumptions are used to determine the number of containers for VE:

- Waste containers are randomly selected and examined to ensure that a representative sample of waste containers is obtained.
- Only waste containers certified for compliance with WIPP WAC and TRAMPAC will be selected.
- There is a definable finite population of waste containers for which the proportion miscertified is to be estimated.
- The certification process is uniform for all waste containers and is unbiased regardless of the waste stream.
- The radiography system is functioning properly and is operated by qualified personnel.

The number of containers to be visually examined for each miscertification rate is presented in Section 4.2 of the sampling plan (Hanford 1998), or may be calculated using the information in Section 5.4.2 and Appendix A of the QAPP.

At a minimum, enough waste containers will be visually examined to achieve the level of confidence required by the QAPP. The replacement strategy cited in WMH-400, Section 7.1.4, for waste containers undergoing homogeneous solids and soil/gravel sampling operations may also be used to satisfy the VE requirement. This strategy allows waste containers randomly selected for sampling to replace waste containers randomly selected for VE from the same waste stream or waste stream lot, provided that QAPP Section 5.4.2 limitations are observed.

When debris waste must be repackaged (e.g., to comply with the WIPP WAC), Project personnel may either perform VE (and thus, confirm AK and conformance with applicable WIPP WAC) during repackaging or conduct radiography on repackaged wastes. VE requirements for all wastes are described in "Transuranic Waste Repackaging, Visual Examination, and Sampling" (WMH-400, Section 7.1.3).

5.4.3 Characterization of Newly Generated Homogeneous Solids, Soil/Gravel, and Debris Wastes

Because processes at Hanford do not currently result in the continuous generation of any newly generated homogeneous solids or soil/gravel waste, the requirements of QAPP Section 5.4.3 currently do not apply. Newly generated debris waste streams will be characterized using AK, in accordance with Section 4.0. Spent solvent EPA hazardous waste numbers will be applied to these debris waste streams on the basis of this AK and be updated using headspace gas sampling results, as described in Section 5.3.

5.5 PROCEDURES PERTINENT TO THIS SECTION

Table 5-1 lists the implementing documents pertinent to this section of the QAPjP.

Table 5-1. Section 5 Implementing Procedures.

Document number	Title
HNF-2605	<i>Hanford Site Transuranic Waste Sampling Plan</i>
WMH-400, Section 7.1.3	<i>Transuranic Waste Repackaging, Visual Examination, and Sampling</i>
WMH-400, Section 7.1.4	<i>Sampling Design and Data Analysis for RCRA Characterization and Visual Examination of Retrievably Stored Transuranic Waste</i>

6. SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Project personnel implement sample handling and custody practices and requirements described in this QAPjP to ensure that Project data meet accepted standards for legal admissibility and defensibility. Facility-specific sample handling and custody procedures comply with the QAPP requirements and EPA guidelines, as prescribed by the National Enforcement Investigation Center (NEIC). Procedures contain examples of the forms (or describe the electronic equivalents) used to implement the requirements of this section.

6.1 FIELD DOCUMENTATION

Sampling personnel record information pertinent to field sampling in reproducible, permanent ink or in Hanford Site-approved electronic format. The individual making a record entry dates and signs the record. Figure 6-1 provides an example field sampling data sheet. The procedures identified in Table 6-3 describe field documentation procedures. At a minimum, sampling personnel record the following pertinent information:

- Sample type/description
- Sampling facility
- Waste container identification number
- Sample identification number (referenced to waste container 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 preservation method and preservatives (e.g., HCl, 4°C)
- Analysis requested
- QC designation, if applicable (e.g., equipment blank, field reference standard)
- Chain of custody (COC) documentation number
- Analytical laboratory to perform the analysis
- Shipping information
- Sampler's name
- Sampling plan/standard operating 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, or equivalent canisters, sampling personnel also record the following information:

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

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

- Sampling tool identification
- Randomly selected coring and sampling location
- Depth of waste and depth of sample
- Visual observations of waste and recovered sample.

These records are maintained in accordance with WMH-400, Section 1.5.1.

6.2 LABELING

Sampling personnel label samples to meet the requirements specified in the QAPP. "TRU Waste Sample and Waste Container Management Activities" (WMH-400, Section 7.1.7) describes the process for labeling samples.

6.2.1 Waste Container Identification Numbers

All stored waste containers are identified with a unique identification number, which is affixed to, inscribed on, or otherwise attached to the waste container. This identification number is recorded on related COC documents in order to trace the sample and data to the original waste container. If, during waste characterization activities, a portion of the waste is transferred from the original waste container to a new waste container, Project personnel assign a unique identification number to the new waste container.

6.2.2 Headspace Gas Sample Identification Number

Each SUMMA[®] canister, or equivalent canister, has a unique identification number. At the time of sample collection, sampling personnel assign a 13-digit sample identification number in the following format:

<u>RL</u>	<u>MMDDYY</u>	<u>#####</u>
Hanford sampling site	Date	Canister identification

where:

RL	=	Site identifier
MMDDYY	=	the month, day, and year of sampling
#####	=	a unique, sequential five-digit number.

6.2.3 Headspace Gas Sample Canister Tag

"Headspace Gas Sampling of TRU Waste Containers" (LO-080-404) includes instructions for completing the sample canister tags (or equivalent documentation). Figure 6-3 is an example of a sample canister tag. Analytical laboratory personnel document the canister pressure (canister gauge reading) for each canister after cleaning the canister. They record this information in reproducible, permanent ink either on a canister tag that is securely fastened to the canister before shipment to the field or on equivalent documentation traceable to the canister.

During sampling operations, sampling personnel record the following information in reproducible, permanent ink on the canister tag (or equivalent documentation):

- Sample identification number
- Sampler's name
- Ambient temperature (°C) and pressure (mm Hg)
- Canister gauge reading in the field (and manifold pressure, if applicable) before and after sampling
- Sampling organization
- Sample description
- Comments
- Requested analyses
- Date and time of sample collection
- Designation of whether the sample is a blank
- Waste container identification number.

Sample ID No:			Sampler's Name (print):		
Waste Container ID No:			Sampler's Signature:		
COC Documentation No:			Sampling Organization:		
Sampling Date _____ Time: _____			QC Designation:		
Sample Description:			Sample Canister No.:		
Location	C/M ¹	P/T ²	Date	Time	Initials
Certifying Laboratory—after cleaning (attach separate form if necessary)					
Field—before sample collection					
Field—at time of sample collection					
Field—after sample collection					
Analytical Laboratory—before sample analysis					
¹ C = canister pressure gauge reading (psig); M = sampling manifold pressure gauge reading (mm Hg) ² P = pressure (mm Hg); T = temperature (°C); ambient conditions					
Analyses Requested:					
Comments (objective, factual observations pertinent to the sampling activity):					

**Figure 6-1. Field Sampling Data Sheet
(EXAMPLE ONLY)**

Analytical personnel record canister pressure and ambient pressure and temperature within 24 hours of the VTSR. Canisters are thermally equilibrated to ambient temperature before these measurements are made. The individual responsible for this entry records the date, time, and initials. After sample analysis, analytical personnel provide the sample canister tag (or equivalent documentation) to the Project records custodian in accordance with WMH-400, Section 7.1.6, and WMH-400, Section 1.5.1.

6.2.4 Homogeneous Solids and Soil/Gravel Sample Identification Number

Sampling personnel assign a 12-digit sample identification number to each sample of homogeneous solids or soil/gravel collected. The sample identification number is in the following format:

RL-XXXXX-XX-L-WP

where:

RL	=	Site identifier
XXXXX	=	5-digit sequential number
XX	=	bottle type
L	=	Lab
WP	=	Data qualifier

Sampling personnel label each sample container before shipping the sample to the analytical laboratory. An example of the sample container label (or description of the electronic system) is provided in WMH-400, Section 7.1.7. Sampling personnel ensure that the following information is recorded on the sample container label in reproducible permanent ink:

- Applicable waste container identification number
- Sample identification number
- Analyses requested
- COC documentation number
- Time and date of sample collection
- Type and number of sample containers
- Sample preservatives
- Sampler's name
- Comments.

6.3 CHAIN OF CUSTODY

Project personnel track waste containers and initiate and maintain sample COC to meet QAPP requirements. Tracking and COC activities are described in the WMH-400, Section 7.1.7, and facilities' operating procedures. Analytical laboratory procedures describe the tracking and COC procedures for samples received for analysis. Sampling personnel initiate sample COC at the time of sample generation and maintain sample COC until a transfer of custody takes place. Analytical personnel maintain sample COC until analysis is complete and direction from the SPM is received to release the sample, and the sample is removed from the Project.

A sample is considered under effective custody control if it is sealed (i.e., unopened) with the custody seal or tamper-indicating device 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 is in the possession of an authorized individual and access to the sample is controlled by locking or placement of a tamper-indicating device or signed custody seal that prevent undetected access.
- It is in a designated secure area (e.g., a controlled access location) with complete documentation of personnel access, or a radiological containment area (hot cell or glove box).

Sampling personnel and analytical personnel record sample COC on a sample COC form (Figure 6-2) or electronic equivalent. Before a transfer of custody takes place, the receiving custodian inspects the custody form and all accompanying documentation (e.g., custody seals, sample tags, shipping forms) to ensure that 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 samples for signs of damage or tampering. Any discrepancy in information or sign of damage or tampering is documented on custody documentation (e.g., COC form, tags, labels) or on receiving checklists (or electronic equivalent) by the receiving custodian. To transfer custody, both parties sign and date a COC form (or electronic equivalent) and the relinquishing party retains a copy of the form. An NCR or CAR may be initiated if discrepancies cannot be resolved, omitted information is unrecoverable, or in cases of repeated documentation problems. COC documentation is maintained in Project files in accordance with WMH-400, Section 1.5.1. Analytical personnel use the comment field to describe sample container disposition.

6.4 WASTE CONTAINER AND SAMPLE HANDLING

Project procedures describe waste container and sample management. Analytical laboratory procedures describe sample management at the analytical facility. These procedures (listed in Table 6-3) include sample preservation, sample holding times, and waste container and sample tracking.

6.4.1 Waste Container Sampling

Sampling personnel allow waste containers and their contents to equilibrate to the ambient temperature of the sampling facility for at least 72 hours before headspace gas sampling. To ensure that waste characterization data collected represent conditions in the WIPP repository, waste containers are sampled at a temperature range of $>18^{\circ}\text{C}$ (65°F). Sampling personnel also ensure that waste containers meet applicable drum age criteria prior to sampling.

6.4.2 Gas Samples

Sampling personnel promptly transfer gas samples collected in SUMMA[®] canisters, or equivalent canisters, to the analytical personnel, ensuring that sample holding times and storage conditions meet the requirements specified in Table 6-1. Sampling and analytical personnel adhere to an overall holding time of 34 days (4 days of field holding time plus 2 days of transfer shipping allowance plus 28 days of laboratory holding time) and maintain headspace samples between 0 and 40°C (32 and 104°F).

Sampling Facility: _____				
Collection Date: _____				
Collection Time: _____				
Waste Container No.: _____			COC No.: _____	
Sampling Personnel (print name[s]): _____				
Sampling Personnel Signature(s): _____				
Sample Type: ☐ Headspace Gas ☐ Homogeneous Solid ☐ Soil/Gravel				
Analytical Laboratory:			Carrier:	
Sample ID No.	Collection Date/Time	Sample Matrix	Type/No. of Containers	Preservative
Relinquished by (name/organization)	Received by (name/organization)	Date	Time	
Comments (objective, factual observations pertinent to the sampling activity):				
Disposition of final sample:				
Sample removed from custody:				
Signature:				

**Figure 6-2. Sample Chain-of-Custody Form.
(EXAMPLE ONLY)**

Figure 6-3. Sample Headspace Gas Sample Canister Tag (Example Only)

Notebook	HNF-N-144	rev 1	page 23	Item 02
Canister	AS481		Analysis	CH4/H2
Sampling Date	_____		Collector	_____
Sampling Time	_____		Sample batch	_____
Drum number	9601929			
Programmatic number	Chain of custody No.		W9109-h	
RL-981125-AS481.9601929				

Cleaning and Analysis Information

SAS SUMMA Canister Number _____

Evacuated	Pressurized	Evacuated	Pressurized
	Date	Initials	Comments
Cleaning			
QC Check			
Analysis			

Sampling personnel load SUMMA[®] canisters, or equivalent canisters, of gas samples into a shipping container and affix a tamper-indicating device or a signed and dated custody seal across the lid and body of the shipping container to provide visual evidence of tampering.

6.4.3 Homogeneous Solids and Soil/Gravel Samples

Project personnel manage samples of homogeneous solids and soil/gravel in accordance with the requirements for preservation and holding time specified in Table 6-2. Sampling procedures describe sample quantities and containers. Sampling and sample handling personnel ensure that samples shipped to the laboratory are maintained at a temperature of 4°C ($\pm 2^\circ\text{C}$) from the time of collection through sample transport to the laboratory.

Before shipping samples to the laboratory, 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. WMH-400, Section 7.1.7, describes the process for shipping and tracking samples of homogeneous solids and soil/gravel samples. Sampling personnel place the COC form in a waterproof plastic bag taped to the shipping container and affix a signed and dated custody seal or tamper-indicating device to the shipping container. Sampling procedures provide example custody seals and accompanying documentation format. If more than one shipping container is used, documentation is placed on each shipping container.

Sample tracking is initiated at the time of sample collection, in accordance with WMH-400, Section 7.1.7. The status of the sample and its associated data will be tracked by the SPM using the sample identification number. Analytical laboratories will provide a weekly status report to the SPM containing the following information, at a minimum, for each Project sample under their control: sample identification number, date of collection, date received by the laboratory, date the analysis was completed, and date data validation was completed.

6.4.4 Laboratory Chain-of-Custody

Sample chain of custody is initiated at the time of sample collection, in accordance with WMH-400, Section 7.1.7. Sample custody is maintained until the associated analyses are completed and direction has been received from the SPM to release the samples. Sample custody is maintained until the sample is expended or until the sample is removed from the Project. The SPM may request that samples be maintained for archive purposes; in these cases, sample custody is maintained in accordance with WMH-400, Section 7.1.7.

Table 6-1. Headspace Gas Holding Conditions and Times.

Parameter	Container	Minimum waste container headspace sample volume ^a	Holding temperature	Field holding time ^b	Shipping allowance	Laboratory holding time ^c
H ₂ , CH ₄	SUMMA canister, or equivalent canister	100 mL	0-40°C	4 days	2 days	28 days
VOCs	SUMMA canister, or equivalent canister	250 ml	0-40°C	4 days	2 days	28 days

^aAlternatively, if available headspace is limited, a single 100-mL sample may be collected for determination of VOCs, H₂, and CH₄.

^bFrom time of headspace sample collection to shipment.

^cProgrammatic-based maximum holding time. Holding time begins at validated time of sample receipt.

CH₄ = methane
H₂ = hydrogen
VOC = volatile organic compound

Table 6-2. Sample Handling Requirements for Homogeneous Solids and Soil/Gravel.

Parameter	Required preservative	Maximum holding time ^a
VOCs	Cool to 4°C (±2°C)	14 days prep/40 days analyze ^b
SVOCs	Cool to 4°C (±2°C)	14 days prep/40 days analyze
Metals	Cool to 4°C (±2°C)	180 days ^c
PCBs	Cool to 4°C (±2°C)	14 days prep/40 days analyze

^aHolding time begins at sample collection (holding times are consistent with SW-846 requirements [EPA 1995]).

^b40-day holding time allowable only for methanol extract—14-day holding time for nonextracted VOCs.

^cHolding time for mercury analysis is 28 days.

SVOC = semivolatile organic compound
VOC = volatile organic compound
PCBs = polychlorinated biphenyls

Laboratories participating in the Project have procedures for sample receiving and log-in, storage and numbering, tracking within the laboratory, and storage of data. These procedures include the following:

- Sample numbering
- Identification of the sample custodian
- Internal sample numbering and tracking
- Custody transfers within the laboratory
- Example custody forms and instructions for use
- Sample storage and disposal
- Custody and maintenance of analytical data.

6.5 PROCEDURES PERTINENT TO THIS SECTION

Table 6-3 lists the implementing procedures pertinent to this section of the QAPjP.

Table 6-3. Section 6 Implementing Procedures

Document number	Title
LO-080-404	<i>Headspace Gas Sampling of TRU Waste Containers</i>
DO-080-009	<i>Obtain Headspace Gas Samples of TRU Waste Containers</i>
LO-090-450	<i>Sample Chain of Custody Acceptance and Disposal</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH 400, Section 7.1.1	<i>TRU Waste Characterization Data Quality Objectives Reconciliation and Reporting</i>
WMH-400, Section 7.1.7	<i>Transuranic Waste Sample and Waste Container Management Activities</i>

7. HEADSPACE GAS SAMPLING

Sampling personnel collect headspace gas samples from the headspace directly under the drum lid of all TRU waste drums. They collect the samples in SUMMA[®] canisters, or equivalent canisters, using the manifold or direct canister method or use an on-line integrated sampling/analysis system. To ensure proper representation, they collect samples 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. "Headspace Gas Sampling of TRU Waste Containers" (LO-080-404) details headspace gas sampling activities. Containers are also allowed to equilibrate to the ambient temperature ($>18^{\circ}\text{C}$ [65°F]) of the sampling facility for at least 72 hours before headspace gas sampling.

7.1 QUALITY ASSURANCE OBJECTIVES

Sampling personnel collect field QC headspace gas samples for analysis at the frequency specified in Section 7.3 to demonstrate that QAOs have been met. The QC headspace gas samples include equipment blanks, field reference standards, field blanks, and field duplicates. Table 7-1 lists the required analytes for each type of headspace gas sample collected.

