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FINAL DRAFT

QUALITY ASSURANCE PROGRAM PLAN FOR THE WASTE ISOLATION PILOT PLANT EXPERIMENTAL-WASTE CHARACTERIZATION PROGRAM

U.S. Department of Energy
Washington, D.C.

Revision 1.0
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U.S. Department of Energy
Washington, D.C.

Approved by _____ Date _____

Associate Director of the Office of
Waste Operations (EM-30)

Approved by _____ Date _____

Director of the Office of Quality Assurance
and Quality Control (EM-20)

Approved by _____ Date _____

WIPP Project Manager (DOE/WPO)

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1.0 PROGRAM DESCRIPTION

This Quality Assurance Program Plan (QAPP) identifies the quality of data necessary to meet the specific objectives associated with the Department of Energy (DOE) Waste Isolation Pilot Plant (WIPP) Experimental-Waste Characterization Program (the Program). In accordance with the American Society of Mechanical Engineers (ASME) NQA-1, *Quality Assurance Program Requirements for Nuclear Facilities* (ASME, 1989), Element 2, this section describes the scope of the Program, the controls required for those activities affecting quality, and the required indoctrination and training of personnel performing activities affecting quality.

DOE plans to conduct experiments in the WIPP during a Test Phase of approximately 5 years (USDOE, 1990a). These experiments will be conducted to reduce the uncertainties associated with the prediction of several processes (e.g., gas generation) that may influence repository performance. The results of the experiments will be used to assess the ability of the WIPP to meet regulatory requirements for the long-term protection of human health and the environment from the disposal of TRU wastes.

This program is only one part of the WIPP Test Phase, both in the short- and long-term, to quantify and evaluate the characteristics and behavior of transuranic (TRU) wastes in the repository environment. Other parts include the bin-scale and alcove tests, drum-scale tests, and laboratory experiments (Figure 1-1). In simplified terms, the purpose of the Program is to provide chemical, physical, and radiochemical data describing the characteristics of the wastes that will be emplaced in the WIPP, while the remaining WIPP Test Phase is directed at examining the behavior of these wastes in the repository environment.

Specifically, this plan:

- Sets forth the data quality requirements that each DOE facility must meet in characterizing TRU wastes intended for inclusion in experimental activities associated with the WIPP Test Phase;
- Addresses the data quality requirements to comply with the Conditional No-Migration Determination;
- Addresses data quality requirements associated with the WIPP's verification of data provided by DOE generator/storage sites in fulfillment of waste characterization requirements established to comply with Resource Conservation and Recovery Act (RCRA) regulations (40 CFR §§265.13 and 264.13); and

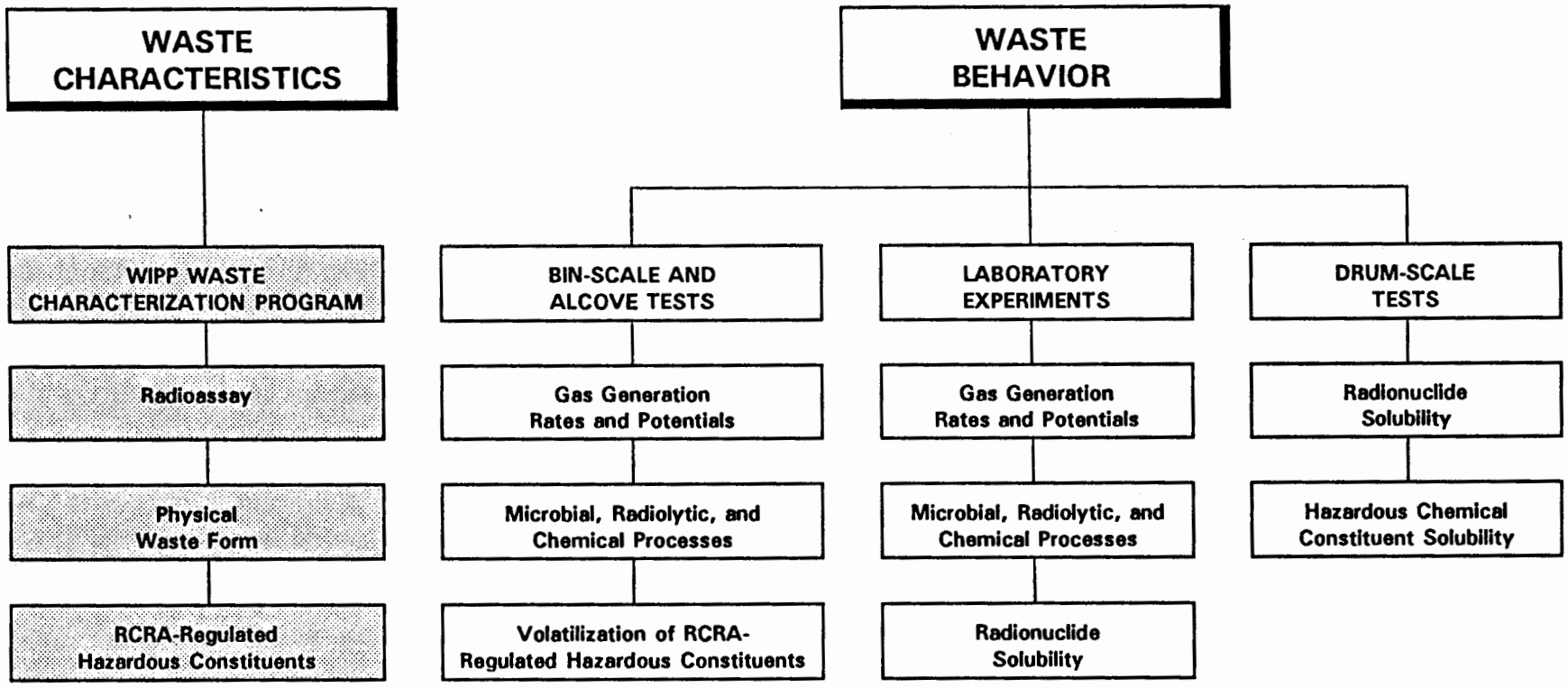

Figure 1-1. Aspects of WIPP Activities Related to Waste Characterization and Test Phase Programs

- Establishes the performance criteria for site Quality Assurance Project Plan (QAPjP) preparation, review, and approval. QAPjPs are site-specific documents that address compliance with the quality assurance requirements provided herein.

DOE Order 5700.6B (USDOE, 1986) requires that DOE facilities managing nuclear materials comply with all applicable quality elements in ASME NQA-1. This program addresses all applicable Elements of ASME NQA-1 unless noted by exception. Any exception to the ASME NQA-1 Elements and their supplements shall be documented in the QAPjPs. This document follows the guidelines recommended by the Environmental Protection Agency (EPA) in its QAMS-005 (USEPA, 1983). A cross reference of QAMS-005, as reflected in this QAPP, and the analogous ASME NQA-1 requirements are provided in Table 1-1. In addition, to assist in the review and identification of the DOE quality requirements specific to ASME NQA-1, the individual elements are noted in the appropriate sections of the QAPP.

Nothing in this document relieves any program participant from the responsibility of complying with applicable federal, state, and local regulations; DOE Orders; existing permits and interagency agreements; or any site-specific Controls on operations. EM-30 shall immediately be notified of any conflicts between the document and any existing requirements.

Program minimum requirements that are mandatory for program participants are specified throughout this document by the use of the terms "shall" or "must." Information that is provided as guidance that constitutes an acceptable means of accomplishing a task is designated by the term "should." The methods included in the "WIPP Waste Characterization Program Sampling and Analysis Guidance Manual," hereafter referred to as the Guidance Manual, are not mandatory. The Guidance Manual contains recommended practices that have been found to be acceptable in achieving the performance requirements for a given task. If a program participant chooses to use other methods, the burden of proof of the efficacy of the methods chosen will be on the program participant.

1.1 Program Overview

The Program is multi-faceted and will provide data necessary to meet a number of objectives. From a programmatic viewpoint, it encompasses the characterization of wastes at DOE TRU waste generator/storage sites, and the verification of this data by WIPP, prior to waste emplacement in the repository for experimental purposes during the Test Phase. Its scope also includes the acquisition of data necessary to support the WIPP operational phase, to the extent that wastes disposed of at WIPP during that period will be limited to those that are comparable in certain characteristics to the wastes that have been tested. The final results from the test program are expected in 3 to 5 years after

Table 1-1. Cross Reference for ASME NQA-1 Elements to Applicable Sections of the QAPP

ASME NQA-1 Elements	Section of the QAPP
1 Organization	*2.0 Organization and Responsibility
2 Quality Assurance Program	*1.0 Program Description 1.8 Indoctrination and Training *14.0 Quality Assurance Reports to Management
3 Design Control	*3.0 Quality Assurance Objectives
4 Procurement Document Control	1.7 Procurement Document Control/Control of Subcontractors
5 Instructions, Procedures, and Drawing	*4.0 Sampling Procedures
6 Document Control	1.5 Document Review, Approval, and Control 1.9 Site Quality Assurance Project Plans
7 Control of Purchase Items and Services	1.7 Procurement Document Control/Control of Subcontractors
8 Identification and Control of Items	1.7 Procurement Document Control/Control of Subcontractors *5.0 Sample Custody
9 Control of Processes	1.7 Procurement Document Control/Control of Subcontractors *7.0 Analytical Procedures *9.0 Internal QC Checks and Frequency
10 Inspection	1.11 Analytical Laboratory Performance Demonstration Program 11.0 Preventive Maintenance
11 Test Control	7.2 Radioassay (software) *9.0 Internal QC Checks and Frequency *12.0 Specific and Routine Procedures to Assess Data Quality
12 Control of Measuring and Test Equipment	*6.0 Calibration Procedures and Frequencies *11.0 Preventive Maintenance
13 Handling, Storage, and Shipping	*4.0 Sampling Procedures *5.0 Sample Custody
14 Inspection, Test, and Operating Status	*6.0 Calibration Procedures and Frequencies
15 Control of Nonconforming Items	*13.0 Corrective Actions
16 Corrective Action	*13.0 Corrective Actions *14.0 Quality Assurance Reports to Management
17 Quality Assurance Records	1.6 Quality Assurance Records *8.0 Data Reduction, Validation, and Reporting
18 Audits	*10.0 Performance and System Audits and Frequency

* These sections are in accordance with EPA QAMS-005 guidelines.

initiation of the bin-scale tests (USDOE, 1990a). The operational phase for the WIPP extends approximately 20 years after completion of the Test Phase (USDOE, 1990a).

From a regulatory compliance viewpoint, the Program addresses several data needs. These are associated with the following regulatory compliance programs:

- The *performance assessment* conducted to evaluate long-term radionuclide containment as required by the Environmental Radiation Protection Standards for Management and Disposal of Spent Nuclear Fuel, High-level, and Transuranic Radioactive Wastes (40 CFR Part 191, Subpart B). The performance assessment effort requires that physical and radiologic data describing the waste be obtained through the Program to support the interpretation of bin-scale and alcove test results.

- The *no-migration demonstration* required by 40 CFR §268.6 with regard to the containment of hazardous chemical constituents. Requirements applicable to the Test Phase are specifically identified in the WIPP's conditional no-migration determination (55 FR 47700), and includes quantification of hazardous constituents identified through the Program to support both compliance with the determination as well as a future efforts by DOE to obtain modification of the determination by EPA to allow disposal of waste at the WIPP after completion of the Test Phase.

  [Note that the final requirements of the conditional no-migration determination were established during preparation of this draft of the QAPP. A future revision to the QAPP will more thoroughly address specific waste characterization requirements required by the determination.]

- *General waste analysis*, specified in 40 CFR §§265.13 and 264.13, with regard to verification of waste characterization data provided by DOE generator/storage sites that ship wastes to WIPP. The data provided under the Program to meet these requirements will, to some extent, overlap or supplement data obtained to support both the performance assessment and no-migration demonstration.

  [Note that the process for establishing these waste characterization requirements are specifically addressed in the WIPP RCRA Part B permit application, currently in preparation. Future revisions of the QAPP will include the QA/QC requirements for the sampling and analytical methods in support of RCRA waste characterization that are established in the WIPP RCRA Waste Analysis Plan.]