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	X	X
Field blank	X	—
Equipment blank	X	—
Field reference standards	X	X

^aSee Table 12-1 for a list of VOCs.

CH ₄	=	methane
H ₂	=	hydrogen
VOC	=	volatile organic compound

Sampling personnel with the facility QA officer prepare, submit, and resolve an NCR if final, reported QC sample results do not meet acceptance criteria. LO-080-404 and "Data Management for Headspace Gas Samples" (WMH-400, Section 8.1.8) identify the methods used to demonstrate compliance with the QAOs. In accordance with LO-080-404, and WMH-400, Section 8.1.8, sampling personnel:

- Collect field duplicates simultaneously into SUMMA[®] canisters, or equivalent canisters, manually or by use of a sequential manifold, or sequentially using an on-line integrated sampling/analysis system for determination of VOCs, hydrogen, and methane to assess the precision of headspace gas sampling and analysis. Project personnel calculate the RPD for the canister field duplicates and initiate corrective action if the RPD exceeds 25.

- Collect a field reference standard into a SUMMA® canister, or equivalent canister, to assess the accuracy of headspace gas sampling and analysis. They calculate the %R for the canister field reference standards, initiating corrective action if the %R of the field reference standard is less than 70 or greater than 130.
- Conduct sufficient headspace gas sampling to ensure a minimum 90 percent completeness where completeness is defined as the number of valid samples as a percentage of the total number of samples collected. The facility QA officer and the SQAQO evaluate the importance of any lost or contaminated headspace gas samples and initiate corrective action, as appropriate. LO-080-404 describes how sampling personnel document any nonroutine events or occurrences that may affect the quality of the headspace gas sample collected.
- Apply uniform procedures and operate equipment consistently, as specified in the *Transuranic Waste Characterization Sampling and Analysis Methods Manual* (DOE 1996e) (Methods Manual), to ensure that headspace gas sampling operations are comparable to those performed at other sampling facilities.
- Follow specific headspace gas sampling steps to ensure that 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

Sampling personnel obtain headspace gas samples directly under the drum using a manifold to collect samples in SUMMA® canisters, or equivalent canisters, or as part of an on-line integrated sampling/analysis system, or direct canister sampling method. Sampling personnel collect headspace gas samples in a controlled radiological area, as described in LO-080-404 and "Obtain Headspace Gas Samples of TRU Waste Containers" (DO-080-009). These procedures meet the requirements of Section 7.2 of the QAPP and procedures 110.1, 110.2, 110.3, and 110.4 of the Methods Manual. When sampling with the direct canister method, a modified canister as described in subsection 7.2.2 may be applied.

The handling of headspace gas samples in SUMMA® canisters requires samples to be equilibrated to ambient temperature conditions prior to measuring canister pressure and ambient pressure and temperature. Canister pressure and ambient temperature/pressure must be measured within 24 hours of sample receipt at the laboratory.

7.2.1 Manifold

Sampling and analytical personnel employ a multiport manifold capable of collecting headspace samples for analysis and QC purposes, as described in LO-080-404. They may use the manifold to collect samples in SUMMA[®] canisters, or equivalent canisters, or as part of an on-line integrated sampling/analysis system. They leak check and clean sampling equipment prior to first use and as needed thereafter. They evacuate the manifold and sample canisters to less than or equal to 0.10 mm Hg prior to sample collection. They attach clean and evacuated sample canisters to the evacuated manifold before the manifold inlet valve is opened. They attach a changeable filter connected to an appropriate sampling head(s) to the manifold inlet.

Sampling and analytical personnel equip the manifold with a purge assembly, which allows QC samples to be collected through all of the same manifold components that the drum headspace passes through. If using SUMMA[®] canisters, or equivalent canisters, for headspace gas sampling, they may collect field blanks directly into the canister, or through the manifold. They calculate the internal volume and document it in the field logbook or datasheet.

The sampling manifold consists of a sample side and a standard side. The sample side is connected to the standard side for cleaning and collecting equipment blanks and field reference standards. The sample side consists of the following major components:

- Applicable sampling head that forms a leak-tight connection with the headspace sampling manifold
- A flexible hose that allows movement of the sampling head from the purge assembly (standard side) to the waste container
- A pressure sensor(s) is pneumatically connected to the manifold and is able to measure absolute pressure in the range from 0.05 mm Hg to 1,000 mm Hg. Resolution is ± 0.005 mm Hg at 0.05 mm of Hg. The manifold pressure sensor(s) has an operating range from approximately 15°C to 40°C.
- Ports for attaching sample canisters. A sufficient number of ports are available to allow simultaneous collection of headspace gas samples and duplicates. Ports not occupied with sample canisters during cleaning or headspace gas sampling activities are plugged or isolated to prevent ambient air from entering the system. Ports have VCR[®] fittings for connection to the sample canister(s) to prevent degradation of the fittings on the canisters and manifold.
- The sample canisters are leak-free welded, stainless steel pressure vessels with a chromium-nickel oxide (Cr-NiO) SUMMA[®] passivated, or equivalent, interior surface, bellows valve, and a pressure/vacuum gauge (see Methods Manual, DOE 1996e). All sample canisters have VCR[®] fittings for connection to sampling and analytical equipment. A pressure/vacuum gauge is mounted on each canister. It is helium leak tested to 1.5×10^{-7} standard cc/sec, has all stainless steel construction, and is capable of tolerating temperatures to 125°C. The gauge range is capable of indicating from 30 psig pressure to 30 in. mercury vacuum.
- A dry vacuum pump with the ability to reduce the pressure in the manifold to 0.05 mm Hg.

- The distance between the sampling head and the valve that isolates the pump from the manifold is minimized in order to minimize the dead volume in the manifold. The outer diameter of the system's tubing is one-eighth inch.
- If real-time blank results are not available, the manifold is equipped with an OVA capable of detecting all analytes listed in Table 12-1 and measuring total VOC concentrations as low as 0.1 ppm. The OVA measurement is confirmed by the collection of equipment blanks (Section 7.3) to check for manifold cleanliness.

The standard side consists of the following major elements:

- A cylinder of compressed zero air, helium, nitrogen, or hydrocarbon and CO₂-free dry air to clean the manifold between samples and provide gas for the collection of equipment blanks or on-line blanks. These high-purity gases are certified by the manufacturer to contain less than 1 ppm total VOCs. The gases are metered into the standard side of the manifold by two-stage stainless steel regulators. Alternatively, a zero air generator may be used if a sample of the zero air is collected and demonstrated to contain less than 1 ppm total VOCs. Zero air from a generator is humidified, unless using Fourier transform infrared spectroscopy (FTIRS) for analysis.
- Cylinders of field reference standard or on-line control sample gases. Each cylinder of field reference or control sample gas has a flow-regulating device. The field reference standard or on-line control sample gases are certified by the manufacturer to contain known analytes at known concentrations.
- If using an analytical technique other than FTIRS, the manifold is equipped with a humidifier filled with American Society for Testing and Materials (ASTM) Type II water, connected, and opened to the standard side of the manifold between the compressed gas cylinders and the purge assembly. In lieu of the humidifier, the compressed gas cylinders (e.g., zero air, field reference standard gas) may contain water vapor in the ration range of 1,000 to 10,000 ppmv. If using FTIRS for analysis, the samples and sampling system is kept dry.
- A purge assembly that allows the sampling head (sample side) to be connected to the standard side of the manifold.
- A flow-indicating device connected downstream of the purge assembly to monitor the flow rate of gases through the purge assembly to ensure that excess flow exists during cleaning activities and during QC sample collection.

In addition to a manifold consisting of a sample side and a standard side, the area in which the manifold is operated contains sensors for measuring ambient pressure and ambient temperature, as follows:

- The ambient pressure sensor has a sufficient measurement range for the ambient barometric pressures expected at the sampling location. It is kept in the sampling area during sampling operations. Its resolution is 1.0 mm Hg or less, and calibration is based on National Institute of Standards and Technology (NIST), or equivalent, standards.
- The temperature sensor has a sufficient temperature range for the ambient temperature expected at the sampling location. The temperature sensor calibration is traceable to NIST, or equivalent, standards.

7.2.2 Direct Canister

For this method, sampling personnel attach sampling heads directly to an evacuated sample canister, as described in LO-080-404, based on Methods Manual Procedure 110.2. A multi-canister sample device may be used to collect additional sample volume. They evacuate canisters to less than or equal to 0.10 mm Hg prior to use and attach the canister to a changeable filter connected to the appropriate sampling head. They determine and document the sampling head internal volume. They use equipment designed to minimize the internal volume of sampling heads.

Field duplicates are collected at the same time, in the same manner, and using the same type of sampling apparatus as used for the headspace gas sample collection. Field blanks are samples of room air collected in the immediate vicinity of the waste drum sampling area. Equipment blanks and field reference standards are collected using a purge assembly equivalent to the standard side of the manifold described in Section 7.2.1. These samples are collected from the needle tip through the same components (e.g., needle, filter) that the headspace gas samples pass through.

Sampling and analytical personnel ensure that each sample canister used with the direct canister method has a pressure/vacuum gauge capable of indicating leaks and sample collection. These gauges are able to indicate from 30 psig pressure to a vacuum of 30 inches of mercury, helium leak tested to 1.5×10^{-7} standard cc/sec, have all stainless steel construction, and are capable of tolerating temperatures to 125°C. If a canister pressure/vacuum gauge indicates an unexpected pressure change, sampling and analytical personnel determine if the change is a result of ambient temperature and pressure differences or a canister leak.

7.2.3 Sampling Heads

Sampling and analytical personnel ensure that sampling heads form a leak-tight connection with the manifold or canister. They sample through the carbon filter or gas-tight seal, or through the drum lid to collect a representative sample.

Sampling and analytical personnel ensure that the lid of the drum's 90-mil polyethylene liner (if present) contains a hole for venting to the drum. If headspace gas samples are collected prior to venting the 90-mil liner, they prepare, submit, and resolve a nonconformance report.

Sampling through the Carbon Filter

Sampling and analytical personnel sample the drum headspace gas through the drum's carbon composite filter or directly through a gas-tight fitting. They ensure that the sample collected is representative by meeting all of the general method requirements, sampling apparatus requirements, and QC requirements described in this section. They seal the drum's carbon composite filter or install a gas-tight fitting to prevent outside air from entering the drum and diluting and/or contaminating the sample.

The sampling head contains a changeable filter to prevent particles from contaminating the gas sample, and an adapter to connect the sampling head to the sample collection canisters. To prevent cross-contamination, sampling and analytical personnel clean or replace the sampling head after sample collection, after field reference standard collection, and after field blank collection. They use a purge assembly that is compatible with the sampling head.

Sampling Through the Drum Lid

To sample the drum headspace gas through the drum lid, sampling and analytical personnel breach the lid using a sparkless punch. They pneumatically connect sampling heads to the drum punch and ensure

that an airtight seal is formed between the drum lid and the manifold or direct canister. They ensure that the sample collected is representative by meeting all of the general method requirements, sampling apparatus requirements, and QC requirements, in addition to the following requirements:

- Drum-punch sampling equipment is securely attached to the drum lid.
- The seal between the drum lid and sampling equipment is designed to minimize intrusion of ambient air.
- All components of the drum punch sampling system that come into contact with sample gases are purged with humidified zero air, nitrogen, helium, or hydrocarbon and CO₂-free dry air prior to sample collection.
- Equipment blanks (or on-line blanks) and field reference standards (or on-line control samples) are collected through all of the components of the punch that contact headspace gas samples.
- Pressure is applied to the sparkless punch until the drum lid has been breached. Then the punch is backed out to expose the headspace gas. Provisions are made to prevent the punch from rotating as it is pressed through the drum lid.
- Provisions are made to relieve potential drum pressure increases during drum-punch operations; pressure increases may occur during sealing of the drum punch to the drum lid.
- During sampling, the drum's carbon composite filter, if present, is sealed to prevent outside air from entering the drum.
- A flow indicating device to verify excess flow of QC gases is pneumatically connected downstream of the drum punch and operated in the same manner as the flow indicating device described in Section 7.2.1.

7.3 QUALITY CONTROL

Sampling personnel collect QC samples on a per sampling batch basis. Table 7-2 summarizes QC sample collection requirements. Table 7-3 summarizes QC sample acceptance criteria. Sampling personnel implement these QC requirements with the facility QA officer providing oversight.

The SQAQ and facility QA officer monitor and document QC sample results. Sampling personnel, with the facility QA officer, initiate an NCR if final, reported QC sample results do not meet the acceptance criteria. The SPM, FQAQ, and facility manager ensure that appropriate corrective action is taken if acceptance criteria are not met.

Sampling personnel collect field blanks before sample collection at a frequency specified in Table 7-2. Equipment blanks are collected once per batch of samples collected using the manifold method. For the direct canister method, if the results of the field blanks are within acceptance criteria, a separate equipment blank need not be collected and analyzed. Sampling personnel use the field blank data to assess impacts of ambient contamination (if any) on the sample results. They use the equipment blank data to assess impacts of potentially contaminated sampling equipment on the sample results and ensure that corrective action is taken when equipment blank data indicate equipment contamination.

Sampling personnel use field reference standards to assess the accuracy with which the sampling equipment collects VOC, hydrogen, and methane samples before first use. They ensure that field reference

standards 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 hydrogen and methane greater than or equal to the PRQLs listed in Table 11-1. 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. For on-line sampling and analysis systems, sampling personnel also collect gas chromatography/mass spectrometry (GC/MS) comparison samples simultaneously into SUMMA[®] canisters, or equivalent canisters, or sequentially using an on-line integrated sampling/analysis system at the frequency specified in Table 7-2 to assess the precision with which the sampling procedure collects samples.

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.

The acceptance criteria for on-line batch QC samples are the same as for the sampling batch and analytical batch QC samples they replace. Acceptance criteria are shown in Tables 7-2, 11-2, and 12-2. A separate field blank is collected and analyzed for each on-line batch. However, if the results of a field blank collected through the sampling manifold meet the acceptance criterion, a separate on-line blank is not collected or analyzed. On-line QC samples are described further in the headspace gas analytical sections (Sections 11 and 12).

Table 7-2. Summary of Headspace Gas Quality Control Sample Frequencies.

QC samples	Manifold systems	Direct canister systems	On-line systems
Field blank ^a	1 per sampling batch ^b	1 per sampling batch ^b	1 per on-line batch ^b
Equipment blank or on-line blank ^c	1 per sampling batch ^b	once ^d	1 per on-line batch ^b
Field reference standard or on-line control sample ^e	1 per sampling batch ^b	once ^d	1 per on-line batch ^b
Field duplicate or on-line duplicate	1 per sampling batch ^b	1 per sampling batch ^b	1 per on-line batch ^b

^aAnalysis of field blanks for VOCs (Table 12-1) only. For on-line integrated sampling/analysis systems, if field blank results meet the acceptance criterion, a separate on-line blank is not analyzed.

^bSee Definitions section.

^cOne equipment blank or on-line blank 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.

^dOne equipment blank and field reference standard is collected after equipment purchase, cleaning, and assembly.

^eOne field reference standard or on-line control sample is collected, analyzed, and demonstrated to meet the acceptance criteria prior to first use, then at the specified frequency thereafter.

QC = quality control
VOC = volatile organic compound

Table 7-3. Summary of Sampling Quality Control Sample Acceptance Criteria.

QC samples	Acceptance criteria	Corrective action ^a
Field blank	VOC amounts <3 x MDLs in Table 12-1	Nonconformance if any VOC amount ≥ 3 x MDLs in Table 12-1
Equipment blank or on-line blank	VOC amounts <3 x MDLs in Table 12-1	Nonconformance if any VOC amount ≥ 3 x MDLs in Table 12-1
Field reference standard or on-line control sample	70 - 130 %R	Nonconformance if %R <70 or >130
Field duplicate or on-line duplicate	RPD ≤ 25	Nonconformance if RPD >25

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

%R = percent recovery
MDL = method detection limit
QC = quality control
RPD = relative percent difference
VOC = volatile organic compound

7.4 EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Sampling equipment components that come into contact with headspace gases are constructed of relatively inert materials (e.g., stainless-steel or Teflon[®]). To minimize the potential for cross-contamination of samples, sampling personnel clean and leak check the sampling equipment and canisters before headspace gas sampling and after the sample is removed from the canister. Sampling personnel test, inspect, and maintain sampling tools and areas according to the QAPD and applicable manufacturer's specifications.

The manifold must be specially cleaned after a field reference standard has been collected, because the field reference standard gases contaminate the standard side of the headspace sampling manifold when they are regulated through the purge assembly. To prevent cross contamination, the needle, adapters, and filter of the sampling heads must be cleaned in accordance with the cleaning procedures described in procedures 110.1 and 110.2 of the Methods Manual, or equivalent. After sample collection, the sample head must be disposed of or cleaned in accordance with the Methods Manual procedures, or equivalent, prior to reuse. As a further QC measure, the needle and filter, after cleaning, should be purged with zero air, nitrogen, or helium and capped for storage to prevent sample contamination by VOCs potentially present in ambient air.

Sampling personnel leak test sampling equipment, including canisters, sampling heads, and manifolds after assembly and prior to use. Sample canisters are subjected to a 24-hour leak test prior to cleaning. For a positive pressure check, if the pressure does not change by more than ± 2 psig in 24 hours, the canister is acceptable. To leak test the sampling manifold, sample canisters must be attached to the canister ports or the ports must be capped or closed by valves and the sampling head must be attached to the purge assembly.