From a technical perspective, then, this plan addresses the activities associated with TRU waste characterization efforts in general, and applies to all such activities conducted under the Program. All waste characterization activities conducted under the Program are collectively addressed under this QAPP, and are differentiated only when necessary to support specific data quality rationale.

1.2 Overview of the WIPP Test Phase

Both the performance assessment and no-migration determination require that the long-term waste containment abilities of the repository be evaluated. In this regard, the potential for and rate at which

gas generation by emplaced wastes will occur, resulting from various chemical, microbial, and radiolytic processes, are considered important factors and are the primary focus of the bin-scale and alcove tests that will be performed during the Test Phase (Molecke, 1990a, 1990b; Molecke and Lappin, 1990). The results of these experiments will be used in assessing what, if any, modifications to operations, the facility, or the waste may be necessary to ensure compliance with applicable regulations.

The specific experimental activities and sampling/analytical methods that will be implemented at WIPP during the Test Phase are not addressed in this document, but rather are described in site-specific documents (the QAPjPs) and the Sandia National Laboratories' (SNL's) Test Plans (Molecke, 1990a, 1990b; Molecke and Lappin, 1990). For additional information describing the rationale for and scientific basis of the WIPP experimental program, several primary documents may be consulted:

- Molecke, M.A., 1990a, "Test Plan: WIPP Bin-Scale CH TRU Waste Tests," SAND90-1974, Sandia National Laboratories, Albuquerque, New Mexico.
- Molecke, M.A., and A.R. Lappin, 1990, "Test Plan Addendum #1: WIPP Bin-Scale CH TRU Waste Tests," SAND90-2082, Sandia National Laboratories, Albuquerque, New Mexico.
- Molecke, M.A., 1990b, "Test Plan: WIPP In Situ Alcove CH TRU Waste Tests," Sandia National Laboratories, Albuquerque, New Mexico.
- Lappin, A.R., C.A. Gotway, M.A. Molecke, and R.L. Hunter, 1991, "Rationale for Revised WIPP Bin-Scale Gas-Generation Tests with CH TRU Wastes," SAND90-2481, Sandia National Laboratories, Albuquerque, New Mexico (in preparation).
- Department of Energy, 1991, "Waste Characterization Program Plan for WIPP Experimental Waste," DOE/WIPP 89-025, Final Draft, Revision 0.0. Waste Isolation Pilot Plant, Carlsbad, New Mexico.
- Marietta, M. G., et al., 1989, "Performance Assessment Methodology Development for Evaluating Compliance with EPA 40 CFR 191, Subpart B, for the Waste Isolation Pilot Plant," SAND89-2027, Sandia National Laboratories, Albuquerque, New Mexico.

In addition to the collection of data necessary to evaluate long-term repository integrity, the RCRA-regulated, hazardous component of TRU mixed waste that will be emplaced at WIPP must be characterized in accordance with established EPA requirements and guidelines (40 CFR §§265.13 and 264.13), which ensure that the general waste characterization data obtained are representative and meet quality assurance objectives. The DOE facilities shipping mixed wastes to WIPP will bear much of the responsibility for complying with these requirements, although the WIPP will verify that specific waste characterization objectives (accuracy and reporting, for example) are met.

1.3 Technical Approach to the Program

The Program focuses initially on the characterization of wastes generated by the Rocky Flats Plant (RFP) in that they are expected to be representative of the majority of the TRU waste inventory in terms of gas generation potential. They are considered representative of nearly all TRU wastes because they contain the full spectrum of waste materials (e.g., cellulosics, plastics, and metals) identified in TRU wastes throughout the DOE complex (Molecke, 1990a, 1990b; Molecke and Lappin, 1990). Some of the wastes generated by RFP will be shipped from the Idaho National Engineering Laboratory (INEL), where they are currently in storage.

The bin-scale and alcove tests at the WIPP have been developed by Sandia National Laboratories (SNL) (Molecke, 1990a; 1990b). The specific types and quantities of TRU wastes that are required for the tests are the responsibility of SNL (Lappin et al., 1991). SNL will provide each DOE generator/storage facility participating in the Program with the types of waste by TRUCON Content Codes (Table 3-2) that must be included in the bin tests. SNL has determined the types and quantities of waste required to represent the TRU waste inventory in terms of the parameters that may affect the rate and potential for gas generation. Based on the results from the initial phase of bin testing, SNL will provide further guidance on any additional types and quantities of waste that may need to be tested in bins or alcoves.

Each facility will select specific waste containers in accordance with SNL requirements from their inventory of TRU waste that are WIPP WAC and TRAMPAC certified. Any deviations or changes from the SNL requirements for waste types included in the tests will require approval by SNL prior to implementation.

The wastes will be characterized prior to shipment to WIPP by the facility originating the shipment. The data provided by RFP and INEL will be required for the interpretation of bin-scale and alcove test results with regard to gas generation.

Other DOE facilities shipping waste to WIPP will be required to provide similar waste characterization data to demonstrate the comparability of each waste stream in terms of gas generation variables (i.e., physical waste matrices and total alpha curies) to those wastes used in the experiments. The specific parameters and number of containers that must be sampled to demonstrate waste comparability will be determined by SNL. This determination will be made by SNL after completion of the test program, and will be based on a statistically-valid sampling plan and results obtained from the experimental program. A detailed description of the methodology and the quantities and types of wastes that will be included in the test program is included in Lappin et al. (1991).

To meet RCRA requirements, each generator/storage facility will in addition be required to evaluate its TRU mixed waste inventory and determine the applicable analyses required to properly characterize each waste stream. The primary criteria used to determine the requirements for the types and frequency of analyses will include the physical form of the waste, the rate and consistency of waste generation, and the hazardous constituents identified through process knowledge. A site-specific RCRA Waste Analysis Plan for TRU waste will be prepared by each generator/storage facility and will be subject to approval by each facility's State regulatory authority. To ensure that waste characterization data are representative, as required by RCRA, each facility will be required to develop a statistically-valid sampling program, as part of its RCRA Waste Analysis Plan, that identifies the specific wastes and the number of containers that will be sampled.

This QAPP shall be revised, as necessary, to ensure that it addresses the quality assurance (QA) requirements applicable to the characterization of all waste forms and experimental parameters that may be included in the experimental program, as well as any sampling and analytical methodologies applicable to mixed waste that may be approved by EPA to facilitate RCRA waste characterization efforts.

1.4 Data Quality Objectives

As previously described, the data obtained through the Program will be used in efforts to ensure that the WIPP project meets regulatory requirements with regard to repository integrity, compliance with the conditional no-migration determination, and that all wastes are properly managed during both the Test Phase and operational phase. The DQOs established for the Program are intended to support these efforts and address the specific waste characterization parameters that will be evaluated, which are identified in Table 1-2. The DQOs are:

<u>Radioassay (RA)</u>

To determine the total alpha activity in each container of waste to assess the potential for radiolytic gas generation.

<u>Real-time radiography (RTR) and visual examination</u>

To determine the waste materials and their weights for those materials that may influence the potential for gas generation.

To verify process knowledge in terms of the Content Code, Item Description Code, physical waste form, and quantity of free liquids.

Table 1-2. Summary of Waste Characterization Requirements and Program Design

PARAMETER	Total Alpha	Waste Descriptors	Headspace Gases and Volatile Organic Compounds
		Content Code/Item Description Code Packaging Configuration Waste Material Category Cellulosics Plastics Rubber Materials Corroding Metals/Steel Corroding Metals/Aluminum Non-Corroding Metals Solid Inorganics Inorganic Sludges Cements/Additives Organics (resins, oils, organic sludges, solvents)	Gases N_2, O_2 H_2, NO_x CO, CO_2 Ar, CH_4 C_2H_6, C_3H_8 Volatile Organic Compounds (VOCs) Carbon tetrachloride, 1,1-Dichloroethane 1,1,1-Trichloroethane, 1,2-Dichloroethane Methylene chloride, 1,1-Dichloroethene 1,1,2-Trichloro-1,2,2-trifluoroethane, cis-1,2-Dichloroethene Ethyl benzene Acetone, 4-Methyl-2-pentanone Methanol, 1,1,2,2-Tetrachloroethane Trichloroethylene, Tetrachloroethene Ethyl ether, Toluene Benzene, 1,3,5-Trimethylbenzene Bromoform, 1,2,4-Trimethylbenzene Butanol, m-Xylene 2-Butanone, o-Xylene Chlorobenzene, p-Xylene Chloroform, Cyclohexane
TECHNIQUES	Radioassay [a] Passive-Active Neutron Segmented Gamma Scan Passive Neutron Coincidence Counting	Waste Examination Non-destructive [a]: Real-time Radiography Destructive [b]: Visual Examination	Headspace Gas Analysis [b] Gas Mass Spectroscopy (MS) Gas Chromatography (GC) Gas Chromatography/Mass Spectrometry (GCMS) Spectrophotometric Methods or Equivalent [c]
DATA QUALITY OBJECTIVES	• Gas Generation Rate and Potential	• Verification of Process Knowledge • Physical Waste Forms • Gas Generation Rate and Potential	Gases • Flammability • Initial Processes: - radiolysis - microbial - chemical VOCs • Flammability • Process Knowledge Verification • No Migration of Hazardous Constituents

[a] Conducted for all wastes.
[b] Conducted for all wastes included in bin-scale tests and a statistical population of wastes included in alcove tests and operational phase.
[c] For NO_2 and NO only.

Gas sampling and analyses

To determine the concentrations of gases in containers of waste to identify the existing, dominant gas generation processes.

To measure concentrations of hydrogen and methane to ensure that no containers with potentially flammable gas mixtures are emplaced within the repository. (A flammable hydrogen-methane mixture is defined by EPA in the no-migration determination as one that exceeds 50 percent of its lower explosive limit.) [Note: this objective will be addressed in future revisions to this plan when procedures are developed for calculating or measuring the lower explosive limit.]

Volatile organic compound (VOC) sampling and analyses

To determine the concentrations of VOCs in the headspace of TRU waste containers to support future revisions of the WIPP No-Migration Variance Petition (USDOE, 1990b) in addressing potential contaminant transport through the air pathway during the operational phase.

To verify the comparability of data describing the dominant VOCs present in the headspace of TRU waste containers to data previously provided in the WIPP No-Migration Variance Petition. [Note: this objective will be addressed in future revisions to this plan as procedures are developed to obtain headspace samples from inner bags of waste.]

To determine if the concentrations of flammable VOCs are greater than 500 ppmv in the headspace of waste containers and, if so, test the potential flammability of the mixture. [Note: this objective will be addressed in future revisions to this plan when a "flame test" is developed.]

RCRA-regulated hazardous waste characterization

To characterize the wastes to be sent to WIPP in accordance with 40 CFR Part 261, Subparts C and D. [Note: this objective will be addressed in future revisions to this plan as sampling and analytical methods are developed.]

1.5 Document Review, Approval, and Control

In accordance with ASME NQA-1, Element 6, the preparation, issue, and change to documents that specify quality requirements or prescribe activities affecting quality for the Program shall be controlled to assure that correct documents are used and referenced. Such documents, including changes thereto, shall be reviewed for adequacy and approved for release by authorized personnel. The QA for the Program is described within a hierarchy of documents. This QAPP includes the performance-based

quality assurance/quality control (QA/QC) requirements that each facility participating in the Program must comply with. All activities in this program affecting quality shall be performed in accordance with written and approved instructions, procedures, or drawings as appropriate to the activity. These documents shall include or reference appropriate qualitative or quantitative criteria for determining that the activities have been satisfactorily accomplished. When activities requiring written instructions are discussed, the required documents are referred to as "SOPs" (standard operating procedures) throughout this QAPP. The organization, format, content, and designation of such instructions will be described in the QAPjPs.

The site QAPjPs provide detailed and comprehensive statements to implement the QA/QC requirements. Each DOE facility managing TRU waste shall prepare a site QAPjP in compliance with this QAPP. In addition, each site shall have SOPs that include the detailed descriptions of procedures for performing the Program tasks. Each site also must have a laboratory QA plan that includes the QA/QC requirements and procedures for the analytical laboratories. All documents pertaining to the Program must be controlled as described in this subsection. Table 1-3 includes the organization(s) and person(s) responsible for review, approval, implementation, and change approval and control of the QAPP and the QAPjPs.