Procedures for cleaning and preparing sampling equipment and sample canisters are described in the procedure, LO-080-404, and the laboratory procedure, "Cleaning SUMMA[®] Canisters" (LO-080-407). These procedures are based on Section 7.4 of the QAPP and the following Methods Manual procedures:

- Procedure 110.1 for cleaning and leak-checking sampling manifolds
- Procedure 110.2 for cleaning and leak-checking the direct canister equipment
- Procedure 210.1 for SUMMA[®] canister, or equivalent canister, cleaning and certification

7.5 EQUIPMENT CALIBRATION AND FREQUENCY

Sampling personnel calibrate the sampling equipment (as necessary) according to "TRU Control of Measuring, Testing, and Data Collection Equipment" (WMH-400, Section 2.4.4), which is based on procedure 110.2 of the Methods Manual. They ensure that the ambient pressure and temperature sensors are certified prior to initial use and then annually, using standards or equipment traceable to the NIST. Pressure and temperature sensors are calibrated in accordance with manufacturer's specifications. Analytical personnel prepare headspace gas sample canisters according to procedure 210.1 of the Methods Manual. They ensure that the dial pressure gauge of each sample canister is certified using standards or equipment traceable to the NIST.

7.6 DATA MANAGEMENT

Sampling personnel record headspace gas sample collection information for each waste container. WMH-400, Section 8.1.8, describes procedures for recording, reviewing, and reporting sampling information and includes an example of the form used to record sample collection information. FQAO validates the data and submits the sampling batch data report to the Project records custodian. The Project records custodian will sign the data report out to the SQAQ as described in WMH-400, Sections 1.5.1 and 7.1.6. The data report, which is described in WMH-400, Section 8.1.8, consists 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 that the data generation level review, validation, and verification have taken place (Section 3.1.1)
- Sampling information specified in Section 6.1
- NCRs or CARs, if applicable.

The sampling facility's records manager provides records to the Project records custodian. The Project records custodian maintains the records, retrievable by sampling batch number, in accordance with "TRU Records Management" (WMH-400, Section 1.5.1). These records include the following:

- Copies of the sampling batch reports submitted to the SQAQ, filed in accordance with the sampling batch number
- Copies of the completed sample COC records
- Reference standard gas cylinder certification information
- Instrument calibration, testing, inspection, maintenance, and repair records.

7.7 PROCEDURES PERTINENT TO THIS SECTION

Table 7-4 lists the implementing procedures pertinent to this section of the QAPjP.

Table 7-4. Section 7 Implementing Procedures.

Document number	Title
LO-080-404	<i>Headspace Gas Sampling of TRU Waste Containers</i>
DO-080-009	<i>Obtain Headspace Gas Samples of TRU Waste Containers</i>
LO-090-450	<i>Sample Chain of Custody Acceptance and Disposal</i>
LO-080-407	<i>Cleaning SUMMA[®] Canisters</i>
WMH-400, Section 1.3.2	<i>TRU Nonconforming Item Reporting and Control</i>
WMH-400, Section 1.3.3	<i>TRU Corrective Action Reporting and Control</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 2.4.4	<i>TRU Control of Measuring Testing, and Data Collection Equipment</i>
WMH-400, Section 8.1.8	<i>Data Management for WIPP Headspace Gas Samples</i>

8. SAMPLING OF HOMOGENEOUS SOLIDS AND SOIL

Project techniques for sampling homogeneous solids (matrix parameter summary category S3000) and soil/gravel (matrix parameter summary category S4000) are designed to obtain a representative sample to characterize a waste stream. These techniques ensure that samples are randomly selected. Sampling procedures are identified in "Transuranic Waste Repackaging, Visual Examination, and Sampling" WMH-400, Section 7.1.3. Sampling personnel sample waste containers in matrix parameter summary categories S3000 and S4000 that have been statistically selected by the SPM (Section 5).

Waste containers may contain bulk homogeneous solids or soil/gravel within smaller containers (e.g., 1-gal. polyethylene bottles). For waste packaged in smaller containers within drums or waste boxes, smaller containers are grouped according to waste stream, and a representative sample is collected from one randomly selected smaller container of each waste stream. The samples are analyzed for VOCs, SVOCs, and metals, as described in Sections 13, 14, and 15, respectively.

8.1 QUALITY ASSURANCE OBJECTIVES

Sampling personnel collect a sample at a location randomly selected in the horizontal and vertical planes of the waste. WMH-400, Section 7.1.3, identifies the methods (summarized below) used to demonstrate compliance with the QAOs. Sampling personnel:

- Collect duplicates (e.g., collocated samples) once per sampling batch (see Definitions section) or once per week during sampling operations, whichever is more frequent. They determine precision by calculating and reporting the RPD between collocated samples.
- Comply with methods and requirements described in this section to minimize sample degradation and maximize sampling accuracy. Because waste containers 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 and achieve a minimum 90 percent completeness. The FQAO and SQAQ evaluate the importance of any lost or contaminated samples and determine whether corrective action is appropriate. WMH-400, Section 7.1.3 describes the process for documenting any nonroutine events or occurrences that may affect the quality of the samples collected.
- Apply uniform procedures and measurement units and use sampling equipment consistently to ensure that sampling operations are comparable. They collect and evaluate collocated samples, as described in Section 8.3.1.
- Ensure representativeness of samples by cleaning sampling tools and equipment before sampling and by coring the entire depth of the waste. The core collected must have a length greater than or equal to 50 percent of the depth of the waste.

- Visually examine the sample to verify minimal waste disturbance, and document the observation (e.g., undisturbed, cracked, pulverized) in sampling records. Sampling operations and tool selection are designed to minimize alteration of the in-place waste characteristics.

8.2 METHODS REQUIREMENTS

WMH-400, Section 7.1.3, describes the sampling apparatus and process used to obtain samples from homogeneous solids and soil/gravel. Procedure 120.1 in the Methods Manual describes the method for collection of representative homogeneous solids and soil/gravel samples via coring the waste. WMH-400, Section 7.1.3, allows for preparing composite samples (except for those undergoing VOC analysis). Sampling personnel provide the minimum amount of required sample to the laboratory based on analytical requirements, minimizing the quantity of investigation-derived waste.

8.2.1 Sample Collection

Sampling personnel collect samples of homogeneous solids and soil/gravel from waste containers, when possible, in a manner that minimizes disturbance to the core. They may use rotational or nonrotational coring tools to collect cores from containers of the waste. They may also use other types of sampling tools to collect representative samples of homogeneous solids and soil/gravel from waste containers.

Sampling personnel meet the following requirements when using coring tools:

- Each coring tool contains a removable tube (liner) that is constructed of fairly rigid material unlikely to affect the composition and/or concentrations of target analytes in the sample core (e.g., Teflon[®], polycarbonate, glass). Sampling personnel interface with site representatives to ensure that analytes of concern are not likely to be present in liner material.

Before use, the liner is cleaned as described in Section 8.3. The liner fits flush with the inner wall of the coring tool and is of sufficient length to hold a core that is representative of the waste along the entire depth of the waste. The liner material has sufficient transparency to allow VE of the core after sampling. If subsampling is not conducted immediately after core collection and liner extrusion, end caps constructed of material unlikely to affect the composition and/or concentrations of target analytes in the core (e.g., Teflon[®]) are placed over the ends of the liner. End caps fit tightly to the ends of the liner.

- A spring retainer is used with each coring tool when the physical properties of the waste are such that the waste may fall out of the coring tool's liner during sampling activities. The spring retainer is constructed of relatively inert material (e.g., stainless steel, Teflon[®]) and its inner diameter is not less than the inner diameter of the liner. Before use, spring retainers are cleaned as described in Section 8.3.
- Coring tools have an air lock mechanism that opens to allow air inside the liners to escape as the tool is pressed into the waste (e.g., ball check valve). This air lock mechanism closes when the core is removed from the waste container.
- After disassembling the coring tool, a device (extruder) to forcefully extrude the liner from the coring tool is used if the liner does not slide freely. All surfaces of the extruder that may come into contact with the core are cleaned as described in Section 8.3 prior to use.

- Coring tools are of sufficient length to hold the liner and are constructed to allow placement of the liner leading edge as close as possible to the coring tools leading edge. The leading edge of the coring tools is sharpened and tapered to a diameter equivalent to, or slightly smaller than, the inner diameter of the liner. All surfaces of the coring tool that have the potential to contact the sample core are cleaned as described in Section 8.3 prior to use.
- Rotational coring tools have a mechanism to minimize the rotation of the liner inside the coring tool during coring activities, thereby minimizing physical disturbance to the core. Rotational coring is conducted in a manner that minimizes transfer of frictional heat to the core, thereby minimizing potential loss of VOCs.
- Nonrotational coring tools are designed such that the tool's kerf width is minimized.

8.2.2 Subsample Collection

Sampling personnel conduct subsampling prior to extruding the core from the liner, in accordance with the following requirements:

- Subsampling is conducted as soon as possible after sample collection. If a substantial delay (i.e., more than 60 min.) is expected between sample collection and subsampling, the sample is protected against loss of VOCs by one of the following methods: leaving the coring tool and core in the waste container with the air lock mechanism attached; leaving the liner and core in the coring tool and capping the coring tool at each end; or removing the liner from the coring tool, leaving the core in the liner, and capping the liner at each end.
- The location for the VOC subsample is randomly selected and a device is immediately used to collect the subsample once the sample has been exposed to air. Immediately after subsample collection, it is placed into a 40-mL vial, the top rim of the vial is visually inspected and wiped clean of any waste residue, and the vial cap secured.
- Subsamples of homogeneous solids and soil/gravel for SVOCs, PCBs, and metals may be collected from the same location and in the same manner as the subsample(s) collected for VOC analysis, or they may be collected by splitting or compositing a representative subsection of the sample. All surfaces of the sampling tools that have the potential to come into contact with the sample are constructed of materials unlikely to affect the composition or concentrations of target analytes in the waste (e.g., Teflon®).

8.3 QUALITY CONTROL

QC requirements for homogeneous solids and soil/gravel sampling include the collection and analysis of collocated samples to determine precision, equipment blanks to verify cleanliness of the sampling tools and equipment, and reagent blanks to ensure that reagents (e.g., deionized or high-pressure liquid chromatography [HPLC] water) are of sufficient quality. WMH-400, Section 7.1.3, includes steps for demonstrating compliance with QAPP QC requirements.

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

8.3.1 Co-located Samples

Sampling personnel collect co-located cores to determine the combined precision of coring and sampling procedures. Cores are collected side-by-side as close as feasible to one another, handled in the same manner, visually inspected, and sampled in the same manner at the same randomly selected sample location. Co-located cores are collected at a frequency of one per week during sampling operations or one per sampling batch, whichever is more frequent. If visual inspection reveals inconsistencies in the waste, sampling personnel randomly select two new co-located samples.

Sampling personnel develop control charts for RPD in cores to establish acceptance criteria for co-located samples for each constituent for each waste matrix or waste type (e.g., organic sludges or inorganic sludges), as needed using historical analysis results. WMH-400, Section 7.1.4, describes the process for developing, updating, and evaluating control charts for RPD in cores.

The historical analysis results will not exist initially, but will be collected over the course of waste characterization activities. An interim method will be used until adequate data are available to develop control charts. Once constructed, RPDs for additional co-located pairs will be compared with the control chart to determine whether the co-located cores are acceptable. Periodically, the control charts will be updated using all available data. For evaluating co-located core results, a two-sided control chart (both upper and lower limits) will be used. The limits may be as much as three standard deviations from the centerline ($Z_p = 3$). RPDs from at least 30 pairs of co-located cores must be used to construct the control charts.

The interim method used to establish acceptance criteria at the beginning of waste characterization activities will compare the variance between co-located cores to the variance measured within the waste stream (exclusive of the containers with co-located core measurements) using a statistical test. The test will be performed for each constituent in each waste stream.

The test is not considered sensitive and is presented as an interim method until the preferred method of control charting can be implemented. Because of the expected difference between co-located core variance and waste stream variance, the test will rarely reject the hypothesis that the co-located core variance is less than the waste stream variance. However, without sufficient data to develop control charts and without established acceptance criteria for field duplicates (i.e., as specified in SW-846), the interim method is a reasonable approach for evaluating co-located cores.

The statistical test used in the interim method will involve calculating the variance for co-located cores by pooling the variances computed for each pair of co-located cores for each constituent. The pooled variance is computed using Equation 8-1:

$$s_p^2 = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + \dots + (n_c - 1)s_c^2}{n_1 + n_2 + \dots + n_c - c} \quad 8-1$$

where n_1 is the number of co-located samples collected from the first of c co-located sets in a waste stream, n_2 is the number of co-located samples collected from the second set, etc.; s_1 is the standard deviation for concentrations of a particular constituent measured in co-located sets in a waste stream, s_2 is the standard deviation for the constituent measured in the second set, etc. For the case of pairs of co-located samples collected, $n_1 = n_2 = \dots = n_c = 2$, and the pooled variance equation reduces to

$$s_p^2 = \frac{s_1^2 + s_2^2 + \dots + s_c^2}{c} \quad 8-2$$

The test hypothesis that the variance between co-located cores is equal to the variance measured within the waste stream is evaluated using the F distribution and the following test statistic:

$$\frac{s_p^2}{s_{ws}^2} \quad 8-3$$

where s_{ws} is the standard deviation of concentration for a constituent within the waste stream.

The test statistic is compared to percentiles of the F distribution to conduct the hypothesis test. If the expression

$$F\left(\frac{\alpha}{2}; n_p - 1, n_{ws} - 1\right) \leq \frac{s_p^2}{s_{ws}^2} \leq F\left(1 - \frac{\alpha}{2}; n_p - 1, n_{ws} - 1\right) \quad 8-4$$

is true, it is concluded that the variances are equal. If the expression is not true, it is concluded that the variances are not equal (Neter and Wasserman, 1974). In the expression, n_p is the number of co-located sets of data for the constituent in the waste stream and n_{ws} is the number of containers sampled in the waste stream, excluding those from which co-located cores were collected. The standard deviation for the waste stream is computed excluding any data from drums with co-located cores because the test requires that the variance estimates be independent. All data must be transformed to normality, as discussed in Section 5.4.1, prior to computing variances and performing the test. A significance level, α , of 0.10 will be used for the test. This interim method will be replaced with the control charting method once sufficient data are available.

8.3.2 Equipment Blanks

Sampling personnel clean sampling equipment and sample containers in accordance with the *Specification and Guidance for Obtaining Contaminant-Free Sample Containers* (EPA 1992). Alternatively, they may purchase certified sampling equipment and containers cleaned in accordance with EPA protocol and certified per the manufacturer.

Sampling personnel clean, identify, and seal in protective wrapping sampling equipment in compliance with the QAPP. They collect equipment blanks from fully assembled sampling tools and equipment before first use at a frequency of one sample per equipment cleaning batch. If sampling personnel use "EPA-certified clean" equipment and discard equipment after one use, they do not collect equipment blanks from that equipment. Equipment blanks are collected using clean water (e.g., deionized or HPLC water). Sampling 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, sampling personnel clean the associated equipment cleaning batch of sampling tools and equipment again and collect and analyze another equipment blank before using the equipment. Sampling personnel review the equipment blank results before using tools and equipment and maintain a sufficient quantity of clean equipment in storage to prevent disruption of sampling operations.

Sampling records trace the equipment blank results to the items in the equipment cleaning batch based on the unique identification number assigned to each sampling tool. Sampling personnel reference the sampling tool identification number in sampling records and relate this number to the waste container number on which it is used. They test one tool for cleanliness from each equipment cleaning batch before use and record the identification number of this tool in the field records.

8.4 EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Sampling 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 the QAPD and applicable manufacturer's specifications. Sampling personnel provide testing, inspection and maintenance records to the Project records custodian, as described in WMH-400, Section 1.5.1.

Prior to initiating coring activities, coring tools will be tested in accordance with manufacturer's specifications to ensure operation within manufacturer's tolerance limits. Other specifications specific to the sampling operations (e.g., operation of containment structure and safety systems) will also be tested and verified as operating properly prior to initiating coring activities. Coring tools will be assembled, including liners, and tested. Air lock mechanisms and rotation mechanisms will be inspected for free movement of critical parts. Coring tools found to be malfunctioning will be repaired and replaced prior to use.

Coring tools and sample collection equipment will be maintained in accordance with manufacturer's specifications. Clean coring tools and sampling equipment will be sealed inside protective wrapping and maintained in a clean storage area prior to use. A sufficient supply of spare parts will be maintained to prevent delays in sampling activities due to equipment down time. Records of equipment testing, inspection, maintenance and repair will be provided to the Project records custodian as directed by the SPM.

Inspection of sampling equipment and supplies will include the following:

- Sample collection equipment in the immediate area of sample collection will be inspected daily for cleanliness. Visible contamination of any equipment (e.g., waste on floor of sampling area, hydraulic fluid from hoses) that has the potential to contaminate a waste core or waste sample will be thoroughly cleaned upon its discovery.
- The waste coring and sampling work areas will be maintained in clean condition to minimize the potential for cross contamination between cores and samples.
- Expendable equipment (e.g., plastic sheeting, plastic gloves) will be visually inspected for cleanliness prior to use and properly discarded.
- Sampling and coring tools must be visually inspected prior to use. Prior to removal of the protective wrapping from a sampling or coring tool designated for use, the condition of the protective wrapping will be visually assessed. Sampling or coring tools with torn protective wrapping will be returned for cleaning. Sampling or coring tools visibly contaminated after the protective wrapping has been removed will not be used and will be returned for cleaning or will be properly discarded.