1.5.1 Review and Approval of Documents

Review of the all quality documents for the Program shall be prior to approval and issuance and shall consider, as appropriate, the technical adequacy, completeness, and correctness of the documents and the inclusion of appropriate quality requirements. Approval shall be indicated by a signature and date page included in each document. Whenever the documents are revised, review and approval of the revision shall be conducted by the same level of approval authority and in accordance with the requirements of review as the original documents.

Review and Approval of the QAPP

This QAPP shall be initially reviewed and approved, and thereafter at least annually reviewed, by the Associate Director of the Office of Waste Operations (EM-30), the Director of the Office of Quality Assurance and Quality Control (EM-20), and the DOE/WPO. It is the responsibility of DOE/EM-30 to schedule and coordinate the annual review.

Review and Approval of the QAPjPs and Supporting SOPs and QA Plans

The site QAPjPs, at a minimum, shall be initially reviewed and approved by the site DOE Operations Office Manager, the WIPP Waste Acceptance Criteria Certification Committee (WACCC) Chairperson,

Table 1-3. Minimum Requirements for Review, Approval, Implementation, and Control of the QAPP and QAPjPs

Responsible Party	DOE/EM-30	DOE/EM-20	DOE/WPO	WIPP/ WACCC	Sandia National Laboratories	DOE Operations Office Manager	Site Project Manager	Site Project Quality Assurance Officer
QAPP								
Review				X	X	X		
Review/Approval	X	X	X					
Implementation[a]				X				
Change Approval	X	X	X					
Change Control	X							
QAPjPs								
Review			X					
Review/Approval				X		X	X	X
Implementation[b]							X	
Change Approval				X		X	X	X
Change Control							X	

[a] The WACCC implements the requirements of the QAPP by its review and approval of the site-specific QAPjPs and by auditing site activities for compliance with the Program QC/QC requirements.

[b] The QAPjPs shall include a description of the line management responsibilities for implementation of the Program requirements. The site Project Manager is responsible for the daily implementation of the Program requirements.

the site Project Manager and the site Project Quality Assurance (QA) Officer. The QAPjPs shall be reviewed at least annually by the site Project Manager. Based on the annual review, if changes to the QAPjP are required, the site Project Manager shall be responsible for scheduling and coordinating the review and approval of the revised document. Each DOE site shall develop and implement a system for the approval of SOPs in compliance with the requirements established by the DOE Operations Office and the operating contractor. Each site also shall develop and implement procedures for the review and approval of the analytical laboratory QA plans in accordance with applicable site documentation.

1.5.2 Document Control

The QAPP shall be distributed by DOE/EM-30 to the applicable DOE Operations Offices. Distribution lists for this QAPP shall be used to control the issuance of revisions and shall be maintained by EM-30.

The site QAPjPs shall include a description of the responsible organization(s) or person(s) for distribution and issuance of revisions to those plans. Sites shall develop and implement procedures for the control of QAPjPs and SOPs in accordance with the requirements of the DOE Operations Office and operating contractor. All members of the site project staff are responsible for reporting any obsolete or superseded information to the site Project Manager. Each site shall include in its QAPjP a description of the procedures for the revision of its project plan.

1.5.3 Change Control

Change from original, approved documents, procedures, and specifications are expected during the course of WIPP's Test Phase and throughout its operational phase. Change does not imply a nonconformance to the work, but rather that original plans must be altered because of new information obtained or events that occur during the Program.

Changes to the QAPP

Changes to the activities or objectives specified in this QAPP shall be reviewed and approved by DOE/EM-30, DOE/EM-20, and DOE/WPO prior to initiation of the change at the applicable DOE facilities. Changes shall be reported by DOE/EM-30 to the DOE Operations Offices for notification to the sites of the required change. Change shall be documented, evaluated, and reported as necessary. The site Project Manager shall be responsible for revision of the QAPjP and SOPs in accordance with the approved change.

As a minimum, revisions to documents shall be denoted by including the current revision number on the document title page, the revised signature page, and each page that has been revised. The revision number and date of the revision shall be indicated on each revised page. Only pages which are revised need to be reissued. A vertical bar, indicating the change to the text, shall be included along the right-hand margin of the page.

Changes to the QAPjPs

All members of the site project staff are responsible for reporting any obsolete or superseded information to the site Project Manager. Each site shall include in its QAPjP a description of the procedures for the revision of its project plan.

The site QAPjPs shall include a detailed description of the reporting and approval requirements for changes to approved documents or procedures. All site-specific changes shall be evaluated and approved by the site Project Manager and the site Project QA Officer prior to implementation. The site Project Manager shall notify the appropriate personnel and affected documents shall be revised as necessary to reflect the work as actually performed. The site Project Manager shall be responsible for notification of the change to the DOE Operations Office Manager. No changes that can affect the performance criteria or data quality shall be made without prior approval of the DOE Operations Office Manager, the WACCC, and DOE/EM-30.

1.6 Quality Assurance Records

In accordance with ASME NQA-1, Element 17, this section specifies the requirements and responsibilities for QA/QC records transmittal, distribution, retention, maintenance, and disposition. A data/records management system shall be defined, implemented, and enforced by each DOE site in accordance with written procedures to maintain evidence of the conduct and quality of the work. This system shall include the management of field, laboratory, and central office project record files at each DOE generator/storage site. It also shall provide for the compilation of all QA/QC records generated for each waste and the transfer of specified records to DOE/WPO.

The data/records management systems shall provide adequate control and retention for all program-related information. All waste characterization data and related QA/QC records are described according to NQA-1, Supplement 17S-1 as Lifetime Records and shall be maintained for the active life of the WIPP facility. Records also shall be maintained during the course of any enforcement action for which they are relevant. Record control shall include receipt from external sources, transmittal, transfer to

storage, and the indication of record status. Retention of records shall include the receipt at the storage areas, indexing and filing, storage and maintenance, and retrieval.

In accordance with the "Waste Characterization Program Plan for WIPP Experimental Waste," Final Draft, Rev. 0.0, the project-specific dosimetry records also shall be maintained at the DOE generator/storage facilities and the recorded doses will be used to assess personnel exposure to radioactivity, and evaluate the relative risks and benefits of different TRU waste handling and characterization options in terms of potential personnel exposures. These records, without any identities to individuals, will be made available to the DOE/WPO.

1.6.1 Central Files

Each DOE site shall develop a records management system for hard copies of all information transmitted to DOE/WPO related to waste characterization. Signed originals or copies of data or information obtained from field work and laboratory analyses related to the Program shall be sent to DOE/WPO who shall maintain all Program information in a central file. Each generator/storage facility shall review all records and make copies of any information that they are also required to maintain. The field or laboratory managers shall develop and maintain a recordkeeping system for all program-related QA/QC information and raw data. All summarized data reported to DOE/WPO must be traceable to the original, raw data records. This information shall be maintained by DOE/WPO or its contractor. Records must be legible, clearly identified, retrievable, and secured in a controlled access facility in accordance with ASME NQA-1, Supplement 17S-1.

DOE sites planning to transmit data electronically also must provide a original, hardcopy of the information for data verification. Electronically transmitted data must be compatible with and formatted in accordance with the WIPP computer system requirements that are currently under development.

Each DOE generator/storage facility shall establish a written procedure for reporting obsolete or superseded information. Each generator/storage facility shall designate a person responsible for records administration. This individual shall notify field and laboratory managers and QA personnel of the resulting status changes in program documents, such as reporting formats or procedures. All individuals involved in the Program shall be responsible for reporting obsolete or superseded program-related information to the records administrator. The records administrator shall be responsible for ensuring that these changes are reflected in all records transmitted to the DOE/WPO.

Each site shall have a system for ensuring that outdated or superseded data are not used. The records administrator may request that the copies of such data be destroyed. However, one copy of voided documents shall be maintained for the Program files with the reasons for and date of voiding clearly indicated.

1.6.2 Record Retention

Documentation of all aspects of QA/QC associated with this program shall be retained in DOE/WPO or its contractor's office central program files. Records sent to DOE/WPO shall be reviewed, approved, and transmitted in accordance with the requirements contained in the "WIPP Project Records Management Handbook," (USDOE, 1989). Sites shall keep a record of all documents or records transmitted to DOE/WPO.

As a minimum, these files shall include field QA/QC data; raw analytical data; summary reports of data; project correspondence; laboratory performance evaluation results and final reports; data reduction, numerical analyses, designs, and associated data validation documentation; nonconformance reports and variance logs; audit plans, checklists, audit reports, audit responses, and audit closures; and quality assurance reports to management. Table 1-4 includes a list of the program records that must be maintained in the central files.

Record storage in the central files shall utilize facilities that provide a suitable environment to minimize deterioration or damage (e.g., from temperature, excessive light, or moisture). Access to these facilities shall be controlled and retrieval of information for reference or use outside the storage area shall be documented and controlled. Magnetic media shall be physically protected from inadvertent damage or deterioration from excessive light, stacking, electromagnetic fields, temperature, and humidity in accordance with ASME NQA-1, Supplement 17S-1.

1.6.3 Waste Operations and Laboratory Files

Waste operations facilities (i.e., waste management facilities, RTR, RA, and waste sampling facilities) and analytical laboratories shall maintain records management systems for all applicable QA/QC documents pertinent to that operation. These systems shall provide record control and retention comparable to that outlined in Sections 1.6.1 and 1.6.2 for program central files. Laboratories shall maintain sampling and analytical records for evidence in accordance with the National Enforcement Information Center (NEIC) guidelines (USEPA, 1986).

Table 1-4. Required Types of Program Records Maintained in Central Files [a]

1. All field sampling data forms and records of data reduction
2. Field and laboratory chain-of-custody forms for all samples
3. Sampler certifications
4. Laboratory analytical data reports for samples
5. Sampling system design documents
6. Analytical results of all QA/QC samples
7. All pertinent incoming and outgoing correspondence, memoranda, and telephone records related to QA/QC
8. Reports and data transmittals
9. Reference materials relevant to the program
10. Nonconformance and corrective action documentation
11. Documentation of calculations and computer programs and associated verification
12. Raw data and summarized results of the Performance Demonstration Program
13. Audit plans, reports, responses, and final closure of corrective actions
14. Quality assurance reports to management
15. Training/qualification records [b]
16. Documentation of revisions or changes to the QAPP or QAPjPs
17. Dosimetry records
18. Calibration records
19. Electronic instrument data (e.g., GCMS files)
20. Procurement records [b]
21. Data reduction, validation, and reporting records

[a] DOE facilities are responsible for maintaining these records until transmittal to DOE/WPO.
[b] These records are required in site-specific project files only and will not be transmitted to DOE/WPO.

1.7 Procurement Document Control/Control of Subcontractors

In accordance with ASME NQA-1, Element 4, the design bases and other requirements necessary to assure adequate quality shall be included or referenced in procurement documents for any equipment and services affecting quality.

1.7.1 Procurement Document Control

DOE facilities are required to include or reference in procurement documents the items and support services for the applicable requirements to maintain the quality of this program. To the extent necessary, procurement documents shall require suppliers of equipment or analytical services to have a QA program that meets or exceeds the applicable criteria of this QAPP. If suppliers do not have a QA program that addresses the requirements included herein, they can agree to comply with the applicable site QAPjP requirements.

1.7.2 Control of Subcontractors

Performance requirements must be communicated to all subcontractors involved in this program. Performance requirements and compliance with this QAPP shall be part of subcontractor agreements. DOE operating contractors shall perform quality control inspections of their subcontractor activities to verify compliance with the performance requirements included in this QAPP. Each subcontractor shall, as necessary, complete the necessary training required for implementing the QAPP requirements.

If necessary, prequalification audits may be performed by QA personnel to determine subcontractor acceptability. Subcontractors shall be required to complete and submit copies of project related records to the site Project Manager.

To verify subcontractor conformance to program QA/QC requirements, the DOE operating contractor shall, as necessary, review subcontractor prepared documentation and perform audits of subcontractor activities. Subcontractors shall provide access to their work areas and records for inspection and auditing. Inspections or audits shall be performed, and the results and tracking of corrective actions to final resolution documented as discussed in Section 10.0.