8.5 EQUIPMENT CALIBRATION AND FREQUENCY

Sampling personnel calibrate the scale used for weighing samples to maintain its operation within manufacturer's specifications and after repair or routine maintenance in accordance with QAPD requirements. Weights used for calibration are traceable to a nationally recognized standard. The sampling facility's records manager provides calibration records to the Project records custodian, as described in WMH-400, Section 1.5.1.

8.6 DATA MANAGEMENT

Sampling personnel record sample collection information for each waste container. WMH-400, Section 7.1.7, describes procedures for recording, reviewing, validating, and reporting sampling information, and includes an example of the form used to record sample collection information. The sampling FQAO validates the data and submits the sampling batch data report to the Project records custodian. The Project records custodian provides the data report to the SQAQ as shown in WMH-400, Sections 1.5.1 and 7.1.6. 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. The sampling batch data report, which is described in WMH-400, Section 7.1.3, consists of the following:

- Sampling facility name, 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 or CARs, if applicable.

The sampling facility records manager provides the records shown below to the Project records custodian. The Project records custodian maintains the records, retrievable by sampling batch number, in accordance with WMH-400, Section 1.5.1. These records include the following:

- Copies of the sampling reports submitted to the SQAQ, filed in accordance with the sampling batch number
- Copies of the completed COC records used to transfer the samples in that sampling batch to the laboratory
- Tool, liner, sampling equipment, and reagent certification information
- Instrument (e.g., scale) calibration, testing, inspection, maintenance, and repair records
- Coring information, including heat and dust generation observations.

8.7 PROCEDURES PERTINENT TO THIS SECTION

Table 8-1 lists the implementing procedures pertinent to this section of the QAPjP.

Table 8-1. Section 8 Implementing Procedures.

Document number	Title
WMH-400, Section 1.3.2	<i>TRU Nonconforming Item Reporting and Control</i>
WMH-400, Section 1.3.3	<i>TRU Corrective Action Reporting and Control</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 7.1.3	<i>Transuranic Waste Repackaging, Visual Examination, and Sampling</i>
WMH-400, Section 7.1.4	<i>Sampling Design and Data Analysis for RCRA Characterization and Visual Examination of Retrievably Stored Transuranic Waste</i>
WMH-400, Section 7.1.6	<i>TRU Waste Project Level Data Validation and Verification</i>
WMH-400, Section 7.1.7	<i>Transuranic Waste Sample and Waste Container Management Activities</i>

9. NONDESTRUCTIVE ASSAY

NDA personnel perform NDA measurements of waste containers using a combination of imaging passive/active neutron (IPAN) and gamma energy assay (GEA) systems. With these systems, NDA personnel determine the radioactive material composition, quantify the radionuclide masses, and compute the associated derived quantities (e.g., total and TRU alpha activity). The facility procedures (Section 9.7) describe the NDA processes and implementation of the requirements specified in Section 9.0 of the QAPP.

9.1 QUALITY ASSURANCE OBJECTIVES

NDA QAOs include precision, accuracy, minimum detectable concentration (MDC), total uncertainty, total bias, completeness, and comparability. Table 9-1 summarizes the QAOs for precision, accuracy, MDC, completeness, and total bias. The methods used for demonstrating compliance with the QAOs, as described in "Imaging Passive/Active Neutron Assay Operations" (WRP1- OP-0905), "Gamma Energy Assay Operations" (WRP1-OP-0906), and "Data Management for NDE/NDA Results" (WMH-350, Section 2.3) comply with Section 9.6 of the QAPP. To demonstrate compliance with the QAOs, NDA/NDE personnel perform the following:

- Determine precision through replicate processing of a waste container containing known quantities of plutonium (Pu) sources in accordance with the nominal activity compliance points specified in Table 9-1.
- Determine accuracy through replicate processing of a waste container containing known quantities of Pu sources in accordance with the nominal activity compliance points specified in Table 9-1. Accuracy is calculated from the ratio of the mean measured estimate to the known value for an accepted verification standard. Verification standards are used to test the validity of a calibration independently of the original calibration standards. Calibration standards are those used to determine the response characteristics of a measurement system. Whenever possible, both radioactive calibration and verification standards shall be obtained from NIST, the New Brunswick Laboratory, or from suppliers maintaining measurement systems traceable to NIST. Evidence of such traceability and certificates for individual standards shall be obtained from the standards suppliers.
- Determine MDC from replicate processing of calibration waste containers 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 percent and 95 percent, respectively.
- Determine total uncertainty for the 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 NDA data for 100 percent of the waste containers characterized for disposal.

Establish and document sensitivity and detection limits for assay systems to optimize the ability to discriminate between low-level and TRU wastes.

- Successfully demonstrate comparability through the NDA PDP between the Hanford Site and NDA facilities at other DOE sites. Results of the NDA PDP are reported to CAO for evaluation. Comparability is also demonstrated by performing independent assays of selected waste containers using each of the systems (IPAN Vault A compared with IPAN Vault B and GEA Vault A compared with GEA Vault B) at least two times per year or prior to use of the similar system. Data generated from these assays are compared and reported in the semiannual QA report in accordance with WMH-400, Section 3.1.2.

9.2 METHODS REQUIREMENTS

All waste containers are assayed using both GEA and IPAN systems. The GEA system establishes radionuclide ratios relative to a radionuclide whose mass is directly determined. The assay process utilizes GEA data in conjunction with IPAN data to determine the radioactive material composition, quantify radionuclide masses, and compute associated derived quantities.

NDA personnel demonstrate and document the performance of software associated with NDA in accordance with the QAPD, ASME *Quality Assurance Program Requirements for Nuclear Facilities* (NQA-1) (ASME 1989), Element 11, Supplement 11S-2; and NQA-2a, Part 2.7. They ensure that software testing covers the full range of expected system application.

NDA personnel also ensure that the methods and systems used for NDA meet the QAOs listed in Table 9-1 for the applicable ranges in which they operate. The WRP1-OP-0905 and WRP1-OP-0906 procedures detail all aspects of NDA operation. These procedures instruct operators to perform all necessary background and performance checks before and after performing any waste container assays. NDA personnel check performance check data against predetermined acceptance criteria as documented in the procedures. They initiate corrective action if any acceptance criteria are not met. They document and justify the disposition and use of any assays performed during a period ending with a suspect performance check.

Table 9-1. Quality Assurance Objectives for Nondestructive Assay.

Range of waste activity in α -Curies ^a	Nominal compliance point α -Curies ^a (g WG Pu) ^b	Parameter				
		Precision ^c (%RSD)	Accuracy ^d (%R)	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-L (55-gal.) waste container 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-L (55-gal.) waste container 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 percent.

^c $\pm$ 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 percent 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 NDA facility procedures.

%R = percent recovery
 %RSD = percent relative standard deviation
 QAO = quality assurance objective
 MDC = minimum detectable concentration
 TRU = transuranic
 WG Pu = weapons grade plutonium

NDA procedures (Section 9.7) 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 (Section 1.2). The procedures have been internally demonstrated in the NDA facility with documented performance characteristics that meet the QAOs listed in Table 9-1 for the waste activity ranges in which the systems are operated.

9.3 QUALITY CONTROL

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

NDA personnel perform routine performance checks on the IPAN and GEA systems, as described in WRP1-OP-0905 and WRP1-OP-0906. Sources are long-lived or corrected for decay, simple to reposition, of sufficiently high activity to obtain adequate counting statistics in short count times, and are relatively insensitive to handling. They operate the NDA systems in statistical control as determined by the control limits established in these procedures. Semiannually, the SQA reports these performance checks in accordance with WMH-400, Section 3.1.2, "Quality Assurance Reports to Management."

NDA 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 the data, plot the data on control charts, and compare the data to preset control limits. They report these data with NDA results to the FQAO, who submits them to the SQA. Performance checks include efficiency checks, background checks, energy calibration checks, and energy resolution checks, as appropriate for NDA systems. System performance check implementation and documentation are described in the referenced NDA procedures.

For the GEA and IPAN systems, NDA personnel perform and document an efficiency check at least twice per work shift before 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. In addition, for the GEA system, NDA personnel perform and document energy calibration checks and energy resolution checks at the beginning and end of each shift.

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

NDA personnel participate in the NDA PDP Plan to provide measurements for comparing performance with that of other DOE sites performing measurements for the same radionuclides under comparable conditions.

NDA personnel who operate the assay equipment are trained according to Project and facility procedures listed in Tables 1-5 and 9-2. Operators are trained in accordance with the requirements of NQA-1, Element 2 (with the exception of Supplement 2S-2) (ASME 1989), as described in the

WMH-400, Section 1.2.1. Requalification of operators is based on evidence of continued satisfactory performance and is done at least every two years. The Training manager ensures that training records are current and unsatisfactory performance results in disqualification, retraining, and demonstration of satisfactory performance before allowing an operator to again operate NDA systems.

9.4 EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Plant personnel ensure that radioassay equipment is tested, inspected, and maintained according to the QAPD, Section 9.4 of the QAPP, administrative and operating procedures, maintenance procedures and applicable manufacturer's specifications. Initial calibration and setup of the assay equipment was documented in acceptance test procedures. Periodic preventive maintenance on the GEA system includes periodic exchange of the liquid nitrogen dewars. Calibration of the assay equipment is performed in accordance with the vendor specifications. Operational and performance checks are performed and documented in accordance with operating procedures. These operational checks include power up sequence, system setup, interlock checks, background assays and test source assays. Control charts are used to verify system operation.

9.5 CALIBRATION PROCEDURES AND FREQUENCIES

NDA personnel perform calibrations on the NDA system following methods specified in vendor manuals and the QAPP. Correction algorithms and the ranges and waste types are documented in the calibration documentation. NDA personnel perform a full calibration of NDA systems following initial set-up. Calibrations are verified for at least one counting geometry/sample matrix combination on an annual basis. Additional calibrations of the GEA system are performed as necessary based on current operating conditions. NDA personnel provide all equipment calibration records to the Project records custodian according to WMH-400, Section 1.5.1.

NDA personnel procure primary calibration standards from suppliers maintaining measurement systems traceable to the 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 the NIST. They document the cross-calibration and provide the documentation to the Project records custodian. They prepare standards using isotopes, geometries, and matrices that closely reflect those expected in the waste.

The NDA procedures specify the range of applicability of system calibrations. Assay instruments will only be used to assay in the ranges for which they are calibrated. If assay measurement values fall outside of the applicable range for a particular instrument, the assay measurement will either be repeated on an alternate system covering the range, or NDA personnel will initiate and document corrective action. They determine and document the range of waste types to which any given calibration and set of correction factors apply.

NDA personnel ensure that all computer programs (and any revisions) are documented, verified, and validated in accordance with the QAPD before use for data generation, as described in WMH-400, Section 6.1.1. They ensure that verification includes both verification of the algorithm and test runs comparing program output to true values. They ensure that test runs exercise all default and boundary values of parameters and that programs are documented in accordance with *Standard for Software User Documentation* (American National Standards Institute [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.

The NDA 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

NDA personnel collect and reduce NDA data in accordance with "Calculation of Assay Results" (WMH 350, Section 2.2) using computer software designed for the IPAN and GEA systems. The computer software is controlled to comply with the QAPD and ANSI, as described in the NDA procedures and WMH-400, Section 6.1.1.

Facility methods for validation, including verification that the QAOs have been met, are described in WMH 350, Section 2.3. Data are verified at the facility level by an independent technical reviewer and a technical supervisor, who ensures that the data generation and reduction functions have been performed in a technically correct manner and that the QA documentation is complete. The facility QA officer ensures that the independent technical and technical supervisory reviews have been completed and validates the data to ensure that the QAOs in Table 9-1 have been met.

NDA personnel record results of data review, validation, and verification for each testing batch data report, as described in Section 3.1.1. Raw data and calibration reports shall be provided to the Project records custodian. Raw data are retained in sufficient detail to allow the calculations to be repeated. All uncertainties are made at the 95 percent confidence level using the terms derived for compliance with the QAOs described in Section 9.1.

NDA personnel compile data reports on a testing batch basis assigning each testing batch data report a unique report number. They provide the final NDA data to the facility QA officer, who reviews them and submits them to the SQAQ. WMH 350, Section 2.3, contains an example of the data report, which includes the following information:

- Facility identifier, testing batch number, waste container numbers included in that testing batch, and signature releases of NDA testing personnel, as described in Section 3.1
- Table of contents

- Facility data review checklists for each testing batch verifying the data generation-level review (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 NDA (i.e., procedure identification)
 - *TRUPACT-II Content Codes (TRUCON)* (DOE 1996b) (TRUCON), and matrix parameter category, as applicable
 - Date of NDA 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 all detected 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.

The facility records manager provides records to the Project records custodian. The Project records custodian maintains the records retrievable by testing batch number, in accordance with WMH-400, Section 1.5.1. These records include raw data, calibration reports, and data reports.

9.7 PROCEDURES PERTINENT TO THIS SECTION

Table 9-2 lists the implementing procedures pertinent to this section of the QAPjP.

Table 9-2. Section 9 Implementing Procedures.

Document number	Title
WMH-400, Section 1.2.1	<i>TRU Training and Qualification Plan</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 3.1.2	<i>Quality Assurance Reports to Management</i>
WMH-400, Section 6.1.1	<i>TRU Software Quality Assurance</i>
WMH 350, Section 2.2	<i>Calculation of Assay Results</i>
WMH 350, Section 2.3	<i>Data Management for NDE/NDA Results</i>
WMH 350, Section 2.4	<i>Quality Assurance Objectives for NDA</i>
WRP1-OP-0905	<i>TRU Waste Certification - Imaging Passive/Active Neutron Assay Operation</i>
WRP1-OP-0906	<i>TRU Waste Certification - Gamma Energy Assay Operations</i>

10. RADIOGRAPHY

The Hanford Site NDE system employs two different types of imaging technologies to examine box and waste container contents: real-time radiography (RTR) and linear diode array (LDA). These systems are used individually or together, depending on the information needed and the waste configuration. Throughout this section, the term radiography refers to either or both of these systems.

Using radiography, NDE personnel determine the matrix parameter category and estimate the weights of the waste material parameters listed in Table 10-1. To verify radiography results, they perform VE on a portion of the waste containers. This section describes the radiography and visual examination processes.

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 container 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, inorganic particulates, etc.
Soils	Generally consist of naturally-occurring soils contaminated with inorganic waste materials
Steel (packaging materials)	55-gal. (208-L) drums
Plastics (packaging materials)	90-mil polyethylene drum liner and plastic bags

Source: *Transuranic Waste Baseline Inventory Report* (DOE 1995e).

10.1 QUALITY ASSURANCE OBJECTIVES

NDE radiography procedures identify the methods used to meet QAOs and the corrective actions to be taken when QAOs are not met. To demonstrate compliance with QAOs, NDE personnel perform the following:

- Measure precision by calculating the RPD between the estimated waste material parameter weights determined by radiography and these same parameters determined by VE (calculated and reported by the SQAQ).
- Determine the accuracy with which the matrix parameter category is assigned by visually examining a statistically selected random portion of waste containers (see Section 5). The SQAQ calculates the percentage of waste containers that require a new matrix parameter category after VE and reports the result as a measure of radiography accuracy.
- Meet the completeness QAO by documenting radiography or visual examinations for 100 percent of the waste containers in the Project. The VE of newly generated waste may be performed at the time the waste is packaged.
- Use standardized radiography procedures and train 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

The procedures used to achieve the radiography objectives are described in "TRU Waste Certification – Operation of the Drum Nondestructive Examination System" (WRP1-OP-0908) and "TRU Waste Certification – Operation of the Box NDE System" (WRP1-OP-0909). Trained radiography operators record data on procedure data sheets and/or audio/videotape or laser disk. NDE personnel initiate an NCR and resolve the inconsistency if radiography indicates that the waste does not match the waste stream description. WMH-350, Section 2.3, includes example radiography data reporting forms. In addition, a list of prohibited waste items and a standard weight lookup table is provided in WRP1-OP-0908. These procedures implement QAPP radiography requirements and are based on Methods Manual Procedures 310.1 and 310.2.

The drum NDE system consists of an enclosure for radiation protection, an X-ray-producing device, waste container handling system, audio/video recording system, and an operator control and data acquisition system. The NDE system employs two types of imaging systems to view the image of the drum contents: RTR and LDA. The RTR imaging chain uses an image intensifier to convert the X-rays to visible light, which is viewed by a television camera and displayed on a television monitor. The LDA uses a solid state scintillator to convert the X-rays to light photons, which are converted to digital signals and displayed on a high-resolution computer monitor.

10.3 QUALITY CONTROL

NDE personnel ensure radiography quality through operator training and experience, and qualitative and semiquantitative evaluations of visual displays. Additionally, NDE personnel verify the radiography results through visual examination of a statistically determined portion of waste containers.

10.3.1 Radiography

Only trained personnel are allowed to operate radiography equipment to ensure quality in regard to radiography system operation and for interpretation and disposition of results. Radiography operators are trained and qualified in accordance with Hanford Site and facility procedures (listed in Tables 1-5 and 10-3) to meet the standardized training requirements based upon existing industry standard training requirements and ASME NQA-1 (1989), Element 2 (except for Supplement 2S-2).

The facility training program provides radiography operators with both formal and on-the-job training (OJT). Radiography operators are instructed in the specific waste generating practices associated with the waste and typical packaging configurations expected to be found in each matrix parameter category at the Hanford Site. An experienced, qualified radiography operator conducts the OJT and apprenticeship before the training candidate is qualified. The facility radiography training program contains the following required elements based on ASME NQA-1 (1989) requirements:

- *Formal Training*
 - Project requirements
 - Federal and state 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
- *OJT*
 - System operation
 - Identification of packaging configurations
 - Identification of waste material parameters
 - Weight and volume estimation
 - Identification of prohibited items.