All subcontractors providing analytical services in support of this program must participate in the Performance Demonstration Program as described in Section 1.11. Each subcontractor for analytical services shall demonstrate their ability to meet the QA objectives for this program by successfully completing the requirements of the Performance Demonstration Program prior to sample analysis.

DOE/WPO shall review and approve the results of the Performance Demonstration Program prior to the initiation of work by the subcontractor for analytical services. DOE/WPO, as the administrator of the Performance Demonstration Program, shall notify each analytical laboratory, in writing, concerning the adequacy of its analytical performance and approval to participate in the Program.

1.7.3 Control of Purchased Items and Services

In accordance with ASME NQA-1, Element 7, the procurement of items and services shall be controlled by DOE or its contractors to assure conformance with specified requirements. Such control must include, as appropriate, the evaluation of selected service or equipment, review and evaluation of the QA/QC provided by the supplier, and inspection, audit and examination of items or services upon delivery or completion. DOE/WPO is responsible for obtaining and maintaining electronic media from subcontractors providing analytical services.

The purchase or use of all equipment and replacement parts, or design modifications to existing equipment used for the Program, shall be documented and controlled. Documents traceable to these items must be maintained in the generator/storage facility records.

Services such as analytical services, engineering and consulting, installation, repair, overhaul, or maintenance work shall include oversight by technical verification of the data produced, surveillance, inspection, audit of the activity, or review of certifications for conformance to the procurement document.

1.7.4 Identification and Control of Items

In accordance with ASME NQA-1, Element 8, DOE contractors shall establish methods for the acceptance of materials or equipment in accordance with written detailed procedures. Identification of accepted items shall be maintained on the items or documents traceable to the items, or in a manner which assures that identification is established and maintained. The methods for accepting material or equipment from a supplier may include source verification, receiving inspection, supplier certificate of conformance, post-installation test, or a combination thereof.

1.7.5 Control of Processes

In accordance with ASME NQA-1, Element 9, processes affecting quality of items or services shall be controlled. Special processes that control or verify quality, such as those used in nondestructive examination (RTR and RA), shall be performed by qualified personnel using qualified procedures in accordance with specified requirements. Other processes affecting quality of the Program that shall

be controlled include sampling procedures (Section 4.0), equipment calibration procedures (Section 6.0), and analytical procedures (Section 7.0).

1.8 Indoctrination and Training

In compliance with ASME NQA-1, Element 2, with the exception of Supplement 2S-2, personnel assigned to perform activities involved in waste handling, sampling, and analytical aspects of waste characterization shall have the education, experience, and training applicable to the functions associated with the work. Evidence of personnel proficiency and demonstration of competence in the task(s) assigned must be demonstrated and documented. All personnel designated to work on specific aspects of the program shall maintain that qualification throughout the duration of the work. Job performance shall be evaluated at periodic intervals not to exceed three years.

Training may not be waived for visual examination of waste. Other training may be waived by line management if the individual can demonstrate proficiency in the activity to be performed. The site Project Manager shall be notified in writing and concur with the waiver. A demonstration of proficiency without training must be documented in writing. All training records that specify the scope of the training and the date of completion shall be maintained in the site project file.

Laboratory line management must ensure that each analyst is qualified to perform the analytical method(s) that will be performed by that person. The minimum qualifications for certain specified positions for this program are summarized in Table 1-5. The QAPjPs or their implementing SOPs shall specify the minimum qualifications and training requirements for personnel performing inspection and testing activities. These documents shall also contain the requirement for maintaining records of the qualifications, training, and demonstrations of proficiency by these personnel.

An evaluation of personnel qualifications shall involve a comparison and evaluation of the requirements specified in the job/position description and the skills, training and experience included in the resume of the person. This evaluation also must be performed for personnel who change positions because of a transfer or promotion as well as personnel assigned to short-term or temporary work assignments that may affect the quality of the program. The QAPjPs shall identify the responsible person(s) for ensuring that all personnel maintain proficiency in the work performed and identify any additional training that may be required.

Table 1-5. Minimum Training and Qualifications Requirements

Personnel	Requirements
Gas Chromatograph Analysts and Data Interpreters	B.S. or equivalent experience and 6 months previous applicable experience
Gas Chromatograph-Mass Spectrometer Data Interpreter Gas Mass Spectrometer Data Interpreter	B.S. or equivalent experience and 2 years independent spectral interpretation
Gas Mass Spectrometer Analysts Gas Chromatograph-Gas Mass Spectrometer Analysts	B.S. or equivalent experience and 1 year applicable experience
UV-VIS Spectrophotometer Analysts	3 months applicable experience

Prior to performing activities that affect quality, all personnel are required to receive indoctrination into the scope, purpose, and objectives of the Program and the specific quality objectives of the assigned task.

1.9 Site Quality Assurance Project Plans

Each facility that participates in the Program shall develop and implement a QAPjP that addresses all the requirements specified in this QAPP. Consistent with EPA QAMS-005, the QAPjP must include the following elements:

- Title Page
- Table of Contents
- Project Description
- Project Organization and Responsibility
- Quality Assurance Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability
- Sampling Procedures
- Sample Custody
- Calibration Procedures and Frequency
- Analytical Procedures
- Data Reduction, Validation and Reporting
- Internal Quality Control Checks and Frequency
- Performance and System Audits and Frequency
- Preventative Maintenance
- Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness
- Corrective Action
- Quality Assurance Reports to Management

The QAPjPs shall identify the organization(s) and person(s) responsible for implementation of the plan. In accordance with ASME NQA-1, Element 6, the QAPjPs shall include a document control format consisting of a unique document identification number, the section number, current revision number, date, and page number placed in the upper right-hand corner of each page of the document. The QAPjPs shall reference site-specific SOPs that detail how each of the required elements of the program will be performed. SOPs will ensure that tasks are performed in a consistent manner that results in achieving the quality required for this program.

In addition to the requirements specified in this QAPP, the QAPjPs shall address the applicable activities associated with the test program. These activities are addressed in the SNL Test Plans (Molecke, 1990a; 1990b; Molecke and Lappin, 1990). For example, the QAPjPs for the program must include bin handling and instrumentation requirements and bin leak testing. Additional headspace gas analytes, that are included as part of the test program at the WIPP but are not relevant to waste characterization requirements at the generator/storage facilities, are not included as a part of this QAPP.

Consistent with DOE Order 5700.6B, TRU waste operations shall be conducted in accordance with applicable requirement of the ASME NQA-1. A cross reference may be used to indicate the applicable sections pertaining to each requirement (Table 1-1).

1.10 Analytical Laboratory Quality Assurance Program

Each analytical laboratory participating in the Program must have in place a documented laboratory QA Program that describes general quality assurance procedures specific to that laboratory's normal operations.

1.11 Analytical Laboratory Performance Demonstration Program

In compliance with ASME NQA-1, Element 10, each analytical laboratory handling and analyzing samples for the Program shall participate in the Performance Demonstration Program and demonstrate conformance to the QA objectives for the Program. Each laboratory, through participation in the Performance Demonstration Program, will demonstrate and document the intra-laboratory performance characteristics. The Performance Demonstration Program Plan includes a detailed description of the requirements of the Performance Demonstration Program.

A DOE/WPO designated, independent organization shall provide independent technical oversight and coordination of the inter-laboratory demonstration program to determine the performance characteristics of the analytical methods. Laboratory performance will be evaluated biannually. If specific standards for performance characteristics are not met by a laboratory, problems will be identified, corrective actions taken, and performance reevaluated prior to the analysis of waste samples.

2.0 ORGANIZATION AND RESPONSIBILITY

DOE Order 5700.6B (USDOE, 1990) establishes the policies and responsibilities to assure quality in DOE programs. In compliance with ASME NQA-1, Element 1, this section includes a description of the organizational structure, functional responsibilities, levels of authority, and lines of communication for activities associated with this Program that affect quality. DOE Headquarters provides support at all levels within DOE and contractor organizations to implement effective quality assurance programs. DOE Headquarters has the responsibility to establish an independent institutional coordination and overview function to develop and coordinate quality assurance policies and guidelines.

The organizations and responsibilities for quality assurance activities in support of DOE programs within DOE Headquarters, the DOE WIPP Project Office (DOE/WPO), and the WIPP Waste Acceptance Criteria Certification Committee (WACCC) are described in this section. Figure 2-1 includes the functional organization chart for the QAPP.

2.1 DOE Headquarters Responsibilities and Authority

The responsibilities and authorities of individuals associated with overall DOE quality assurance policies are described in the following subsections. DOE Headquarters has the overall responsibility for all activities within the DOE complex related to the TRU waste characterization. Because the QAPP must be implemented at all DOE facilities planning to send waste to the WIPP, the QAPP shall be approved, issued, and controlled by DOE Headquarters (Table 1-3). By this means, DOE can monitor and assess the needs and potential impacts of the quality assurance/quality control requirements on the DOE TRU waste management system.

2.1.1 Director of Office of Environmental Restoration and Waste Management (EM-1)

The Director of the Office of Environmental Restoration and Waste Management provides policy guidance and centralized management for DOE waste operations. The Director has the overall responsibility and authority for ensuring that DOE quality assurance policy is implemented. The Director provides guidance and direction to the field organizations consistent with requirements related to quality assurance. The Director also ensures that proper planning for resources and budget are provided in DOE programs for effective quality assurance activities that are responsive to the program objectives.

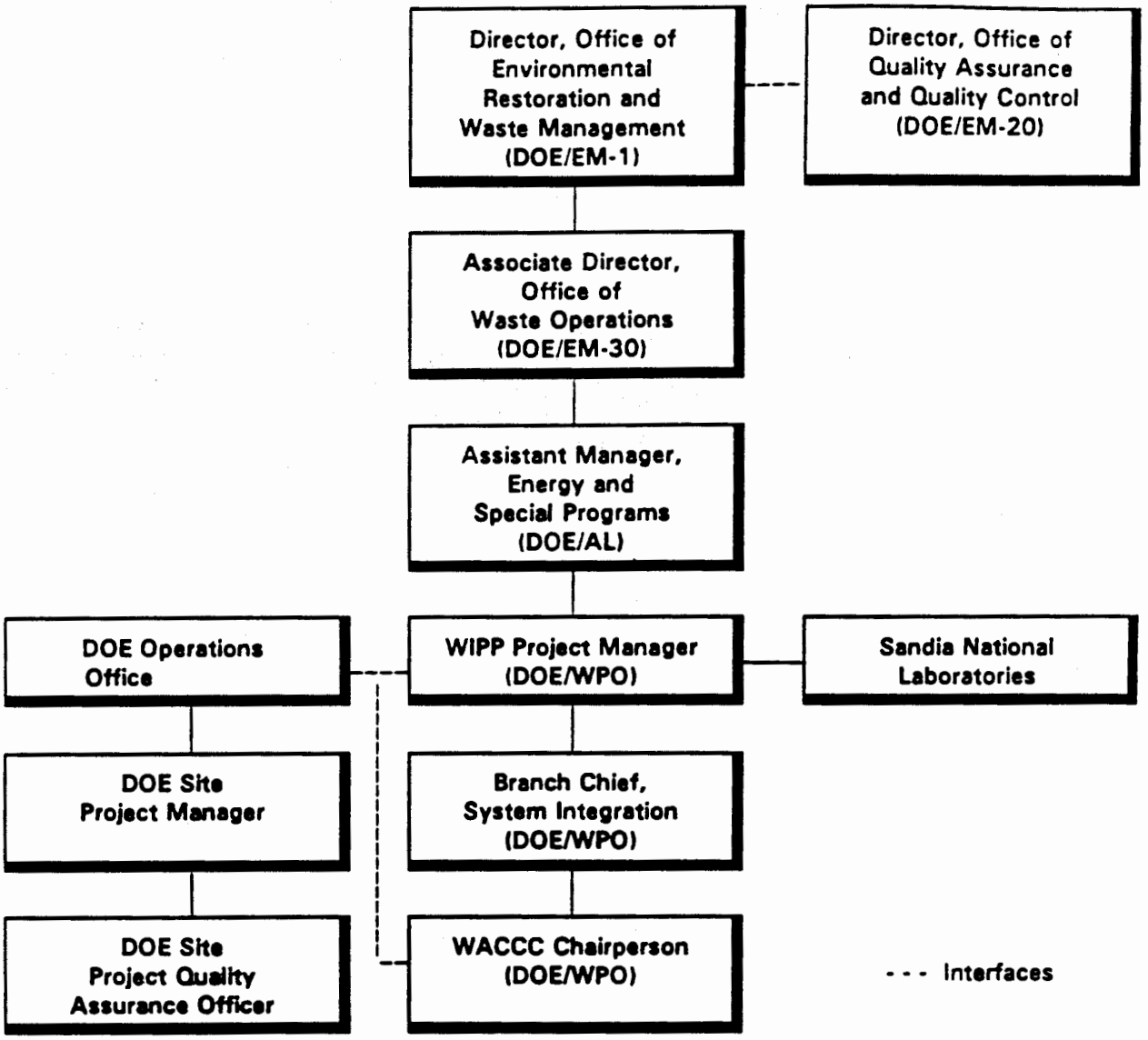

Figure 2-1. QAPP Functional Organization Chart

2.1.2 Director of the Office of Quality Assurance and Quality Control (EM-20)

The Director of the Office of Quality Assurance and Quality Control performs an independent internal oversight function to assure compliance with environmental and safety laws and regulations to ensure the technical validity in programs within EM. The Director provides interpretations of requirements and interfaces and coordinates with other federal agencies for programs and activities managed by EM.

The Director develops and oversees the implementation of the EM QA Program based on DOE Orders, national standards, EPA requirements, and EM program requirements. The Director shall provide an independent oversight function regarding the requirements of this QAPP and shall approve this QAPP for consistency with all applicable regulations and requirements. The Director may participate in selected field organization audits/appraisals of contractor facilities/operations that are scheduled annually by the field organizations.

2.1.3 Associate Director of the Office of Waste Operations (EM-30)

The Associate Director for Waste Operations is responsible for the waste management activities at all DOE facilities, operations, or sites that are used for the storage, treatment, or disposal of radioactive, hazardous, mixed, and solid wastes. The Associate Director manages the on-site and off-site preparation, packaging, and transportation of waste within the DOE complex.

The Associate Director develops, promulgates, and monitors the effectiveness of DOE policies related to the safe handling, storage, treatment, or disposal of wastes throughout the system. As part of this overall responsibility, the Associate Director shall review and approve this QAPP.

2.2 Assistant Manager, Energy and Special Programs (DOE/AL)

DOE Albuquerque Operations is the Field Office responsible for the WIPP facility. DOE/AL has the oversight responsibility and authority for defining and assuring that required WIPP program quality assurance activities are established and implemented.

2.3 DOE WIPP Project Office Authority and Responsibility

The DOE WIPP Project Office, located at the WIPP site, holds the overall responsibility for the successful implementation of the WIPP Project. The DOE WIPP Project Office is supported by Westinghouse Electric Corporation/Waste Isolation Division, the WIPP Management and Operating Contractor, and by SNL, the Scientific Advisor.

2.3.1 WIPP Project Manager (DOE/WPO)

The WIPP Project Manager coordinates and approves all programs and activities related to waste management operations at the WIPP. As a part of this responsibility, the WIPP Project Manager shall review and approve the QAPP for consistency with DOE policies and regulatory requirements pertaining to the WIPP (Table 1-2). The WIPP Project Manager interfaces with DOE/AL to ensure that adequate funding and personnel are available to implement and oversee the activities related to waste characterization and management at the WIPP.

The WIPP Project Manager also is responsible for approving and managing all activities related to conducting of the Test Phase at the WIPP. The WIPP Project Manager reviews and concurs on all proposed experimental and supporting waste characterization activities to ensure that all regulatory and programmatic requirements are met. The WIPP Project Manager shall be responsible for ensuring that all waste characterization data received at the WIPP is reviewed and validated, and that all records pertaining to waste characterization and the experimental program are managed and controlled according to the requirements of the QAPP.

In addition, the WIPP Project Manager shall be responsible for administration of the Analytical Performance Demonstration Program (Section 1.11). In this role, the WIPP Project Manager shall be responsible for coordinating implementation of the Program and reviewing the results of the performance characteristics of the analytical methods from the inter- and intra-laboratory comparisons provided by a designated independent organization. The WIPP Project Manager shall be responsible for scheduling and coordinating the Performance Demonstration Program, and resolving any issues related to conformance with the Quality Assurance Objectives (Section 3.0) of this Program. The WIPP Project Manager also shall be responsible for formally qualifying and approving each analytical laboratory's participation in this Program.

2.3.2 Sandia National Laboratories (SNL)

As a Scientific Advisor to DOE/WPO, SNL is responsible for providing the necessary information and data to address the requirements for the long-term repository performance under 40 CFR Part 191. SNL reports directly to the WIPP Project Manager concerning experimental objectives and data requirements.

SNL is responsible for the development of the WIPP experimental program and has published the Test Plans that describe the experimental objectives and data requirements. They are responsible for identifying the data quality objectives for those experimental parameters that may influence the final

test results. In this capacity, SNL shall review the QAPP and provide comments to the WIPP Project Manager as well as input to the plan based on the test requirements.

2.3.3 Branch Chief, System Integration (DOE/WPO)

The System Integration Office is the lead field office for the TRU waste transportation programs and TRU waste certification. The Branch Chief for System Integration is responsible for the oversight, management, and implementation of short-term and long-term national TRU waste program plans. Other responsibilities related to the QAPP activities include the oversight of TRU waste facility development and TRU waste system program integration. The Branch Chief provides guidance on DOE Headquarters policy and program direction through primary contractors and participating field offices.

2.3.4 WACCC Chairperson (DOE/WPO)

The WACCC Chairperson reports to the Branch Chief for System Integration. The DOE WIPP Project Office has established the WACCC. The authority of the WACCC is established by DOE Order 5820.2A (USDOE, 1988). Two of the primary responsibilities of this committee are the development of the WIPP Waste Acceptance Criteria (WAC) and the verification and certification of TRU waste at the generator/storage facilities for compliance to the WAC through audits and surveillance.

The WACCC Chairperson establishes and maintains a record management system for the WACCC. The Chairperson identifies and manages the budget required to operate the WACCC organization and shall be responsible for coordinating audits and approving the participation of all audit team members and observers (Section 10.0).

In addition, the WACCC Chairperson shall be responsible for conducting the following WACCC activities associated with the Program:

- Review of the QAPP.
- Review and approve all site-specific QAPjPs for compliance with the requirements established in this QAPP.
- Conduct periodic audits at the DOE facilities of applicable waste characterization activities to verify compliance with the site QAPjPs and applicable SOPs.
- Verify, through periodic surveillance conducted at the DOE facilities, that the samples, wastes, and test bins are handled and prepared in accordance with the approved QAPjPs and SOPs.

2.4 DOE Operations Offices Authority and Responsibility

DOE has a number of field offices responsible for operations at various DOE facilities throughout the country. These offices are responsible for ensuring that DOE policies and Orders are implemented.

2.4.1 DOE Operations Office Managers

As a part of this Program, each DOE Operations Office Manager will review and approve the site QAPjPs for the facility under that office's responsibility. The Operations Office Managers are responsible for ensuring that the requirements of the QAPjPs are in compliance with all DOE Orders and that the resources and funding are available to accomplish this Program. All revisions to the QAPjPs that impact compliance with the QA/QC requirements specified in this QAPP must be approved by the DOE Operations Office Managers prior to implementation of the change. The DOE Operations Office Managers are responsible for providing an interface between the operating contractors at the various facilities, the WIPP WACCC Chairperson, and DOE Headquarters to resolve any problems that can affect the quality of the Program.

2.4.2 DOE Site Project Managers

Each DOE facility's operating contractor participating in the Program must designate a site Project Manager who shall be responsible for the oversight of the project at the facility. The site QAPjPs must include a description of the role and define the responsibility and authority of the site Project Manager in relation to the other organizational functions at the site. The site Project Manager shall review and approve the site QAPjP prior to its implementation (Table 1-2). The site QAPjPs shall include a description of the line management responsibilities for implementation of the Program.

2.4.3 DOE Site Project Quality Assurance Officer

Each DOE facility's operating contractor shall designate a site Project QA Officer for this Program and include a detailed description of the responsibility of this person in the site QAPjP. The site Project QA Officer shall review and approve the site QAPjP (Table 1-2). The site Project QA Officer is responsible for verifying the implementation of the quality assurance requirements for the Program and providing the necessary day-to-day guidance to the project staff on quality-related matters. The site Project QA Officer shall identify and report quality problems to the site Project Manager, and initiate, recommend, and track corrective actions to closure.

3.0 QUALITY ASSURANCE OBJECTIVES

The establishment of quality assurance objectives for measurement data in terms of precision, accuracy, representativeness, completeness, and comparability provides definition, control, and verification of waste characterization in accordance with ASME NQA-1, Element 3. The Program is primarily aimed at obtaining data to support the following five data quality objectives:

1. Identify and estimate the weight of the waste material categories that can influence the potential and rate for gas generation. This information is needed to evaluate the long-term repository performance as required under 40 CFR Part 191 and 40 CFR Part 268.

2. Establish the total alpha activity associated with the waste used in the test program. This information is needed to assess the potential for gas generation due to radiolysis.

3. Determine the concentrations of gases in the headspace of waste containers at the DOE generator/storage facilities. These data will be used to indicate the dominant gas generation processes occurring in the containers. These data also will be used in conjunction with the data on the concentrations of volatile organic compounds to verify that flammable mixtures of gases do not exist in containers of waste to be sent to the WIPP.

4. Determine the concentrations of volatile organic compounds in the headspace of waste containers at the DOE generator/storage facilities. These data will be used to verify compliance with the no-migration requirements of 40 CFR Part 268. In addition, these data will be used to verify that flammable concentrations of volatile organic compounds do not exist in containers of waste to be sent to the WIPP.

5. Verify existing waste characterization information that is based on process knowledge using the waste characterization information obtained from this Program.

As recommended by EPA in QAMS-005, this section of the QAPP addresses quality assurance objectives for each waste characterization technique used during this Program. The waste characterization tests will establish baseline values for a variety of parameters that will also be measured during the WIPP bin-scale and alcove tests.

Quantitative data obtained during waste characterization activities must be evaluated in terms of precision, accuracy, representativeness, comparability, and completeness. Five waste characterization techniques must be used:

- Real-time radiography (RTR)
- Radioassay (RA)
- VOC analysis
- Gas analysis
- Visual examination.

The relationships between these waste characterization techniques and the objectives listed above are illustrated in Table 3-1.

Experience obtained during similar programs at the Rocky Flats Plant and at the Idaho National Engineering Laboratory, as well as operator training programs, will provide the foundation for quality assurance during this Program. Program requirements and quality assurance objectives for each of the waste characterization techniques are discussed below.

3.1 Real-Time Radiography

RTR is a non-destructive, qualitative technique that involves X-ray scanning of waste containers to identify and verify their contents. RTR is one of the methods used at DOE generator/storage facilities to demonstrate compliance with WIPP WAC and TRUPACT-II Authorized Methods for Payload Control (TRAMPAC) requirements. All waste must be examined by RTR and excluded from this program if WAC or TRAMPAC requirements are not met.

The objectives of RTR for the Program are the following:

1. Verify waste Content Code (Table 3-2) and Item Description Code (IDC), as applicable.
2. Provide a description of the waste packaging configuration.
3. Provide an inventory of the waste material types.
4. Estimate the quantity of free liquids, if present.
5. Provide the information necessary for making an estimate of each waste item's weight for well segregated and characterized waste. (This will not be possible for heterogeneous waste.)

All activities required to achieve these objectives shall be described in site-specific documentation. During the Test Phase, it is important to derive as much information as possible from RTR in order to reduce the extent of the required visual examination. RTR output data will be used in conjunction with site-specific data on standard weights of waste items and residual material to estimate weights of the waste material categories specified in Table 3-3.