NDE personnel have assembled radiography test waste containers that include items common to the waste streams stored at the Hanford Site. The test waste containers must be divided into layers with varying packing densities or different waste containers may be used to represent different situations that may occur during radiography examination at the site. At least one test waste container contains the following required elements:

- Aerosol can with puncture
- Horsetail bag
- Pair of coveralls
- Empty bottle
- Irregularly shaped pieces of wood
- Empty 1-gal. 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)
- Lead glove or lead apron
- Wrench.

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

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

Requalification of operators is based on evidence of continued satisfactory performance (primarily audio/video image reviews) and is performed at least every 2 years. Unsatisfactory performance results in disqualification. Unsatisfactory performance is defined as the misidentification of a prohibited item in a training waste container or a score of less than 80 percent on the comprehensive exam. Retraining and demonstration of satisfactory performance are required before an operator is again allowed to operate the radiography system.

Each operator performs scans of waste containers in testing batches. A supervisor reviews 10 percent of the images to ensure that operator interpretations remain consistent and accurate. Imaging system characteristics of the monitoring system are verified on a routine basis.

Radiography operators perform independent replicate scans and replicate observations of the video output of the radiography process under uniform conditions and procedures. The operators perform independent replicate scans on one waste container per day or one per testing batch, whichever is less frequent. A qualified radiography operator, other than the individual who performed the first examination, performs an independent observation of one scan (not the replicate scan) once per day or once per testing batch, whichever is less frequent.

A radiography-qualified person, other than the operator who initially scanned the waste container, performs the oversight functions identified above. The results of this verification are available to the radiography operator. The SQA and NDE personnel are responsible for monitoring the quality of the radiography data and calling for corrective action, when necessary.

10.3.2 Visual Examination

Project personnel perform VE, as described in WMH-400, Section 7.1.3, as a QC check on radiography. Waste containers are randomly selected from the population of waste containers expected to be certified in a 12-month period. The number of waste containers to be visually examined is based on the previous year's miscertification rate. VE personnel perform the waste examination, weighing the waste and reporting the data, including waste material parameter weights and providing a brief description of the waste container contents. Other Project personnel perform the VE expert functions, select waste containers to undergo visual examination (Section 5), and evaluate the VE data. The VE data evaluation includes verifying the matrix parameter category and comparing radiography and VE results.

The SQA compares radiography and VE results and verifies matrix parameter categories, as described in WMH-400, Section 7.1.1, and provides the results of the comparison to the NDE facility.

For VE, VE personnel perform a semiquantitative and qualitative evaluation of the waste container contents and record the examination on audio/videotape or laser disk. Facility procedures describe standardized training for VE, including both formal classroom and OJT. VE personnel will also be trained in the waste-generating processes for the waste that they examine. A facility VE technician who is experienced and qualified in visual examination conducts the OJT and apprenticeship before qualifying the

candidate. VE personnel are requalified once every 2 years. The VE expert is familiar with the specific waste-generating processes that have taken place at the site, all of the types of waste being characterized at the Hanford Site, typical packaging configurations, and waste material parameters expected to be found in each matrix parameter category at the Hanford Site. NDE facility training programs contain the applicable required elements based on ASME NQA-1 requirements shown in Table 10-2.

Table 10-2. Visual Examination Training Elements.

Training	Visual examination technician	Visual examination expert
• <i>Formal</i>		
— Project requirements	✓	✓
— State and federal regulations	✓	✓
— Application techniques	✓	✓
— Site-specific instruction	✓	✓
• <i>On-the-job</i>		
— Identification of packaging configurations	✓	✓
— Identification of waste material parameters	✓	✓
— Weight and volume estimation	✓	✓
— Identification of prohibited items	✓	✓

The VE expert is responsible for the management and implementation VE at the NDE facility. The VE expert is selected by the SPM on the basis of his/her familiarity with the waste-generating processes that have occurred at the Hanford Site and his/her qualification and training as presented in the preceding paragraphs and in Table 10-2. These qualification and training requirements are summarized in the WMH-400, Section 1.2.1.

The VE expert decides the extent of waste segregation required to achieve Project objectives. The VE expert bases his/her examination decisions on practical waste handling protocols, the type of waste matrix being examined, and his/her professional judgement. These decisions and associated rationale are documented on the VE forms. The VE expert's decision-making criteria are described in WMH-400, Section 7.1.3, and include AK documentation, waste streams, and other pertinent waste description information.

VE personnel record a description of the waste container contents on VE data sheets. In cases where bags are not opened, they provide a brief description of the contents of the bags and an estimate of the amount of each waste type in the bags. They supplement the records of VE with the audio/video recording.

10.4 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Plant personnel ensure that radiography equipment is tested, inspected, and maintained according to the QAPD, Section 10.4 of the QAPP, administrative and operating procedures, maintenance procedures and applicable manufacturer's specifications. Penetrameter tests were performed and documented in acceptance test procedures during facility start-up. Periodic preventive maintenance includes a function check of the NDE systems safety interlock and warning devices and inspection and lubrication of the high-voltage cable connections. Operational and performance checks are performed and documented in

accordance with operating procedures. These checks include a 3-month equipment inspection, 6-month interlock and radiation survey, background test drum, warm-up sequence, Image Quality Indicator (IQI) reading in lines-paired per millimeter, and oscilloscope analysis for detector element dropout. NDE personnel maintain and record pertinent information in the RGD Operational Daily Log and RGD Maintenance Log according to WMH-400, Section 1.5.1.

10.5 INSTRUMENT CALIBRATION AND FREQUENCY

Operators ensure that radiography equipment is calibrated and maintained in accordance with controls and performance criteria established and described in facility radiography procedures listed in Table 10-3. These procedures address the performance criteria identified in the QAPP. When radiography equipment is in use, the operators conduct operational checks that include observation of a test pattern to verify video quality at the beginning of each work shift.

10.6 DATA MANAGEMENT

NDE personnel record radiography information for each testing batch. WMH-350, Section 2.3, describes procedures for recording, reviewing, validating, and reporting radiography information. The facility QA officer validates the data and submits radiography batch data reports for each testing batch to the Project records custodian. WMH-350, Section 2.3, includes example checklists.

The results of radiography are also stored using electronic reporting forms that can be downloaded in hard copy format for transmittal and storage. As part of the examination, an audio/video tape record of the examination is also made and becomes part of the QA record. WMH-400, Section 7.1.3, contains an example standard reporting form or VE.

Radiography batch data reports consist of the following:

- NDE facility identifier, testing batch number, waste container 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 container in the testing batch, including the following:
 - Title "Radiography Data Sheet"
 - Date of radiography examination
 - Waste container number
 - TRUCON code, Item Description Code, and matrix parameter category, as applicable
 - Any changes made to matrix parameter category
 - Estimate of each applicable waste material parameter weight
 - Presence/absence of waste container 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 identification number
 - Operator signature/date
 - Reviewer signature/date.
- NCRs or CARs, if applicable
 - For waste containers undergoing visual examination, NDE personnel report the following additional data on a standard reporting form:
 - Title "Visual Examination Drum Log"
 - Matrix parameter category determined by VE
 - Waste material parameter weights.

NDE personnel provide records to the Project records custodian. The Project records custodian maintains the records, retrievable by testing batch number, in accordance with WMH-400, Section 1.5.1. These records include the following:

- Audio/videotapes or laser disks
- 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

Table 10-3 lists the technical implementing procedures pertinent to this section of the QAPjP.

Table 10-3. Section 10 Implementing Procedures.

Document number	Title
WMH-400, Section 1.2.1	<i>TRU Training and Qualification Plan</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 7.1.1	<i>Transuranic Waste Characterization Data Quality Objectives Reconciliation and Reporting</i>
WMH-400, Section 7.1.3	<i>Transuranic Waste Repackaging, Visual Examination, and Sampling</i>
WMH-350, Section 2.3	<i>Data Management for NDE/NDA Results</i>
WRP1-OP-0729	<i>Visual Examination</i>
WRP1-OP-0908	<i>TRU Waste Certification - Operation of the Drum Nondestructive Examination System</i>
WRP1-OP-0909	<i>TRU Waste Certification - Operation of the Box Nondestructive System</i>

11. HYDROGEN AND METHANE ANALYSIS

Analytical personnel receive samples in SUMMA[®] canisters, or equivalent canisters, from the headspace gas sampling operations (described in Section 7) or samples may be collected directly into an on-line integrated sampling/analysis system. Project personnel analyze the samples for hydrogen and methane, as described in this section.

11.1 QUALITY ASSURANCE OBJECTIVES

Table 11-1 lists the QAOs for hydrogen and methane analysis. Key data quality indicators are defined below, and Section 3.2 presents the methods used to assess compliance with these indicators.

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 (%)
Hydrogen	1333-74-0	≤ 25	70-130	0.05	0.1	90
Methane	74-82-8	≤ 25	70-130	0.05	0.1	90

^aCriteria apply to PRQL concentrations.

%R = percent recovery
 %RSD = percent relative standard deviation
 CAS = Chemical Abstract Service
 RPD = relative percent difference
 MDL = method detection limit
 PRQL = Program required quantitation limit
 vol% = volume percent

To demonstrate compliance with the QAOs, analytical personnel perform the following:

- Measure precision by analyzing laboratory duplicates 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 control samples or on-line control samples and PDP blind audit samples and calculating %R based on the results.
- Determine MDLs, expressed in units of volume percent for hydrogen and methane, and ensure that 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 percent completeness criterion in Table 11-1 by calculating 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.

- Ensure representativeness through cleaning and certification of SUMMA® canisters, or equivalent canisters, in accordance with Section 7.4.

In addition, sampling personnel ensure representativeness through the use of the standardized headspace gas sampling methods described in Section 7.

The FQAO is responsible for monitoring the results from these measurements and determining whether the precision, accuracy, and completeness criteria listed in Table 11-1 are met. The facility QA officer evaluates performance and decides whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

11.2 METHODS REQUIREMENTS

Analytical personnel analyze headspace gas samples for hydrogen and methane using GC/thermal conductivity detection (TCD) based on Methods Manual Procedure 520.1. The handling of headspace gas samples in SUMMA® canisters requires samples to be equilibrated to ambient temperature conditions prior to measuring canister pressure and ambient pressure and temperature. Canister pressure and ambient temperature/pressure must be measured within 24 hours of sample receipt at the laboratory.

11.2.1 Standards

Analytical personnel purchase manufacturer-certified primary hydrogen and methane standards from commercial sources. The procedure "Determination of Permanent Gases in Waste Container Headspace" (LA-523-426) identifies the purity requirements and describes the procedures for preparing the standards.

11.2.2 Qualitative and Quantitative Analysis

All instruments used to analyze Project samples for hydrogen and methane meet the Methods Manual requirements. Analytical personnel quantitate hydrogen and methane 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.

Analytical personnel identify hydrogen and methane by retention time (RT) using GC/TCD. They establish RT windows around the mean absolute RT of analyzed standards (calibration standards, continuing calibration verification standards, and/or laboratory control samples). They establish the RT windows so the occurrence of both false positive and false negative results are minimized. Analytical procedures describe RT window determination and quantitation of hydrogen and methane based on signal response against a reference standard.

11.3 QUALITY CONTROL

The facility manager and QA officer are responsible for monitoring and documenting procedure performance, including analyzing QC samples, and implementing corrective action when acceptable procedure performance is not met. The facility manager ensures that 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. LA-523-426 describes the procedures to ensure hydrogen and methane analysis data quality. Analytical personnel perform operations in accordance with Project QA requirements and provide records to the SPO documenting the quality of the data generated. Analytical procedures describe the QC sample requirements and acceptance criteria, which are summarized in Table 11-2.

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 duplicate or on-line duplicate	One (1) per analytical batch or on-line batch	RPD $\leq 25^a$	Nonconformance if RPD > 25
Laboratory blank or on-line blank	One (1) per analytical batch or on-line batch	Analyte concentrations $< \text{PRQL}$	Nonconformance if analyte concentrations $\geq \text{PRQL}^b$
Laboratory control sample or on-line control sample	One (1) per analytical batch or on-line batch	70-130 %R	Nonconformance if %R < 70 or $> 130^c$
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan

^aApplies only to concentrations greater than the PRQL listed in Table 11-1.

^bNonconforming laboratory or on-line blanks shall be flagged as "B" and a nonconformance report is not required.

^cA nonconformance report, per Section 2.1.2, is required when laboratory or on-line control samples associated with final reported sample data do not meet Table 11-2 acceptance criteria.

%R = percent recovery
PDP = Performance Demonstration Program
PRQL = Program required quantitation limit
QAO = quality assurance objective
RPD = relative percent difference

Analytical personnel demonstrate acceptable performance through the analysis of method performance samples before analyzing any headspace gas samples. They purchase or prepare method performance samples that contain hydrogen and methane at concentrations appropriate to verify that all QAOs listed in Table 11-1 are met. Initially, they analyze seven method performance samples to demonstrate acceptable precision and accuracy and determine MDLs for hydrogen and methane. They demonstrate acceptable procedure performance semiannually by analyzing four method performance samples.

Analytical personnel analyze headspace gas samples for hydrogen and methane in analytical batches. Specific QC samples for each analytical batch include a laboratory blank or on-line blank, a laboratory duplicate or on-line duplicate, and a laboratory control sample or on-line control sample.

Analytical personnel analyze field samples in duplicate at a frequency of one per analytical batch. They prepare laboratory blanks from high purity nitrogen (99.999 percent pure) using the same procedure used to prepare field samples for analysis.

Analytical personnel prepare laboratory or on-line control samples using commercially purchased gas standards independent of those used for instrument calibration and ensure that control samples contain hydrogen and methane at concentrations within the calibration range of the analytical instrument. They also analyze PDP blind audit samples to determine acceptable laboratory performance.

11.4 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Analytical personnel test, inspect, and maintain analytical equipment according to the QAPD, Section 11.4 of the QAPP, and applicable manufacturer's specifications. Analytical personnel test, inspect, and maintain analytical equipment to ensure that the QAOs in Table 11-1 are met.

11.5 INSTRUMENT CALIBRATION AND FREQUENCY

Analytical personnel calibrate all instruments before use, verify calibration at routine intervals during analysis, maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency, and ensure that all initial and continuing calibration requirements listed in Table 11-3 are met.

Analytical personnel obtain an initial GC/TCD multi-point calibration curve for hydrogen and methane that consists of a minimum of three standards. They ensure that one 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. Analytical personnel use average response factors to construct the calibration plot and ensure that initial calibrations meet the criteria listed in Table 11-3.

Table 11-3. Summary of GC/TCD Calibration Requirements for Hydrogen and Methane Analysis.

Technique	Procedure	Frequency of procedure	Acceptance criteria
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 ≤ 30 for each analyte

%D = percent difference
 %R = percent recovery
 %RSD = percent relative standard deviation
 GC = gas chromatography

Analytical personnel analyze a continuing calibration verification standard at the beginning of each 12-hour shift, before 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 Table 11-3 acceptance criteria are not met, analytical personnel either run another continuing calibration or generate a new calibration plot. They do not continue sample analysis until the criteria in Table 11-3 are met.

11.6 DATA MANAGEMENT

Analytical personnel do not blank-correct hydrogen and methane results, but report results from blank analysis separately from field sample results. The facility QA officer ensures that all analytical data are reviewed, validated, and verified, as described in Section 3.1.1, and reports all hydrogen and methane results in units of volume percent.

WMH-400, Section 8.1.8, describes the data reduction, validation, and reporting processes and includes or references example forms to be used to record and report data. Analytical personnel reduce raw data to reportable results and report analytical results in units of volume percent, limited to two significant figures. Analytical personnel review, validate, and verify data, as described in Section 3.1.1.

Analytical personnel validate analytical data and submit batch data reports to the Project records custodian. Analytical or on-line batch data reports, which are described in WMH-400, Section 8.1.8, include the following:

- Facility identifier, analytical or on-line batch number, sample numbers included in that batch, a cross reference to field sample numbers, and the signature releases of analytical personnel specified in Section 3.1.1
- Table of contents
- A COC report 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 (not applicable when using on-line integrated sampling/analysis system)
- Copies of canister documentation
- Data review checklists for each batch verifying that data generation-level review, validation, and verification have taken place
- QC sample results (e.g., laboratory or on-line duplicates, laboratory or on-line control samples) if not included in data review checklists
- A separate analytical report sheet entitled, "Hydrogen and Methane Analysis Data Sheet," for each sample in the batch, including facility identifier, Project name, batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date analyzed, method number, listing of Project analytes, and analytical results in volume percent. The following data qualifying flags are used:
 - B (analyte detected in blank above the acceptance criteria)
 - 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 (estimated, one or more QC samples do not meet acceptance criteria)
- NCRs or CARs, if applicable.

SPO personnel maintain records, documented and retrievable by analytical batch numbers, in accordance with WMH-400, Section 1.5.1. These records include the following:

- Original COC records for analytical batches (not applicable when using on-line integrated sampling/analysis systems)
- All raw data, including original (or legible copy of) 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.
- A reference to instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the facility 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, QC data include blanks, control samples, duplicates, initial calibration data, initial and continuing calibration verifications, and all other method-specific QC data listed in Section 11. The QC result summary includes the facility identifier, the batch number, and method names.
- Original field sample canister tags (or equivalent documentation) for headspace gas samples.

11.7 PROCEDURES PERTINENT TO THIS SECTION

Table 11-4 lists the implementing procedures pertinent to this section.