Data to meet these objectives shall be obtained from an audio/videotaped scan provided by trained RTR operators at the sites. Results must also be recorded on an RTR data form. The precision, accuracy, completeness, and comparability objectives for RTR data are presented below.

Table 3-1. Examinations Required to Meet Waste Characterization Objectives

Waste Characterization Technique	Objectives				
	Identify and Estimate Weights of Waste Material Categories	Total Alpha Activity	Gas Concentrations/ Flammability	VOC Concentrations/ Flammability	Verify Process Knowledge
Real-time radiography	YES	NR	NR	NR	YES
Radioassay	NR	YES	NR	NR	YES
Headspace sampling and analysis	NR	NR	YES	YES	YES
Visual examination	YES	NR	NR	NR	YES

NR = characterization not required to meet objective.

Table 3-2. TRUCON Content Codes and Their Description

TRUCON* Content Code	Description
111 [211]	Solidified aqueous waste
112 [212]	Solidified organic liquids
113 [213]	Solidified organic laboratory waste
114 [214]	Cemented inorganic solids
115 [215]	Graphite waste
116 [216]	Paper, plastic, cloth
117 [217]	Metal waste
118 [218]	Glass waste
119 [219]	Filters, mostly organic
121 [221]	Other organic solid waste
122 [222]	Inorganic solid waste
123 [223]	Leaded rubber gloves and aprons
124 [224]	Pyrochemical salt waste
125 [225]	Solid organic and inorganic waste
126 [226]	Cemented organic process solids

*The distinction between a 1XX and a 2XX Content Code is based on the age of the waste.

Table 3-3. Waste Material Categories for Quantification During Visual Examination

Materials
Cellulosics
Plastics
Rubber
Corroding metal/steel
Corroding metal/aluminum
Noncorroding metal
Solid inorganics
Inorganic sludges
Cements
Other organics

3.1.1 Precision and Accuracy

The qualitative determinations made during RTR do not lend themselves to statistical error evaluations. However, previous testing at RFP and INEL indicates that RTR operators can verify the Content Code and IDC of a waste container. This testing also indicates that RTR operators can provide descriptions of the waste packaging configuration and inventories of waste materials in a waste container when RTR data are compared to the actual contents of the waste container during a visual examination. RTR operators also routinely estimate the quantity of free liquids present in a waste container. Precision and accuracy for these qualitative evaluations shall be ensured by standardized RTR procedures and operator qualifications. In addition, the RTR output data will now be used in conjunction with an itemized site standard weight chart provided in QAPjPs or supporting documents to estimate the weight of waste material in the waste container. The precision and accuracy with which these estimates can be made must be evaluated and reported.

3.1.2 Completeness

If the results are unclear or inconclusive during RTR examination, the waste container scan will either be rerun after corrective action has been taken or this container shall not be included in this program. Verified data must be obtained from 100% of the waste used in the waste characterization program.

3.1.3 Comparability

RTR data shall be verified by comparison with data from the visual examination and previous process knowledge. If RTR data are found to be comparable with visual data and process knowledge, then RTR may be used instead of visual examination to characterize certain well-characterized, homogeneous waste for the WIPP bin-scale and alcove tests.

3.2 Radioassay

DOE and its contractors have developed and implemented nondestructive RA technologies that are used throughout the United States (Nuclear Packaging, Inc., 1989). The following three techniques are commonly used in conjunction with isotope ratio calculations and are acceptable for addressing the objectives of the Program:

- Passive/Active Neutron Counting (PAN)
- Segmented Gamma Scan Counting (SGS)
- Passive Neutron Coincidence Counting (PNCC).

For the purposes of the Program, the total alpha activity of the waste must be known, because this value is an indicator and controller of the amount of radiolysis and associated radiolytic gas generation. The total alpha activity, obtained by the nondestructive RA techniques listed above, is considered adequate for use in interpreting the bin-scale and alcove gas generation test results. It is not a requirement of this Program that the waste be representative of all the waste in the DOE system in terms of its radionuclide content.

Each generator/storage facility must use one or more of the RA techniques. The basis for using a specific assay technique should be the physical form of the waste, the radionuclide content, and waste generating process. In all cases, the RA errors must be calculated and reported with the data. The actual precision and accuracy values obtained for waste containers will be a function of waste type, total Plutonium, and its distribution.

The accuracy, precision, and completeness quality assurance objectives to be used for the three RA techniques are summarized in Table 3-4. These objectives are expected to be achievable in the presence of substantial backgrounds generated by alpha-emitting sources and substantial quantities of neutron absorbing and moderating material. This Program's data quality objective for RA is that the total alpha activity, reported as equivalent grams of weapons grade Plutonium, be known with an accuracy of ±0.5 g per waste container for 1 g total per waste container (Lappin et al., 1990).

3.2.1 Precision and Accuracy

The precision and accuracy of the measurement technique must be determined through comparison with calibration standards and replicate processing, respectively. Whenever possible, radioactive calibration standards shall be obtained from sources which maintain measurement systems traceable to NIST. Evidence of such traceability and certificates for individual standards shall be obtained from the standards supplier(s).

3.2.2 Completeness

If the results are unclear or inconclusive during RA examination, the waste container count will either be rerun after corrective action has been taken or this container shall not be included in the Program. Verified assay data shall be obtained on 100% of the waste containers.

3.2.3 Comparability

If multiple systems are available, the DOE sites should perform multiple independent assays of waste containers. These independent assays may include a passive gamma assay such as SGS followed by

Table 3-4. Quality Assurance Objectives for Radioassays

Parameters	SGS		PAN		PNCC	
Precision[a]	WG[b] Pu (g)	Precision	WG Pu (g)	Precision	WG Pu (g)	Precision
	1	±100%	1	±40%	1	±100%
	10	±20%	10	±25%	10	±30%
Accuracy[a]		±15%		±30%		±30%
Completeness	100%		100%		100%	

[a] ±2 standard deviations
[b] WG Pu = Weapons grade plutonium

passive/active neutron assay. When multiple independent RAs are run on a waste container, the sites must document and report the comparability of the techniques to DOE/WPO in the monthly QA reports in accordance with Section 14.0.

3.3 Headspace Sampling

The accuracy and precision of field headspace sampling operations must be assessed by analyzing field QC headspace samples. The field QC headspace samples must include headspace sampling manifold blanks, field reference standards, field blanks, and field duplicates. If the quality assurance objectives described below are not met, then remedial action, such as headspace sampling manifold cleaning, must be taken.

3.3.1 Precision

The precision of the headspace sampling operation must be assessed by collecting field duplicates for VOCs, gases, and NO_x determinations. Remedial actions must be taken if 1) for VOCs, the relative percent difference (RPD) exceeds ±50%; 2) for gases, the RPD exceeds ±20% for concentrations greater than 5X the PRDL, or the absolute difference is greater than ± PRDL for concentrations less than 5X the PRDL; and 3) for NO_x, the RPD exceeds ±40% for concentrations greater than 2.5X the PRDL, or the absolute difference is less than ± PRDL for concentrations less than 2.5X the PRDL.

3.3.2 Accuracy

A field reference standard must be collected using the headspace sampling manifold to assess the relative percent accuracy (RPA) of the headspace sampling operation. Remedial action must be taken if 1) for VOCs, the RPA exceeds ±30%; 2) for gases, the RPA exceeds ±10% for concentrations greater than 10X the PRDL, or the absolute difference exceeds ± PRDL for concentrations less than 10X the PRDL; and 3) for NO_x, the RPA exceeds ±50% for concentrations greater than 2.5X the PRDL, or the absolute difference exceeds ± PRDL for NO_x concentrations less than 2.5X the PRDL.

3.3.3 Representativeness

Specific headspace sampling steps to ensure the representativeness of headspace samples include:

- Headspace sample canister cleaning and leak check
- Headspace sampling manifold cleaning
- Use of headspace sampling canisters with passivated internal surfaces
- Use of a low dead volume headspace sampling manifold

- Careful pressure regulation
- Performance audits
- Collection of headspace sampling manifold blanks, field reference standards, field blanks, and field duplicates.

3.3.4 Completeness

The amount and type of data that may be lost during the headspace sampling operation cannot be predicted in advance. The importance of any lost or contaminated headspace samples must be evaluated and remedial action must be taken as appropriate. Operator training and timely reporting of analytical results should ensure that enough headspace samples are collected to meet the Program objectives.

3.3.5 Comparability

Consistent use and application of uniform procedures and equipment should ensure that headspace sampling operations are comparable at the different headspace sampling sites.

3.3.6 Pressure and Temperature Measurements

Pressure (P) and temperature (T) measurements are required for headspace sampling manifold and headspace sample canister leak checking during the headspace sampling operation. The equipment required for these measurements is described in Section 4.0. Table 3-5 is a summary of the pressure and temperature measurement quality assurance objectives.

3.4 Visual Examination

The visual examination of the contents of waste containers shall provide data on the type and amount of material in each waste container. These data are necessary for the development of a mathematical model that can be used to predict the gas generation behavior of the contact-handled transuranic (CH TRU) waste during the bin-scale and alcove tests and after disposal in the WIPP (reference Sandia Justification Document). The objectives of the visual examination are the same as those outlined for RTR in Section 3.1.

The waste material categories that control the gas generation rates of CH TRU waste are listed in Table 3-3. The estimated or measured values of these ten variables will be used as input to the gas generation behavior model mentioned above.

Table 3-5. Headspace Sampling Quality Assurance Objectives

Measurement	Accuracy[a]	Precision[b]
Manifold pressure	±20 Percent	±0.005 mm Hg[c]
Canister pressure	±20 Percent	±2 in Hg/10 psig
Room temperature	±20 Percent	±1°C

[a] Percent relative difference
[b] Standard deviation of repeat measurements
[c] At 50 μm

3.4.1 Precision and Accuracy

The precision and accuracy of weight estimates obtained from visual examination is unknown at the present time. Estimates of the precision and accuracy of these estimates will be provided by an operator training and evaluation program. The precision and accuracy of weights that are measured during visual examination will be determined by industry standards for balances and must be reported with the weight data.

3.4.2 Completeness

Data on the types and quantities of waste in the waste container must be obtained for all waste that are to be sent to the WIPP. Validated visual examination data must be obtained for 100% of this waste.

3.4.3 Comparability

Visual examination data must be compared to RTR data to verify the RTR findings and process knowledge. During this Program, if RTR and visual data are found to be comparable, the RTR may, in certain cases, be used to identify waste materials and estimate their weights.

3.5 Gas and VOC Analyses

The development of Data Quality Objectives specifically for this Program have resulted in the data quality assurance objectives listed in Tables 3-6 and 3-7. The specified quality assurance objectives represent the required quality of data necessary to draw valid conclusions regarding the objectives of this Program. Key data quality indicators for laboratory measurements are defined below and the methods to quantitatively and qualitatively assess these indicators are discussed in Section 12.0.

3.5.1 Accuracy

Accuracy is the degree of agreement of a measured analyte concentration (or the average of replicate measurements of a single analyte concentration) with the true or known analyte concentration. Accuracy will be expressed as percent recovery. Percent recovery is the ratio of the measured analyte concentration to the known analyte concentration expressed as a percentage. Accuracy will be assessed for the laboratory operations through the analysis of EPA blind samples and laboratory control samples. Results from these measurements will be compared to the criteria listed in Tables 3-6 and 3-7. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Table 3-6. VOC Quality Assurance Objectives

Compound	Precision [a] (%RSD or RPD)	Accuracy [a] (%R)	MDL (ng)	Completeness (%)
Benzene Bromoform Butanol Carbon Tetrachloride Chlorobenzene Chloroform Cyclohexane 1,1-Dichloroethane 1,2-Dichloroethane 1,1-Dichloroethene cis-1,2-Dichloroethene Ethyl Benzene Ethyl Ether Methylene Chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane Trichloroethene 1,1,2-Trichloro-1,2,2-trifluoroethane 1,3,5-Trimethylbenzene 1,2,4-Trimethylbenzene m-Xylene o-Xylene p-Xylene	±25%	70-130%	8	90%
Acetone	±25%	70-130%	50[b]	90%
1-Butanol	±25%	70-130%	50[b]	90%
2-Butanone	±25%	70-130%	50[b]	90%
Methanol	±25%	70-130%	50[b]	90%
4-Methyl-2-pentanone	±25%	70-130%	50[b]	90%

%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
MDL = Method detection limit (maximum permissible value)

[a] Criteria apply to concentrations listed in Table 3-8
[b] Estimate, to be determined

Table 3-7. Gas Analysis Quality Assurance Objectives

Compound	CAS Number	Precision (%RSD or RPD)	Accuracy (%R)	PRDL (vol%)	Completeness (%)
Argon	7440-37-1	±10%	90-110%	0.1	90%
Carbon Monoxide	630-08-0			0.01	
Carbon Dioxide	124-38-9			0.1	
Hydrogen	1333-74-0			0.1	
Nitrogen	7727-37-9			1.0	
Oxygen	7782-44-7			0.1	
Methane	74-82-8			0.1	
Ethane	74-84-0			0.1	
Propane	74-98-6			0.1	
Nitrogen Oxides (combined)		±20%	50-150%	0.01	90%
a. Nitric Oxide	10102-43-9				
b. Nitrogen Dioxide	10102-44-0				

%RSD = Percent relative standard deviation
%RPD = Relative percent difference
%R = Percent recovery
PRDL = Program required detection limit

3.5.2 Precision

Precision is a measure of the mutual agreement among individual measurements of analyte concentrations. Precision will be expressed as the RPD between duplicate measurements or as the percent relative standard deviation (%RSD) between replicate measurements. Precision will be assessed through the analysis of laboratory duplicates and replicate analyses of laboratory control samples and EPA blind samples. Results from measurements on these samples will be compared to the criteria listed in Tables 3-6 and 3-7. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

3.5.3 Completeness

Completeness is a measure of the amount of valid data (meets all QA/QC requirements) obtained from the overall measurement system compared to the amount of data collected and submitted for analysis. Laboratory completeness will be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis.

3.5.4 Method Detection Limit (MDL)

The method detection limit is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL will be expressed in nanograms for VOCs and volume percent for gases. The MDLs will be determined by replicate measurements of samples containing the analytes listed in Tables 3-7 and 3-8. Laboratory determined MDLs must be less than or equal to those listed in Tables 3-6 and 3-7. Procedures for determination of MDLs must be described in site SOPs.

3.5.5 Comparability

Comparability is the degree to which one data set can be compared to another. For this Program, data sets generated through analysis of samples from different sites will be comparable. Comparability shall be achieved by the use of standardized methods, traceable standards, and participation by all sites in the WIPP Performance Demonstration Program.

3.5.6 Representativeness

Representativeness is a quality characteristic which expresses the degree to which a sample or group of samples represent the population being studied. Representativeness for this Program shall be achieved by collecting sufficient numbers of samples with a clean sampling manifold that does not introduce sample bias.

Table 3-8. VOC Headspace Target Compound List (TCL) and Program Required Quantitation Limit (PRQL)

Volatiles	CAS Number	PRQL (ppmv)
1. Acetone	67-64-1	100
2. Benzene	71-43-2	1
3. Bromoform	75-25-2	1
4. 1-Butanol	71-36-3	100
5. 2-Butanone	78-93-3	100
6. Carbon Tetrachloride	56-23-5	1
7. Chlorobenzene	108-90-7	1
8. Chloroform	67-66-3	1
9. Cyclohexane	110-82-7	1
10. 1,1-Dichloroethane	75-34-3	1
11. 1,2-Dichloroethane	107-06-2	1
12. 1,1-Dichloroethene	75-35-4	1
13. cis-1,2-Dichloroethene	156-59-2	1
14. Ethyl Benzene	100-41-4	1
15. Ethyl Ether	60-29-7	1
16. Methanol	67-56-1	100
17. Methylene Chloride	75-09-2	1
18. 4-Methyl-2-pentanone	108-10-1	100
19. 1,1,2,2-Tetrachloroethane	79-34-5	1
20. Tetrachloroethene	127-18-4	1
21. Toluene	108-88-3	1
22. 1,1,1-Trichloroethane	71-55-6	1
23. Trichloroethene	79-01-6	1
24. 1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1
25. 1,3,5-Trimethylbenzene	108-67-8	1
26. 1,2,4-Trimethylbenzene	95-63-6	1
27. m-Xylene	108-38-3	1
28. o-Xylene	95-47-6	1
29. p-Xylene	106-42-3	1

4.0 SAMPLING PROCEDURES

The headspace sampling requirements described in this section must be followed to collect representative headspace samples from the 55-gallon poly bag within a TRU waste drum. The headspace composition may indicate the dominant process occurring in a drum (e.g., production of CO_2 and depletion of O_2 due to radiolysis of paper or cloth) and provide evidence of hazardous constituents in the waste. The material in this section constitutes minimum requirements for sampling. As required by ASME NQA-1, Element 5, detailed sampling procedures will be issued as approved, controlled SOPs implementing the site QAPjPs. These SOPs shall include controls based on the QAPP requirements stated in this section for the handling, storage, cleaning, packaging, shipping, and preservation of items to prevent damage or loss and to minimize deterioration in compliance with ASME NQA-1, Element 13. A guidance headspace sampling procedure can be found in the Guidance Manual.

In the following text, "gas and NO_x" or "gases and NO_x" is used to represent the analytes listed in Table 3-7, and "VOCs" is used to represent the analytes listed in Table 3-8. "Headspace gas" should be interpreted to mean the VOCs and gas within the drum. "Protocol" material constitutes the absolute minimum requirements for compliance with this Program. "Guidance" material consists of recommended practices, and "procedure" consists of detailed step-by-step descriptions of the sequence of actions to be followed to perform a given activity. The procedures outlined in the Guidance Manual are acceptable approaches to meeting the minimum requirements stated in a protocol.

4.1 Headspace Sampling Protocol

This section describes requirements for the collection of samples from the headspace within waste containers. The protocol is designed to ensure that representative headspace samples, including quality control samples, are consistently collected and transferred to the responsible laboratory in a manner that maintains their integrity. Headspace samples for the determination of the analytes listed in Tables 3-7 and 3-8 must include:

- Headspace samples from within the 55-gallon poly drum liner bag(s), Figure 4-1
- Field duplicate samples
- Field blank samples
- Sampling manifold blanks
- Field reference standard samples.

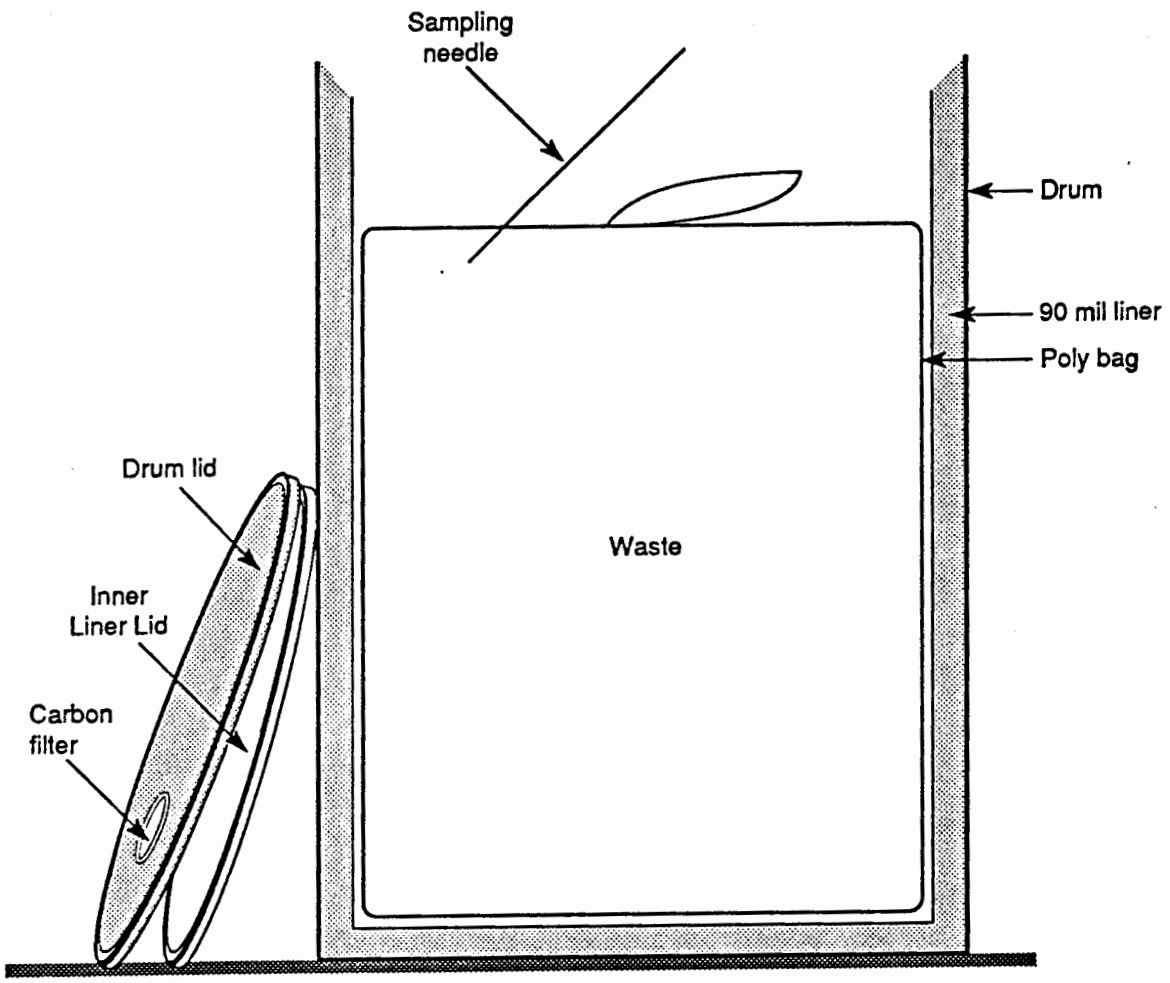

Figure 4-1. Schematic Diagram of Waste Drum with Minimum Layers of Containment and Sampling Location (Not to Scale)

4.1.1 Scope and Application

This protocol for sampling of headspace gases within waste drums is based on guidelines in EPA's Method TO-14 (USEPA, 1988) and SW-846 (USEPA, 1986). As such, it is applicable to sampling the headspace gases in drums containing all types of waste. The method can be used for gases and specific VOCs that are vapors at room temperature and pressure. It is based on the collection of headspace samples in SUMMA® passivated canisters.

4.1.2 Method Summary

The headspace sampling protocol described here employs a multiport manifold capable of collecting multiple simultaneous headspace samples for analysis and quality control purposes. The sampling equipment must be leak checked and cleaned prior to first use and as needed thereafter. Waste containers must be allowed to equilibrate to the temperature of the sampling area for 72 hours prior to headspace sampling. All headspace sampling, which includes the opening of waste containers, must be accomplished within an alpha containment area.

The configuration of the containment area at each headspace sampling location is expected to differ. A description of the containment area must be provided in the site QAPjP. The configuration of remote handling equipment at each headspace sampling location is also expected to differ. If applicable, descriptions of the remote handling equipment must be provided in the site QAPjP.

Headspace samples must be collected from within the innermost 55-gallon poly bag drum liner after removing the drum lid and rigid liner lid. The headspace sampling manifold must be evacuated to 0.05 mm Hg prior to sample collection. Cleaned and evacuated headspace sample canisters must be attached to the evacuated sampling manifold before the sampling manifold inlet valve is opened. The sampling manifold inlet valve must be attached to a changeable needle and filter capable of puncturing the poly bag.

The headspace sampling manifold must be equipped with a purge assembly that allows headspace sampling manifold blanks, field reference standards, and duplicates to be collected through the entire sampling manifold, from the needle tip through all of the same sampling manifold components that the headspace gas passes through. Field blanks must be samples of room air collected in the headspace sampling area, in the immediate vicinity of the waste drums prior to removal of the drum lid. Field blanks shall be collected either by opening an evacuated canister which is detached from the headspace sampling manifold, or by collecting a sample of room air through the headspace sampling manifold after the sampling manifold has been cleaned and verified to contain less than 0.5 ppm VOCs

by using a photoionization detector (PID). The collection of a field blank through the headspace sampling manifold must be immediately preceded by the collection of a headspace sampling manifold blank.