Table 11-4. Section 11 Implementing Procedures.

Document number	Title
LA-523-426	<i>Determination of Permanent Gases in Waste Container Headspace</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 8.1.1	<i>Logkeeping Practices for WIPP Activities in SAS</i>
WMH-400, Section 8.1.8	<i>Data Management for WIPP Headspace Gas Samples</i>

12. GAS VOLATILE ORGANIC COMPOUND ANALYSIS

Analytical personnel receive samples in SUMMA[®] canisters, or equivalent canisters, from the headspace gas sampling operations described in Section 7 or samples may be collected directly into an on-line integrated sampling/analysis system. They analyze the samples for gas VOCs, as described in this section.

12.1 QUALITY ASSURANCE OBJECTIVES

Table 12-1 lists the QAOs for headspace gas VOC analysis. Key data quality indicators are defined below, and Section 3.2 presents the methods used to assess compliance. To demonstrate compliance with the QAOs, analytical personnel perform the following:

- Measure precision by analyzing laboratory duplicates or on-line duplicates, replicate analysis 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 or on-line control samples and PDP blind audit samples and calculate %R based on the results.
- Determine MDLs, expressed in ng, and ensure that MDLs are less than or equal to those 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 percent 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.
- Ensure representativeness through cleaning and certification of SUMMA[®] canisters, or equivalent canisters, in accordance with Section 7.4.

In addition, sampling personnel ensure representativeness through the use of the standardized headspace gas sampling methods described in Section 7.

The FQAO is 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. The FQAO evaluates performance and decides whether corrective action should be initiated based on the results of the precision, accuracy, and completeness calculations.

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)	PRQL ^b (ppmv)	Completeness (%)
Benzene	71-43-2	≤25	70-130	10	10	90
Bromoform	75-25-2	≤25	70-130	10	10	90
Carbon tetrachloride	56-23-5	≤25	70-130	10	10	90
Chlorobenzene	108-90-7	≤25	70-130	10	10	90
Chloroform	67-66-3	≤25	70-130	10	10	90
Cyclohexane	110-82-7	≤25	70-130	10	10	90
1,1-Dichloroethane	75-34-3	≤25	70-130	10	10	90
1,2-Dichloroethane	107-06-2	≤25	70-130	10	10	90
1,1-Dichloroethylene	75-35-4	≤25	70-130	10	10	90
<i>cis</i> -1,2-Dichloroethylene	156-59-2	≤25	70-130	10	10	90
Ethyl benzene	100-41-4	≤25	70-130	10	10	90
Ethyl ether	60-29-7	≤25	70-130	10	10	90
Methylene chloride	75-09-2	≤25	70-130	10	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤25	70-130	10	10	90
Tetrachloroethylene	127-18-4	≤25	70-130	10	10	90
Toluene	108-88-3	≤25	70-130	10	10	90
1,1,1-Trichloroethane	71-55-6	≤25	70-130	10	10	90
Trichloroethylene	79-01-6	≤25	70-130	10	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤25	70-130	10	10	90
1,2,4-Trimethylbenzene	95-63-6	≤25	70-130	10	10	90
1,3,5-Trimethylbenzene	108-67-8	≤25	70-130	10	10	90
<i>m</i> -Xylene ^c	108-38-3	≤25	70-130	10	10	90
<i>o</i> -Xylene	95-47-6	≤25	70-130	10	10	90
<i>p</i> -Xylene ^c	106-42-3	≤25	70-130	10	10	90
Acetone	67-64-1	≤25	70-130	150	100	90
Butanol	71-36-3	≤25	70-130	150	100	90
Methanol	67-56-1	≤25	70-130	150	100	90
Methyl ethyl ketone	78-93-3	≤25	70-130	150	100	90
Methyl isobutyl ketone	108-10-1	≤25	70-130	150	100	90

^aCriteria apply to PRQL concentrations.^bValues based on delivering 10 ml to the analytical system.^cThese xylene isomers cannot be resolved by the analytical methods employed in this Project.

%R = percent recovery

%RSD = percent relative standard deviation

CAS = Chemical Abstract Service

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

PRQL = program-required quantitation limit

RPD = relative percent difference

12.2 METHODS REQUIREMENTS

Analytical personnel analyze headspace gas samples for the VOCs listed in Table 12-1 using GC/MS and/or GC/flame ionization detection (FID). The handling of headspace gas samples in SUMMA® canisters requires samples to be equilibrated to ambient temperature conditions prior to measuring canister pressure and ambient pressure and temperature. Canister pressure and ambient temperature/pressure must be measured within 24 hours of sample receipt at the laboratory.

The GC/MS method is based on Methods Manual Procedure 430.1. The GC/FID method is based on Methods Manual Procedure 440.1. Specific procedures for the analysis of headspace gas samples are described in LA-523-410 and LA-523-425.

Analytical personnel use equipment and materials that meet all Methods Manual requirements for both the GC/MS and/or GC/FID methods. They equip gas chromatographs with appropriate columns based on those recommended in the Methods Manual and operate GC/MS systems in the full scan mode to detect and quantitate target analytes and identify nontarget compounds. Alternate analytical methods, which meet all of the QAOs in Section 12.1, may be submitted to CAO for approval.

12.2.1 Standards

Analytical personnel purchase certified primary standards from a commercial source. They prepare all secondary and calibration standards as specified in LA-523-410. All standards are analyzed at the same temperature ($\pm 2^{\circ}\text{C}$) as the samples.

12.2.2 Gas Chromatography/Mass Spectrometry Qualitative and Quantitative Analysis

Analytical personnel qualitatively identify target VOCs 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 determine RT windows around the mean absolute RT of standards (calibration standards, continuing calibration verification standards, and/or laboratory control samples) such that the occurrence of both false positive and false negative results are minimized. Analytical procedures detail RT window determination. They determine RTs for all analytes before analyzing any samples and whenever a new GC column is installed.

Analytical personnel use internal standard quantitation for GC/MS quantitative VOC analysis. They ensure that internal standard area counts for sample analyses are within 50 to 200 percent of the average internal standard area counts from the most recent continuing calibration. They meet %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 five 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 Gas Chromatography/Flame Ionization Detector Qualitative and Quantitative Analysis

Analytical 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 around the mean absolute RT of standards (calibration standards, continuing calibration verification standards, and/or laboratory control samples) such that the occurrence of both false positive and false negative results are minimized. Analytical procedures detail RT window determination. They determine RT windows for all analytes on each GC column before analyzing any samples, whenever a new initial calibration is performed, and whenever a new GC column is installed.

Analytical 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.

12.3 QUALITY CONTROL

The facility manager and FQAO are responsible for monitoring and documenting procedure performance, including analyzing QC samples and implementing corrective action when procedure performance is not acceptable. They ensure that QC sample results are flagged, as appropriate, or an NCR is initiated if final reported QC sample results do not meet acceptance criteria. LA-523-410 describes the actions to ensure that the of data for headspace gas VOC analysis is of known and documented quality, specifies qualitative and quantitative acceptance criteria for the QC checks, and specifies the corrective action measures to be taken when these criteria are not satisfied.. Analytical personnel comply with the Project QA requirements and provide records to the SPO to document the quality of the data generated. The referenced analytical procedures summarized in Table 12-2 describe QC sample requirements and acceptance criteria.

Analytical personnel demonstrate acceptable performance through the analysis of method performance samples before analyzing field samples. They purchase or prepare method performance samples and ensure that the samples contain the analytes listed in Table 12-1 at concentrations appropriate to verify that all QAOs are met. Initially, analytical personnel 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.

Analytical personnel analyze samples in analytical batches. Specific QC samples for each analytical batch include a laboratory or on-line blank, a laboratory or on-line duplicate, and a laboratory or on-line control sample.

Analytical personnel analyze field samples in duplicate at a frequency of one per analytical batch. They prepare laboratory blanks by filling SUMMA[®] canisters, or equivalent canisters, with high-purity humid zero air or nitrogen (99.999 percent 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 duplicate or on-line duplicate	One (1) per analytical batch or on-line batch	RPD $\leq 25^a$	Nonconformance if RPD > 25
Laboratory blank or on-line blank	Daily prior to sample analysis	Analyte amounts $< 3 \times$ MDLs	Nonconformance if analyte amounts $\geq 3 \times$ MDLs ^b
Laboratory control sample or on-line control sample	One (1) per analytical batch or on-line batch	70-130 %R	Nonconformance if %R < 70 or $> 130^c$
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan

^aApplies only to concentrations greater than the PRQLs listed in Table 12-1.

^bNonconforming laboratory or on-line blanks shall be flagged as "B" and a nonconformance report is not required.

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

%R = percent recovery
MDL = method detection limit
PDP = Performance Demonstration Program
PRQL = Program required quantitation limit
QAO = quality assurance objective
RPD = relative percent difference

Analytical personnel prepare laboratory or on-line control samples from gas or liquid standards independent of those used for instrument calibration. They ensure that GC/MS laboratory or on-line control samples contain at least six of the analytes listed in Table 12-1 and GC/FID control samples contain all target compounds. They ensure that 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 to determine acceptable laboratory performance.

12.4 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Analytical personnel test, inspect, and maintain analytical equipment according to the QAPD, Section 12.4 of the QAPP, and applicable manufacturer's specifications. Analytical personnel test, inspect, and maintain analytical equipment to ensure that the QAOs in Table 12-1 are met according to analytical procedures for GC/MS and/or GC/FID equipment.

Analytical personnel meet GC/MS instrument performance criteria before analyzing 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.

Analytical 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.

12.5 INSTRUMENT CALIBRATION AND FREQUENCY

Analytical personnel calibrate all analytical instruments before use, verify calibration at routine intervals during analysis, and comply with the calibration requirements summarized in Table 12-3. Analytical personnel ensure that all initial and continuing calibration requirements are met. Analytical personnel calibrate all instruments and maintain instrument run logs that permit the reconstruction of the calibration sequence and frequency. The procedures listed in Section 12.7 identify and describe calibration procedures.

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 $\pm$ standard deviations of initial calibration

%D = percent difference
 %R = percent recovery
 %RSD = percent relative standard deviation
 BFB = 4-bromofluorobenzene
 GC/FID = gas chromatography/flame ionization detector
 GC/MS = gas chromatography/mass spectrometry
 RT = retention time

Analytical 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. Analytical personnel operate GC/MS systems in the full scan mode to allow the detection and quantitation of all analytes listed in Table 12-1 and the identification of nontarget compounds. They generate relative response factors for each specified target analyte and ensure that the initial calibration meets all acceptance criteria listed in Table 12-3. If linearity is not demonstrated, they use a second- or third-order regression for calibration. They ensure that a valid initial calibration exists before any samples are analyzed.

Analytical 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 BFB. They determine the %D using continuing calibration response factors and average relative response factors from the most recent calibration and ensure that the %Ds meet Table 12-3 acceptance criteria. If second- or third-order regression is used, they ensure that the continuing calibration verification standard point falls within 30 percent 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.

Analytical personnel use at least three external standards to define the calibration range of the GC/FID system 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 curve, plotting area response versus concentration, and ensure that 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 calibration criteria.

12.6 DATA MANAGEMENT

Analytical 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.

Analytical 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 equivalent mass spectral library on the 20 TICs with the greatest area counts and with total ion current peaks greater than 10 percent 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 percent of all samples from a given waste stream. Analytical personnel assume a relative response factor equal to one, using the nearest internal standard, when estimating concentration for TICs.

WMH-400, Section 8.1.8, describes the data reduction, validation, and reporting processes and includes or references example forms to be used to record and report data. Analytical personnel reduce raw data to reportable results and report analytical results in units of ppmv, limited to two significant figures. Analytical personnel review, validate, and verify data, as described in Section 3.1.1.

Analytical personnel validate analytical data and submit batch data reports to the Project records custodian. For on-line sample and analysis systems, the sampling batch data report and analytical batch data report can be combined into a single batch data report for each batch. Analytical or on-line batch data reports, which are described WMH-400, Section 8.1.8, include the following:

- Facility identifier, analytical or on-line batch number, sample numbers included in that batch, a cross-reference to field sample numbers, and the signature releases of analytical personnel specified in Section 3.1.1
- Table of contents
- A COC report 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 (not applicable when using on-line integrated sampling/analysis systems)
- Copies of canister documentation
- Data review checklists for each batch verifying that data generation-level review, validation, and verification have taken place
- QC sample results (e.g., laboratory or on-line duplicates, laboratory or on-line 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 identifier, Project name, batch number, data report number, sampling batch number, laboratory sample number, field sample number, date sampled, date analyzed, method number, listing of Project analytes, and analytical results in ppmv. The following data qualifying flags are used:
 - B (analyte detected in blank above the acceptance criteria)
 - 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 (estimated, one or more QC samples do not meet acceptance criteria)
- NCRs or CARs, if applicable.

Analytical personnel provide records to the Project records custodian. The Project records custodian maintains the records, retrievable by analytical or on-line batch numbers, in accordance with WMH-400, Section 1.5.1. These records include the following:

- Original COC records for analytical batches (not applicable when using on-line integrated sampling/analysis systems)
- 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.

- A reference to instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. The calibration reports also contain the facility 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 facility identifier, the batch number (if applicable), and method names.
- Original field sample canister tags (or equivalent documentation) for headspace gas samples.

12.7 PROCEDURES PERTINENT TO THIS SECTION

Table 12-4 lists the implementing procedures pertinent to this section.

Table 12-4. Section 12 Implementing Procedures.

Document number	Title
LA-523-410	<i>Determination of Volatile Organic Compounds in TRU Waste Container Headspace</i>
LA-523-426	<i>Determination of Permanent Gases in Waste Container Headspace</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
WMH-400, Section 8.1.1	<i>Logkeeping Practices for WIPP Activities in SAS</i>
WMH-400, Section 8.1.8	<i>Data Management for WIPP Headspace Gas Samples</i>

13. TOTAL VOLATILE ORGANIC COMPOUND ANALYSIS

Analytical personnel receive solidified samples from the homogeneous solids and soil/gravel sampling operations described in Section 8. They analyze the samples for total VOCs as described in this section.

13.1 QUALITY ASSURANCE OBJECTIVES

Table 13-1 lists the QAOs for total VOC analysis. Key data quality indicators are defined below, and Section 3.2 presents the methods used to assess compliance. To demonstrate compliance with the QAOs, analytical personnel:

- Measure precision by analyzing matrix spike duplicates, replicate analysis 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 that 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 percent 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 addition, sampling personnel ensure representativeness of samples through the use of standardized sampling methods described in Section 8.

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

13.2 METHODS REQUIREMENTS

Analytical personnel prepare and analyze homogeneous solids and soil/gravel samples for total VOCs in accordance with LA-523-455. In this case, they follow a procedure based on Methods Manual Procedure 440.2.

Table 13-1. Total Volatile Organic Compounds 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 (%)
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
<i>ortho</i> -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
<i>m</i> -xylene	108-38-3	≤50	60-150	1	10	90
<i>o</i> -xylene	95-47-6	≤50	60-150	1	10	90
<i>p</i> -xylene	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

^aCriteria apply to PRQL concentrations.^bCan also be analyzed as a semivolatile organic compound.^cDetected; result must be greater than zero.^dEstimate; to be determined.

%R = percent recovery

%RSD = percent relative standard deviation

CAS = Chemical Abstract Service

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 percent analyte extraction

RPD = relative percent difference

13.2.1 Standards

Analytical personnel purchase certified primary standards from the best available source for target analytes. Commercially-purchased primary standards are certified by the manufacturer. They prepare standards in an appropriate solvent. They prepare secondary analytical standards, surrogate standards, calibration standards, and matrix spiking standards, as specified in "Volatile Organics by Gas Chromatography/Mass Spectrometry Using SW-846" (LA-523-134) and LA-523-455.

13.2.2 Qualitative and Quantitative Analysis

Analytical 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.

Analytical personnel qualitatively identify analytes using GC/FID by ensuring the analytes elute within the RT window set by the most recent continuing calibration for each analytical column. Initially, they run at least three standards for those analytes being identified by GC/FID and determine RT windows on each analytical column around the mean absolute RT of analyzed standards (calibration standards, continuing calibration verification standards, and/or laboratory control samples) such that the occurrences of both false positive and false negative results are minimized.

Analytical personnel use internal standards for quantitating target analytes by GC/MS. They 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 tune and calibration criteria for BFB, 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.

Analytical personnel use external standard quantitation for GC/FID analysis. They base analyte quantitation on the peak area response or peak height response from one of the two analytical columns. They ensure that the column used for quantitation is interferant-free in the analyte's RT window. They perform quantitation by three-point calibration curve or by response factor. They consider the GC/FID to be in control if, after analyzing a mid-range continuing calibration standard, the response factor (or calculated concentration) for any analyte is less than 15 %D from the initial calibration average response factor (or true concentration) and all criteria for RT windows are met.

13.3 QUALITY CONTROL

The laboratory manager and the facility QA official are responsible for monitoring and documenting procedure performance, including analyzing QC samples, and for implementing corrective actions when procedure performance is not acceptable. They ensure that data are flagged as appropriate or an NCR is initiated if final reported QC sample results do not meet the acceptance criteria. LA-523-455 describes the actions to ensure that the of analytical data for total VOC analysis is of known and documented quality and quantity, specifies acceptance criteria for Project QC samples, and specifies corrective action measures to be taken when these criteria are not satisfied. Analytical personnel comply with the Project QA requirements and provide records to the SPO documenting the quality of the data generated. They implement all required QC practices summarized in Table 13-2.

Analytical personnel demonstrate acceptable performance through the analysis of method performance samples before analyzing any solid samples. They ensure that 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.

Analytical personnel analyze samples in analytical batches. 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. Matrix spikes, matrix spike duplicates, and surrogate compounds that do not meet acceptance criteria due to matrix interference effects are flagged as "Z" and a nonconformance report is not required.

Analytical 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 mid-range on the calibration.

Analytical personnel demonstrate ongoing laboratory performance through the analysis of laboratory control samples and meet the acceptance criteria listed in Table 13-2. They prepare laboratory control samples to contain at least 10 of the analytes listed in Table 13-1 in a non-interfering matrix, at a concentration of 20 µg/L from standards independent of those used for instrument calibration. Laboratory control sample concentrations are specified in analytical procedures and are carried through the same preparation procedures as samples. Analytical personnel also demonstrate acceptable laboratory performance by analysis of PDP blind-audit samples.

13.4 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Analytical personnel test, inspect, and maintain analytical equipment according to the QAPD, Section 13.4 of the QAPP, and applicable manufacturer's specifications. Analytical personnel use equipment and materials that meet 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.

Analytical personnel meet GC/MS instrument performance criteria before analyzing any standards or samples by tuning with perfluorotributylamine (PFTBA) and meeting the BFB criteria specified in "Semivolatile Sample Analysis by SW-846, Method 8270B" (LA-523-456). They check the BFB criteria at the beginning of each 12 hours of operation.

Analytical personnel equip gas chromatographs used for GC/FID analysis with two chromatographic columns selected from among those recommended by the method (based on availability). They periodically clean splitless glass injection liners with nitric acid to dissolve salts deposited from the injection of aqueous samples.

Table 13-2. Summary of Laboratory Quality Control Samples and Frequencies for Total 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 13-1 QAOs	Repeat until acceptable
Laboratory duplicates ^a	One (1) per analytical batch	Meet Table 13-1 RPDs	Nonconformance if RPDs > Table 13-1 values
Laboratory blanks	One (1) per analytical batch	Analyte concentrations <3x MDLs	Nonconformance if analyte concentration $\geq$ 3x MDLs ^b
Matrix spikes	One (1) per analytical batch	Meet Table 13-1 %Rs	Nonconformance if %Rs are outside the range specified in Table 13-1 ^c
Matrix spike duplicates	One (1) per analytical batch	Meet Table 13-1 RPDs and %Rs	Nonconformance if RPDs and %Rs > Table 13-1 values ^c
Laboratory control samples	One (1) per analytical batch	80–120 %R	Nonconformance if %R <80 or >120 ^d
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples for a given matrix $\pm$ 3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviations) or > (average %R + 3 standard deviations) ^c
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^aMay be satisfied using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table 13-1.

^bNonconforming laboratory blanks shall be flagged as "B" and an NCR is not required.

^cMatrix spikes, matrix spike duplicates, and surrogate compounds that do not meet acceptance criteria due to matrix interference effects shall be flagged as "Z" and an NCR is not required.

^dAn NCR per Section 2.1.2 of this QAPjP is required when laboratory control samples associated with final reported data do not meet acceptance criteria.

%R = percent recovery
MDL = method detection limit
NCR = nonconformance report
PDP = Performance Demonstration Program
QAO = quality assurance objective
QC = quality control
RPD = relative percent difference

13.5 INSTRUMENT CALIBRATION AND FREQUENCY

Analytical personnel ensure that all analytical instruments are calibrated before use and verify calibration at routine intervals. They ensure that all 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. Procedures listed in Section 13.7 describe laboratory calibration processes.

Analytical 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 BFB. They ensure that the continuing calibration standard meets all of the daily calibration criteria for surrogate compound recovery, system performance check compounds (SPCCs), calibration check compounds (CCCs), internal standard area count criteria, and RTs, as specified in Table 13-3. They choose SPCCs and CCCs common to Table 13-1. 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.

Analytical personnel perform an initial GC/FID calibration consisting of at least three analytical standards (and a blank), injected in triplicate, that define the calibration range of the instrument for those analytes being identified by GC/FID. They consider the initial calibration to be valid if for calibration curves, the computed correlation coefficient is greater than or equal to 0.93, or for response factors, the %RSD of all analytes is less than 35. They verify the GC/FID calibration curve using a midpoint calibration standard (the continuing calibration standard). They analyze the continuing calibration standard at the beginning of every 12 hours of operation. They ensure the continuing calibration standard meets the acceptance criteria for response factor or measured concentration %D and RTs as specified in Table 13-3. If any analyte in the continuing calibration standard does not satisfy the acceptance criteria for linearity and consistency, they prepare a new calibration curve or response factor for that analyte. They do not proceed with sample analysis until the GC/FID system has satisfied the appropriate daily calibration criteria.

13.6 DATA MANAGEMENT

WMH-310, Section 9.2, includes or references example forms (or describes the electronic system) used to record and report data. Analytical personnel reduce raw data to reportable results in accordance with "ACE Program Implementation and Operation of Spreadsheet Interface" (WMH-310, Section 9.1). The laboratory QA officer ensures that data are reviewed, validated, and verified, as described in section 3.1.1.

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

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 8240B or 8260A)
	5-pt initial calibration (5 standards)	Initially, and as needed	Response factor %RSD for CCCs ≤ 30 ; response factor for SPCCs $\geq 0.30^a$; average relative response factor is used if %RSD ≤ 15 ; linear regression equation is generated if %RSD > 15
	Continuing calibration	Every 12 hours	Response factor or conc. %D for CCCs ≤ 20 ; response factor for SPCCs $\geq 0.30^a$; RT for internal standards must be ± 30 seconds from last daily calibration check; internal standard area count must be > 50 percent or < 200 percent of the area counts from the last daily calibration check; surrogate compound %R must be met (see SW-846 Method 8240B or 8260A)
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

^aBromoform 0.10.

%D	=	percent difference
%RSD	=	percent relative standard deviation
BFB	=	4-bromofluorobenzene
CCC	=	calibration check compounds
GC/FID	=	gas chromatography/flame ionization detector
GC/MS	=	gas chromatography/mass spectrometry
RT	=	retention time
SPCC	=	system performance check compounds

Analytical personnel report nontarget compounds as TICs with a higher uncertainty than target analyte concentrations. They perform appropriate search routines of the latest NIST or equivalent mass spectral library on the 20 TICs with the greatest area counts and with total ion current peaks greater than 10 percent 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 percent of all samples from a given waste stream. Analytical personnel assume a relative response factor equal to one, using the nearest internal standard, when calculating concentration for TICs.

Analytical personnel submit analytical batch data reports to the Project records custodian. These records are subject to assessment by the SQAQO on a regular basis as discussed in Section 3.1.2. Analytical batch data reports include the following:

- Laboratory identifier, 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
- Table of contents
- COC report 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 identifier, Project 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 Project analytes, and analytical results in mg/kg on a wet weight basis. The following data qualifying flags are used:
 - B (analyte detected in blank above the acceptance criteria)
 - 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 (estimated, one or more QC samples do not meet acceptance criteria)
- NCRs or CARs, if applicable.

Analytical personnel provide records to the Project records custodian. The Project records custodian maintains the records, retrievable by analytical batch numbers and data report numbers, in accordance with WMH-400, Section 1.5.1. These records include the following:

- 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.

- A reference to instrument calibration reports that include the accepted and measured values of calibration verification for all analytes. 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

Table 13-4 lists the implementing procedures pertinent to this section.

Table 13-4. Section 13 Implementing Procedures.

Document number	Title
LA-523-134	<i>Volatile Organics by Gas Chromatography/Mass Spectrometry using SW-846</i>
LA-523-456	<i>Semivolatile Sample Analysis by SW-846, Method 8270B</i>
WMH-310, Section 9.1	<i>ACE Program Implementation and Operation of Spreadsheet Interface</i>
WMH-310, Section 9.2	<i>Laboratory Report Generation</i>
WMH-310, Section 8.2	<i>Analytical Report Review</i>
LA-325-106	<i>Mercury Analysis by Cold Vapor Atomic Absorption using the FIAS-400</i>
EPA 8260	<i>Volatile Organic Analysis by GS/MS</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>

14. TOTAL SEMIVOLATILE ORGANIC COMPOUND ANALYSIS

Analytical personnel receive solid samples from the homogeneous solids and soil/gravel sampling operations described in Section 8. They analyze the samples for total SVOCs and PCBs, as described in this section.

14.1 QUALITY ASSURANCE OBJECTIVES

Table 14-1 lists the QAOs for total SVOC and PCB analyses. Key data quality indicators are defined below, and Section 3.2 presents the methods used to assess compliance. To demonstrate compliance with the QAOs, analytical personnel perform the following:

- Measure precision by analyzing matrix spike duplicates, replicate analysis 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 that the MDLs are less than or equal to those listed in Table 14-1.
- Demonstrate the ability to quantitate at or below the PRQLs given in Table 14-1 by setting the concentration of at least one calibration standard below the PRQL.
- Measure adherence to the 90 percent 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 addition, sampling personnel ensure representativeness of samples through the use of standardized sampling methods described in Section 8.

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

14.2 METHODS REQUIREMENTS

Analytical personnel extract and analyze homogeneous solids and soil/gravel samples for total SVOCs in accordance with LA-523-456, based on Methods Manual Procedure 430.6. They analyze for PCBs in accordance with "Polychlorinated Biphenyls (PCBs) by Gas Chromatography" (LA-523-427).

Table 14-1. Semivolatile Organic Compounds 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 (%)
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 ^d	5	40	90
2,4-Dinitrotoluene	121-14-2	≤46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤319	D-152 ^d	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
PCBs						
Aroclor 1016 ^c	12674-11-2	≤33	50-114	5	40	90
Aroclor 1221 ^c	11104-28-2	≤110	15-178	5	40	90
Aroclor 1232 ^c	11141-16-5	≤128	10-215	5	40	90
Aroclor 1242 ^c	53469-21-9	≤49	39-150	5	40	90
Aroclor 1248 ^c	12672-29-6	≤55	38-158	5	40	90
Aroclor 1254 ^c	11097-69-1	≤62	29-131	5	40	90
Aroclor 1260 ^c	11096-82-5	≤56	8-127	5	40	90
Pyridine ^b	110-86-1	≤50	60-150	5	40	90

^aCriteria apply to PRQL concentrations.^bCan also be analyzed as a volatile organic compound.^cPCBs; required only for matrix parameter category S3220 (organic sludges).^dDetected; result must be greater than zero.

%R = percent recovery

%RSD = percent relative standard deviation

CAS = Chemical Abstract Service

MDL = method detection limit (maximum permissible value)

PCBs = polychlorinated biphenyls

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 percent analyte extraction

RPD = relative percent difference

14.2.1 Standards

Analytical personnel purchase certified primary standards from the best available source for target analytes. Commercially purchased primary standards are certified by the manufacturer. They prepare secondary analytical standards, surrogate standards, internal standards, calibration standards, matrix spiking standards, and QC standards as specified in LA-523-456 and LA-523-427. Spiking amounts are specified in analytical procedures and are generally assigned such that the matrix spike sample concentrations are greater than or equal to mid-range on the calibration.

14.2.2 Qualitative and Quantitative Analysis

Analytical personnel qualitatively identify analytes using GC/MS by ensuring the analytes elute within an RRT window of plus or minus 0.06 RRT unit of the continuing calibration standard RRT 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.

Analytical personnel qualitatively identify PCBs based on the analytes eluting within the RT windows determined by the most recent standards (calibration standards, continuing calibration verification standards, and/or laboratory control samples). Initially, they run at least three standards for the aroclors listed in Table 14-1 and define the RT windows around the mean absolute RT of the standards such that the occurrence of both false positive and false negative results are minimized. Analytical personnel also rely on pattern recognition to identify PCBs.

Analytical personnel use internal standards for quantitating target analytes by GC/MS. They meet the criteria for SPCCs and CCCs and calculate response factors and %RSD for all analytes. They meet all daily tune and calibration criteria for decafluorotriphenylphosphine (DFTPP), 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.

Analytical personnel use external standards for quantitating target analytes by GC/electron capture detection (GC/ECD). They base quantitation on the total area or height response measured from a common baseline under three to five major peaks for each aroclor. After confirming qualitative analysis, they develop a calibration curve for the appropriate aroclor or aroclor mixture.

14.3 QUALITY CONTROL

The laboratory manager and laboratory QA officer are responsible for monitoring and documenting procedure performance (including the analysis of QC samples) and for implementing corrective actions when procedure performance is not acceptable. They ensure that data are flagged, as appropriate, or that an NCR is initiated if final reported QC sample results do not meet acceptance criteria. LA-523-456 and LA-523-427 describe the actions to ensure that the daily quality of analytical data for total SVOC and PCB analyses is of known and documented quality and quantity, specify acceptance criteria for Project QC samples, and specify corrective action measures to be taken when these criteria are not satisfied. Analytical personnel comply with the Project QA requirements and provide records to the SPO. documenting the quality of the data generated They implement all required QC practices summarized in Table 14-2.

Analytical personnel demonstrate acceptable performance before analyzing any solid samples through the analysis of method performance samples. They ensure that method performance samples contain all analytes listed in Table 14-1 at concentrations appropriate to verify that 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.

**Table 14-2. Summary of Laboratory Quality Control Samples and Frequencies
for Total Semivolatile Organic Compounds 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 duplicates ^a	One (1) per analytical batch	Meet Table 14-1 QAOs	Nonconformance if RPDs > Table 14-1 values
Laboratory blanks	One (1) per analytical batch	Analyte concentrations < 3 x MDLs	Nonconformance if analyte concentrations $\geq 3x$ MDLs ^b
Matrix spikes	One (1) per analytical batch	Meet Table 14-1 %Rs	Nonconformance if %Rs are outside the range specified in Table 14-1 ^c
Matrix spike duplicates	One (1) per analytical batch	Meet Table 14-1 RPDs and %Rs	Nonconformance if RPDs and %Rs > Table 14-1 values ^c
Laboratory control samples	One (1) per analytical batch	80-120 %Rs	Nonconformance if %R < 80 or > 120 ^d
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples from a given matrix ± 3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviations) or > (average %R + 3 standard deviations) ^c
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^aMay be satisfied by using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table 14-1.

^bNonconforming laboratory blanks shall be flagged as "B" and an NCR is not required.

^cMatrix spikes, matrix spike duplicates, and surrogate compounds that do not meet acceptance criteria due to matrix interference effects shall be flagged as "Z" and an NCR is not required.

^dAn NCR per Section 2.1.2 of this QAPjP is required when laboratory control samples associated with final reported sample data do not meet Table 14-2 acceptance criteria.

%R	=	percent recovery
MDL	=	method detection limit
NCR	=	nonconformance report
QAO	=	quality assurance objective
QAPjP	=	Quality Assurance Project Plan
QC	=	quality control
PDP	=	Performance Demonstration Program
RPD	=	relative percent difference

Analytical personnel analyze samples in analytical batches. Specific QC samples for each analytical batch include a laboratory blank, a matrix spike, a matrix spike duplicate, a laboratory control sample, and surrogate spike compounds for SVOC analysis. Matrix spikes, matrix spike duplicates, and surrogate compounds that do not meet acceptance criteria due to matrix interference effects are flagged as "Z" and a nonconformance report is not required. Laboratory procedures detail the preparation and analysis of QC samples.

Analytical personnel choose surrogate compounds for SVOC analysis 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 three of the SVOCs and at least one aroclor (for PCB analysis only) listed in Table 14-1. Spiking amounts are specified in analytical procedures and are generally assigned such that the matrix spike sample concentrations are greater than or equal to mid-range on the calibration. SVOC and PCB spikes are at concentrations at the PRQLs listed in Table 14-1. Matrix spike results are acceptable if the Table 14-1 criteria for accuracy are met. Matrix spike duplicate results are acceptable if the Table 14-1 criteria for precision and accuracy are met.

Analytical 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 in methylene chloride to contain, at a minimum, 1,4-dichlorobenzene, 2,4-dinitrotoluene, hexachloroethane, and nitrobenzene at a concentration of 100 µg/L per laboratory control analyte. For PCBs, the laboratory control sample contains only the most representative mixture. Laboratory control sample concentrations are specified in analytical procedures. Laboratory control samples are carried through the same preparation procedures as samples. Analytical personnel also demonstrate acceptable laboratory performance by analysis of PDP blind audit samples.

14.4 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Analytical personnel test, inspect, and maintain analytical equipment according to the QAPD, Section 14.4 of the QAPP, and applicable manufacturer's specifications. Analytical personnel use equipment and materials that 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 to allow the detection and quantitation of all analytes listed in Table 14-1 and the identification of nontarget compounds.

Analytical personnel meet GC/MS instrument performance criteria before analyzing any standards or samples by tuning with PFTBA and meeting the DFTPP criteria specified in LA-523-456. They check the DFTPP criteria at the beginning of each 12 hours of operation by analyzing DFTPP.

Analytical personnel prime the GC/ECD system daily with a solution containing an aroclor at a concentration approximately 20 times the midpoint standard.

14.5 INSTRUMENT CALIBRATION AND FREQUENCY

Analytical personnel calibrate all analytical instruments before use and verify calibration at routine intervals. They ensure that all 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. Laboratory calibration procedures and records are described and identified in the cited analytical procedures.

Analytical 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, setting 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 instrument performance criteria using DFTPP. They ensure that the continuing calibration standard meets all daily calibration criteria for %D, surrogate compound recovery, SPCCs, internal standard area count criteria, and RTs, as specified in Table 14-3. They choose SPCCs and CCCs common to Table 14-1. 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 Semivolatile Organic Compounds 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 8250A or 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 ; linear 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 percent or < 200 percent of the area count from daily calibration check; surrogate compound %R must be met (see SW-846 Method 8250A or 8270B)
GC/ECD	3-pt initial calibration (3 standards)	Initially and as needed	Response factor %RSD ≤ 20
	Continuing calibration	Every 12 hours as needed	%D ≤ 15 for all analytes compared to initial calibration
<hr/> %D = percent difference %RSD = percent relative standard deviation CCC = calibration check compounds DFTPP = decafluorotriphenylphosphine GC/ECD = gas chromatography/electron capture detector GC/MS = gas chromatography/mass spectrometry RT = retention time SPCC = system performance check compounds			

For GC/ECD analysis, analytical personnel identify the appropriate analytes by qualitative analysis, then perform an initial multipoint calibration using at least three analytical standards that define the calibration range of the instrument. They use the average response factor for each analyte if the %RSD is less than or equal to 20. They use a midpoint calibration standard (continuing calibration standard) to verify the initial GC/ECD calibration curve at the beginning of every 12 hours of operation. They ensure that the continuing calibration standard meets the daily calibration criteria for %D, as specified in Table 14-3, and the RT of each analyte is ± 3 standard deviations from initial calibration.

14.6 DATA MANAGEMENT

WMH-310, Section 9.2 includes or references example forms (or describes the electronic system) to be used to record and report data. Analytical personnel reduce raw data to reportable results in accordance with "ACE Program Implementation and Operation of Spreadsheet Interface" (WMH-310, Section 9.1). The laboratory QA officer ensures that data are reviewed, validated, and verified as described in Section 3.1.1.

Analytical personnel quantify analytes using average relative response factors (or regressions as specified in Section 14.5) and do not blank-correct results. They report results of blanks run in association with samples separately. They report all SVOC and PCB results in mg/kg on a weight/wet-weight basis, limited to two significant figures.

Analytical personnel report nontarget compounds as TICs with a higher uncertainty than target analyte concentrations. They perform appropriate search routines of the latest NIST or equivalent mass spectral library on the 20 TICs with the greatest area counts and with total ion current peaks greater than 10 percent 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 percent of all samples from a given waste stream. Analytical personnel assume a relative response factor equal to one, using the nearest internal standard, when calculating concentrations for TICs.

Analytical personnel submit analytical batch data reports to the Project records custodian. These records are subject to assessment by the SQA on a regular basis, as discussed in Section 3.1.2. Analytical batch data reports, which are described in WMH-310, Section 9.1, include the following:

- Laboratory identifier, 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
- Table of contents
- COC report 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" for each sample in the analytical batch, including laboratory identifier, Project 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 Project analytes, and analytical results in mg/kg wet weight basis. The following data qualifying flags are used:
 - B (analyte detected in blank above acceptance criteria)
 - 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 (estimated, one or more QC samples do not meet acceptance criteria)
- NCRs or CARs, if applicable.

Analytical personnel provide records to the Project records custodian. The Project records custodian maintains the records, retrievable by analytical batch numbers and data report numbers, in accordance with WMH-400, Section 1.5.1. These records include the following:

- 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.
- A reference to 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

Table 14-4 lists the implementing procedures pertinent to this section.

Table 14-4. Section 14 Implementing Procedures.

Document number	Title
WMH-400, Section 1.5.1	<i>TRU Records Management</i>
LA-523-427	<i>Polychlorinated Biphenyls (PCBs) by Gas Chromatography</i>
LA-523-456	<i>Semivolatile Sample Analysis by SW-846, Method 8270B1</i>
WMH-310, Section 9.2	<i>ACE Program Implementation and Operation of Spreadsheet Interface</i>
WMH-310, Section 9.1	<i>Laboratory Report Generation</i>
LA-325-106	<i>Mercury Analysis by Cold Vapor Atomic Absorption using the FLAS-400</i>
EPA 8260	<i>Volatile Organic Analysis by GS/MS</i>
WMH-310, Section 8.2	<i>Analytical Report Review</i>

15. TOTAL METAL ANALYSIS

Analytical personnel receive solid samples resulting from the homogeneous solids and soil/gravel sampling operations described in Section 8. They analyze the samples for total metals in accordance with the analytical procedures listed in Table 15-4.

15.1 QUALITY ASSURANCE OBJECTIVES

Table 15-1 lists the QAOs for total metals analysis. Key data quality indicators are identified below, and Section 3.2 presents the methods used to assess compliance. To demonstrate compliance with the QAOs, analytical personnel perform the following:

- 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 $\mu\text{g/L}$, and ensure that IDLs are less than or equal to the 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 percent 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 QAPP requirements.

In addition, sampling personnel ensure representativeness through the use of standardized and approved sampling methods described in Section 8.

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

15.2 METHODS REQUIREMENTS

Analytical personnel prepare and analyze homogeneous solids and soil/gravel samples for total metals using acid digestion followed by spectrometric analysis. They follow the analytical procedures listed below, which cite and are based on the applicable Methods Manual procedures:

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 (g/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

^a ≤30 percent control limits apply when sample and duplicate concentrations are ≥ 10 x IDL for inductively coupled plasma/atomic emission spectroscopy (ICP/AES) and AA techniques, and ≥ 100 x IDL for inductively coupled plasma/mass spectrometry (ICP/MS) 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 percent solid samples, assuming a 100x dilution during digestion.

%R	=	percent recovery
%RSD	=	percent relative standard deviation
AA	=	atomic absorption
CAS	=	Chemical Abstract Service
ICP/AES	=	inductively coupled plasma/atomic emission spectroscopy
ICP/MS	=	inductively coupled plasma/mass spectrometry
IDL	=	instrument detection limit
PRDL	=	Program required detection limit (i.e., maximum permissible value for IDL)
PRQL	=	Program required quantitation limit
RPD	=	relative percent difference

- “Mercury Analysis by Cold Vapor Atomic Absorption” (LA-325-106) (Methods Manual Procedure 650.3)
- “Inductively Coupled Plasma (ICP) Emission Spectrometric Method for Jarrel Ash Type 61E” (LA-505-161 and LA-505-151) (Methods Manual Procedure 640.1)
- “Inductively Coupled Plasma (ICP) Emission Spectrometric Method for Applied Research Laboratories Model 3580/AdaM11 Upgrade” (LA-505-151) (Methods Manual Procedure 640.1)
- “Determination of Trace Elements and Radionuclides by Inductively Coupled Plasma-Mass Spectrometry using TJA Poems” (LA-506-101) (Methods Manual Procedure 630.1)
- “Metal Analysis by Graphite Furnace Atomic Absorption (GFAA) using the Perkin Elmer 5100PC” (LA-505-102) (Methods Manual Procedures 650.2, 650.4, and 650.6)

Sample preparation is described in “Acid Digestion/Dilutions of Aqueous Samples and Extracts” (LA-505-158), “Acid Digestion of Sediments, Sludges, and Soils for Inductively Coupled Plasma (ICP) and Atomic Absorption (AA) Analysis” (LA-505-159), and “Acid Digestion of Solid and Liquid Samples for Graphite Furnace Atomic Absorption Analysis (GFAA)” (LA-505-100) and is based on SW-846-Methods 3020A and 3050B (Methods Manual Procedure 610.1).

15.2.1 Standards

Analytical personnel purchase stock standard solutions or prepare these solutions from ultra-high-purity grade chemicals or metals (99.99 to 99.999 percent pure). All stock standard solutions are certified by the manufacturer and a certificate of analysis documenting traceability is maintained by the laboratory. They label and track commercial stock solutions to ensure that they are not used beyond their manufacturer-specified shelf life.

Analytical 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.

Analytical personnel prepare working calibration and QC standards for ICP/MS and ICP/AES at least weekly. They prepare working calibration and QC standards for graphite furnace atomic absorption spectroscopy (GFAA), cold-vapor atomic absorption spectroscopy, and hydride atomic absorption. The analytical procedures listed in Table 15-4 describe standards preparation.

15.2.2 Quantitative Analysis

All analytical instruments used to quantitate metal analytes meet Methods Manual and SW-846 requirements. Operating requirements are described in the analytical procedures listed in Table 15-4. Analytical personnel establish and document instrument settings on each applicable instrument for each analyte. They quantitate all analytes within the calibration range of the analytical instruments, diluting samples with concentrations greater than the calibration range of the instrument. They use a minimum of two integrations to quantitate all analytes and report the average.

Analytical procedures specify reagent purity, instrument operating conditions, background correction procedures, and interference detection and evaluation if applicable. These procedures describe background correction and the method of standard additions based on SW-846 guidance.

15.3 QUALITY CONTROL

The laboratory manager and laboratory QA officer are responsible for monitoring and documenting procedure performance, including the analysis of QC samples, and implementing the corrective actions when procedure performance is not acceptable. They ensure that data are flagged as appropriate or an NCR is initiated if final reported QC sample results do not meet the acceptance criteria. The analytical procedures describe the actions to ensure that the daily quality of analytical data for total metals analysis, specify acceptance criteria for Project QC samples, and specify corrective action measures to be taken when these criteria are not satisfied. Analytical personnel operate a formal QA program and maintain records to document the quality of the data generated. They implement all QC practices summarized in Table 15-2.

Analytical personnel demonstrate acceptable performance through the analysis of method performance samples before analyzing any solid samples. They ensure that method performance samples contain all analytes listed in Table 15-1 at concentrations appropriate to verify that 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 semi-annually by analyzing at least four method performance samples.

Analytical 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, and a laboratory control sample. Matrix spike duplicates are analyzed in place of laboratory duplicates.

Analytical 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 the analytical procedures and are generally assigned such that the matrix spike sample concentrations are greater than or equal to midrange on the calibration. If matrix spikes and matrix spike duplicates do not meet acceptance criteria due to matrix interference effects, they ensure that the data are flagged as "Z." 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 by analyzing PDP blind audit samples.

15.4 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Analytical personnel test, inspect, and maintain analytical equipment according to the QAPD, Section 15.4 of the QAPP, and applicable manufacturer's specifications. Analytical personnel use equipment and materials that meet SW-846 and Methods Manual requirements. They ensure that all the QAOs listed in Table 15-1 are met. They ensure that 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 analytical procedures.

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	$\leq 3 \times \text{IDL}$ ($\leq 5 \times \text{IDL}$ for ICP/MS) ^a	Redigest and reanalyze any samples with analyte concentrations which are $\leq 10 \times$ blank value and $\geq 0.5 \times \text{PRQL}$
Matrix spikes	One (1) per analytical batch	80–120 %R	Nonconformance if %R <80 or >120 ^b
Matrix spike duplicates	One (1) per analytical batch	RPD ≤ 30 80–120 %R	Nonconformance if RPD >30 or if %R <80 or >120 ^b
Laboratory control samples	One (1) per analytical batch	80–120 %R	Redigest and reanalyze for affected analytes ^c
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^a Applies only to concentrations greater than the PRQLs listed in Table 15-1.

^b Matrix spikes and matrix spike duplicates that do not meet acceptance criteria due to matrix interference effects shall be flagged as "Z" and an NCR is not required.

^c An NCR per Section 2.1.2 of the QAPjP is required when laboratory control samples associated with final reported data do not meet Table 15-2 acceptance criteria.

%R = percent recovery
 ICP/MS = inductively coupled plasma/mass spectrometry
 IDL = instrument detection limit
 PDP = Performance Demonstration Program
 PRQL = Program required detection limit
 QAO = quality assurance objective
 RPD = relative percent difference

15.5 INSTRUMENT CALIBRATION AND FREQUENCY

Analytical personnel ensure that all analytical instruments are calibrated before use and verify calibration at routine intervals as described in the analytical procedures. They ensure that 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.

Table 15-3. Summary of Calibration Requirements and Analysis Quality Control for Total Metals Analysis.

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
ICP/MS	Mass tune	Daily	4 Replicate %RSD <10; mass calibration within 0.1 amu; resolution <1.0 amu full width at 10 percent peak height
	1-pt. initial calibration (1 standard and a blank)	Daily	90-110 %R for independent calibration verification solution 95-105 %R for highest calibration stand
	Continuing calibration	Every 10 samples plus beginning and end of run	90-110 %R for check standard; blank m measure $\leq 5 \times$ IDL; internal standard area must be ≤ 20 %D of calibration blank int standard areas
	Interference correction verification	Beginning and end of run or every 12 hours, whichever is more frequent	Solution containing interferants only must measure $\leq 5 \times$ IDL for analytes; solution containing interferants plus analytes must within 80-120 %R for all analytes
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	5x dilution of sample which is $>100 \times$ ID must be ≤ 10 %D of initial value
	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
	Internal standard area verification	Every sample	Area must be ≤ 30 %D of calibration blank internal standard areas
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 stand

Table 15-3. Summary of Calibration Requirements and Analysis Quality Control for Total Metals Analysis (continued).

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
GFAA	Continuing calibration	Every 10 samples plus beginning and end of run	90-110 %R for check standard; blank m measure $\leq 3 \times$ IDL
	Interference correction verification	Beginning and end of run or twice per 8 hours, whichever is more frequent	Solution containing interferants only must measure $\leq 3 \times$ IDL for analytes; solution containing interferants plus analytes must 80-120 %R for all analytes
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	5x dilution of sample which is $>50 \times$ IDL must be ≤ 10 %D of initial value
	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
	3-pt. initial calibration (3 standards and a blank)	Daily	90-110 %R for independent initial calibration verification solution 95-105 %R for highest calibration stand Regression coefficient (r^2) must be ≤ 0.99
	Continuing calibration	Every 10 sample injections plus beginning and end of run	80-120 %R for check standard (mandatory blank (optional) should measure $\leq 3 \times$ IDL
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	5x dilution of sample which is $>25 \times$ IDL must be ≤ 10 %D IDL
	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch	85-115 %R

Table 15-3. Summary of Calibration Requirements and Analysis Quality Control for Total Metals Analysis (Concluded).

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
CVAA	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 stand Regression coefficient (r^2) must be ≥ 0.99
	Continuing calibration	Every 10 samples plus beginning and end of run	80–120 %R for check standard (mandatory blank (optional) should measure $\leq 3 \times$ IDL)
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	5x dilution of sample which is $>25 \times$ IDL must be ≤ 10 %D of initial value
	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch	85–115 %R

%D	=	percent difference
%R	=	percent recovery
%RSD	=	percent relative standard deviation
CVAA	=	cold vapor atomic absorption spectroscopy
GFAA	=	graphite furnace atomic absorption spectroscopy
ICP/AES	=	inductively coupled plasma/atomic emission spectroscopy
ICP/MS	=	inductively coupled plasma/mass spectroscopy
IDL	=	instrument detection limit
MSA	=	method of standard additions

15.6 DATA MANAGEMENT

WMH-310, Section 9.1, and WMH-310, Section 9.2, describe data reduction, validation, and reporting processes and include or reference example forms to be used to record and report data. Analytical personnel reduce raw data to reportable results. They report total metals quantitative values in mg/kg wet weight basis, limited to two significant figures. The laboratory QA officer ensures that data are reviewed, validated, and verified, as described in Section 3.1.1.

Analytical personnel submit analytical batch data reports to the Project records custodian. These records are subject to assessment by the SQAQO on a regular basis as discussed in Section 3.1.2. Analytical batch data reports, which are described in WMH-310, Section 9.2, include the following:

- Laboratory identifier, 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
- Table of contents
- COC report 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 identifier, Project 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 Project 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 percent of the sample concentration prior to dilution correction)
 - H (Holding time exceeded)
 - J (Analyte is greater than or equal to PRDL but less than five times the IDL before dilution correction)
 - U (Analyte was undetected (report IDL corrected for dilution))
 - Z (Estimated, one or more QC samples do not meet acceptance criteria)
- NCRs or CARs, if applicable

Analytical personnel provide records to the Project records custodian. The Project custodian maintains the records, retrievable by analytical batch numbers, in accordance with WMH-400, Section 1.5.1. These records include the following:

- 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.
- A reference to 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

Table 15-4 lists the implementing procedures pertinent to this section.

Table 15-4. Section 15 Implementing Procedures.

Document number	Title
LA-325-106	<i>Mercury Analysis by Cold Vapor Atomic Absorption</i>
LA-505-100	<i>Acid Digestion of Solid and Liquid Samples for Graphite Furnace Atomic Absorption Analysis (GFAA)</i>
LA-505-102	<i>Metal Analysis by Graphite Furnace Atomic Absorption (GFAA) using the Perkin Elmer 5100PC</i>
LA-505-151	<i>Inductively Coupled Plasma (ICP) Emission Spectrometric Method for Applied Research Laboratories Model 3580/AdaM11 Upgrade</i>
LA-505-158	<i>Acid Digestion/Dilutions of Aqueous Samples and Extracts</i>
LA-505-159	<i>Acid Digestion of Sediments, Sludges, and Soils for Inductively Coupled Plasma (ICP) and Atomic Absorption (AA) Analysis</i>
LA-505-161	<i>Inductively Coupled Plasma (ICP) Emission Spectrometric Method for Jarrel Ash Type 61E</i>
LA-506-101	<i>Determination of Trace Elements and Radionuclides by Inductively Coupled Plasma-Mass Spectrometry using TJA Poems</i>
WMH-310, Section 9.1	<i>ACE Program Implementation and Operation of Spreadsheet Interface</i>
WMH-310, Section 9.2	<i>Laboratory Report Generation</i>
WMH-310, Section 8.2	<i>Analytical Report Review</i>
LA-325-106	<i>Mercury Analysis by Cold Vapor Atomic Absorption using the FIAS-400</i>
EPA 8260	<i>Volatile Organic Analysis by GS/MS</i>
WMH-400, Section 1.5.1	<i>TRU Records Management</i>

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