The headspace sampling manifold design and headspace sample volume requirements must ensure that headspace sampling will not withdraw more than 10% of the available headspace gas from within the poly bag. Site SOPs must include a provision for the determination of the dead volume in the headspace sampling manifold and provisions must be made to estimate the available volume of headspace in the poly bag. The total volume of headspace gases collected during each headspace sampling operation is then determined by the combined volume of canisters attached to the headspace sampling manifold, the headspace sampling manifold volume, and ambient temperature and pressure.

Any change to the SOPs which implement the headspace sampling protocol outlined in this section of the QAPP must be described in a Record of Variance, Figure 4-2. The Record of Variance must travel with the headspace sample canister tag. See Section 13.3 for site implementation. The site Project QA Officer shall evaluate the impact of the variance and notify the appropriate personnel.

4.1.3 Headspace Sampling Manifold

As illustrated in Figure 4-3, the system must consist of an evacuated side and a pressurized side. The dotted line indicates how the evacuated side shall be connected to the pressurized side for cleaning the system and collecting headspace sample manifold blanks and field reference standards. The evacuated side must consist of the following major components:

- A needle assembly that allows the poly bag to be pierced to gain access to the waste container's headspace. The needle assembly must contain a filter to prevent particulates from contaminating the headspace sample. To prevent cross contamination, the needle assembly must be cleaned or replaced after headspace sample collection, after field reference standard collection, and after field blank collection if the headspace sampling manifold is used.

- A flexible hose that will allow movement of the needle assembly from the purge assembly (pressurized side) to the waste container's poly bag.

- A sampling manifold pressure sensor that must be pneumatically connected to the headspace sampling manifold, as illustrated in Figure 4-3. The sampling manifold pressure sensor must be able to measure absolute pressure in the range from 0.02 mm Hg to 1000 mm Hg. The usable resolution of the measuring range must be 1×10^{-5} of full scale, or better. The sampling manifold pressure sensor must have an operating range from approximately 15°C to 40°C. The sampling manifold pressure sensor must accurately measure and indicate pressures from 0.02 mm Hg to 1000 mm Hg.

Sample Identification Number:	
Reason for change in QAPjP, or SOP requirements:	
Variation from field or analytical procedure:	
Special equipment or personnel required:	
Initiator's Name:	Date:
Project Manager:	Date:
Project QA Officer/Reviewer:	Date:

Figure 4-2. Record of Variance
EXAMPLE ONLY

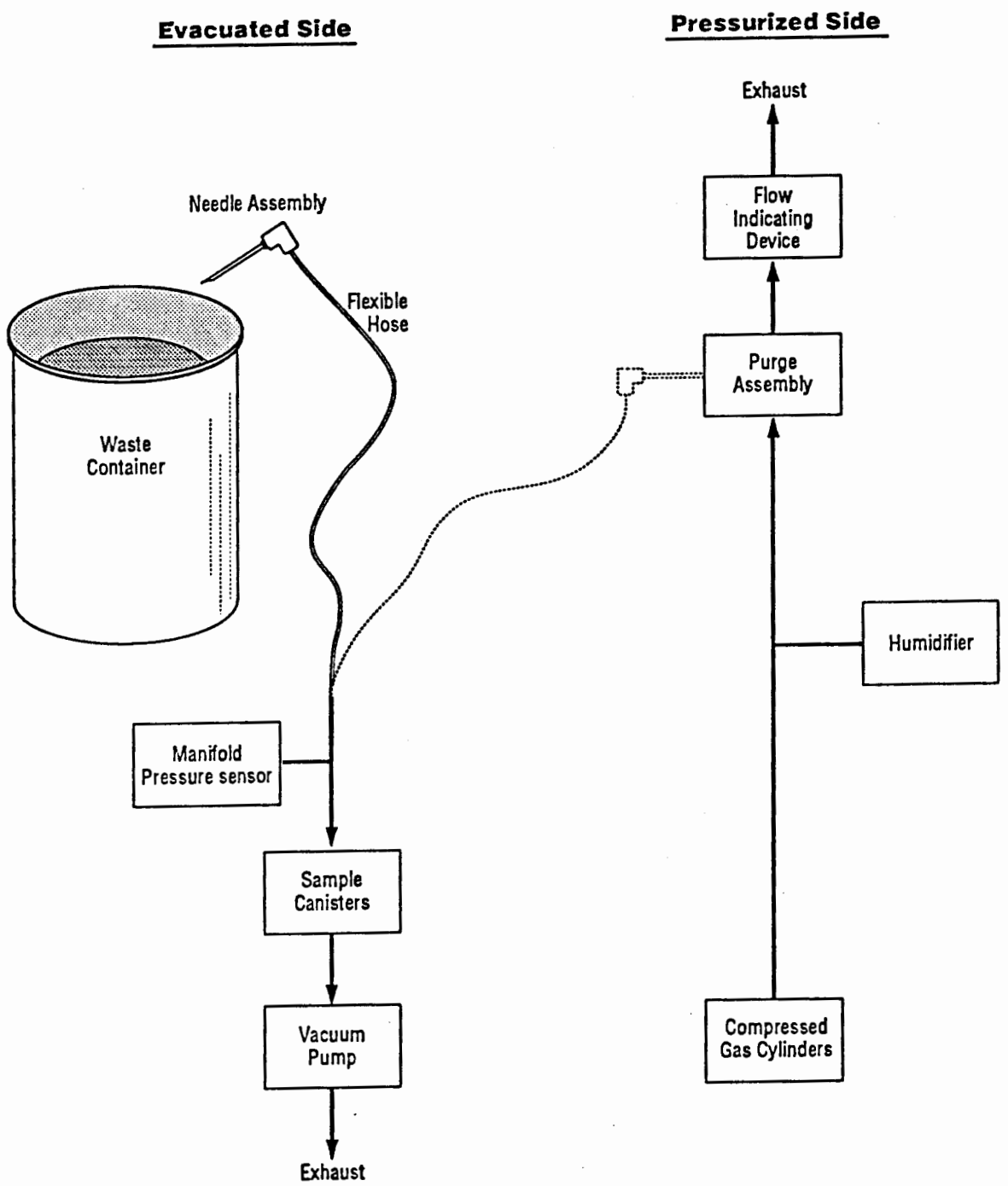

Figure 4-3. Headspace Sampling Manifold Schematic

- Ports for attaching headspace sample canisters. Enough ports are required to allow simultaneous collection of headspace samples and duplicates for NO_x, gas and VOC analyses. Ports not occupied with a headspace sample canister during cleaning or headspace sampling activities require a plug to prevent ambient air from entering the system. In place of using plugs, sites may choose to install valves that can be closed to prevent intrusion of ambient air into the headspace sampling manifold.

- A dry vacuum pump with the ability to reduce the pressure in the headspace sampling manifold to 0.05 mm Hg. A vacuum pump that requires oil may be used, but precautions must be taken to prevent diffusion of oil vapors back through the headspace sampling manifold. Precautions may include the use of a molecular sieve and a cryogenic trap in series between the headspace sampling ports and the pump.

- All components that come into contact with headspace sample gases must be constructed of relatively inert materials such as stainless steel or Teflon®. A passivated interior surface on the stainless steel components is recommended. The distance between the tip of the needle and the valve that isolates the pump from the headspace sampling manifold must be minimized in order to minimize the dead volume in the headspace sampling manifold. The outer diameter of the system's stainless steel and Teflon® tubing should be 1/4-inch.

- [optional] An organic vapor analyzer with a PID capable of detecting all of the VOCs, listed in Table 3-8, with the exception of 1,1,2-Trichloro-1,2,2-trifluoroethane.

The pressurized side must consist of the following major elements:

- A cylinder of compressed zero air and a cylinder of helium, nitrogen, or argon to clean the headspace sampling manifold between headspace samples and provide gas for the collection of sampling manifold blanks. These gases must be of ultra-high purity quality. The gases must be metered into the pressurized side of the headspace sampling manifold by two-stage stainless steel regulators.

- Cylinders of field reference standard gases. These cylinders provide gases for evaluating the accuracy of the headspace sampling process. Each field reference standard gas cylinder must deliver gas through a two-stage stainless steel regulator. The field reference standard gases must be certified by the manufacturer to contain known analytes at known concentrations.

- A humidifier filled with American Society for Testing and Materials (ASTM) Type II water, and connected to the pressurized side of the headspace sampling manifold between the compressed gas cylinders and the purge assembly. Dry gases flowing to the purge assembly will pick up moisture from the humidifier. Moisture is added to the dry gases to condition the headspace sampling manifold blanks and field reference standards and to assist with system cleaning between headspace sample collection.

- A purge assembly to allow the needle assembly (evacuated side) to be connected to the pressurized side of the headspace sampling manifold. The ability to make this connection is required to transfer gases from the compressed gas cylinders to the canisters. This connection is also required for system cleaning.

- A flow-indicating device connected downstream of the purge assembly to monitor the flow rate of gases through the purge assembly. The flow rate through the purge assembly must be monitored to assure that excess flow exists during headspace sampling and cleaning

activities. Maintaining excess flow will prevent ambient air from contaminating the headspace sampling manifold blanks and field reference standards and allow samples of gas from the compressed gas cylinders to be collected near ambient pressure.

In addition to a headspace sampling manifold consisting of an evacuated side and a pressurized side, the area in which the headspace sampling manifold is operated must contain sensors for measuring ambient pressure and ambient temperature, as follows:

- The ambient pressure sensor must have a full range of approximately 508 to 790 mm Hg and must be kept in the sampling area. Its resolution must be 0.1 mm Hg and calibration shall be based on NIST standards. A dual scale thermometer should be included on the pressure sensor case for temperature corrections, if necessary.

- The temperature sensor should have a range from approximately -30°C to 50°C. The temperature sensor must be traceable to NIST standards.

4.1.4 Headspace Sample Canister

Samples must be collected in the headspace sample canisters listed in Table 4-1 and promptly transferred to the responsible laboratory. An overall holding time of 34 days (4 days field holding time + 2 days transfer shipping allowance + 28 days laboratory holding time) is specified to expedite the sampling and analytical process. The customary 28 days holding time for VOCs analysis is also specified for gases and NO_x to ensure uniform sample treatment and to simplify program operations. Headspace samples for VOC analyses must be kept between 0°C and 40°C. There are no storage conditions for NO_x and gas samples and these headspace sample canisters may be held at ambient. Headspace samples (gas, VOCs, and NO_x) must not be retained at the sampling site longer than four working days. Headspace sample canisters must be shipped from the sampling site to the laboratories using the fastest and most appropriate means available. All headspace samples must be handled in accordance with the chain-of-custody guidelines outlined in Section 5.0.

The headspace sample canisters, as illustrated in Figure 4-4, must be leak-free welded stainless steel pressure vessels with a chromium-nickel oxide (Cr-NiO) SUMMA® passivated interior surface, bellows valve, and an optional dial pressure/vacuum gauge. This type of vessel has been previously used for VOC sample collection and has demonstrated VOC sample storage stability of many specific organic compounds (Oliver et al., 1986). Two sizes of headspace sample canisters must be used for the headspace sampling: 100 milliliter headspace sample canisters to collect samples for NO_x and gas analyses, and 250 milliliter headspace sample canisters to collect headspace samples for VOC analyses (Table 4-1).

Table 4-1. Headspace Sample Containers and Holding Times

Parameter	Container	Headspace Sample Minimum Volume	Storage Conditions[a]	Field Holding Time[b]	Laboratory Holding Time[c]
Gases	SUMMA® Canister	100 milliliters	Ambient	4 days	28 days
VOCs	SUMMA® Canister	250 milliliters[d]	10-40°C	4 days	28 days
NO_x[e]	SUMMA® Canister	100 milliliters	Ambient	4 days	28 days

[a] Storage conditions do not refer to transportation conditions.
[b] From time of headspace sample collection to shipment.
[c] This is based on the customary maximum holding time for VOC analysis.
[d] Alternatively, if available headspace is limited, 100 ml samples may be collected for determination of volatiles.
[e] A separate headspace sample will not be required if mass spectrometry is used for inorganic gas analysis.

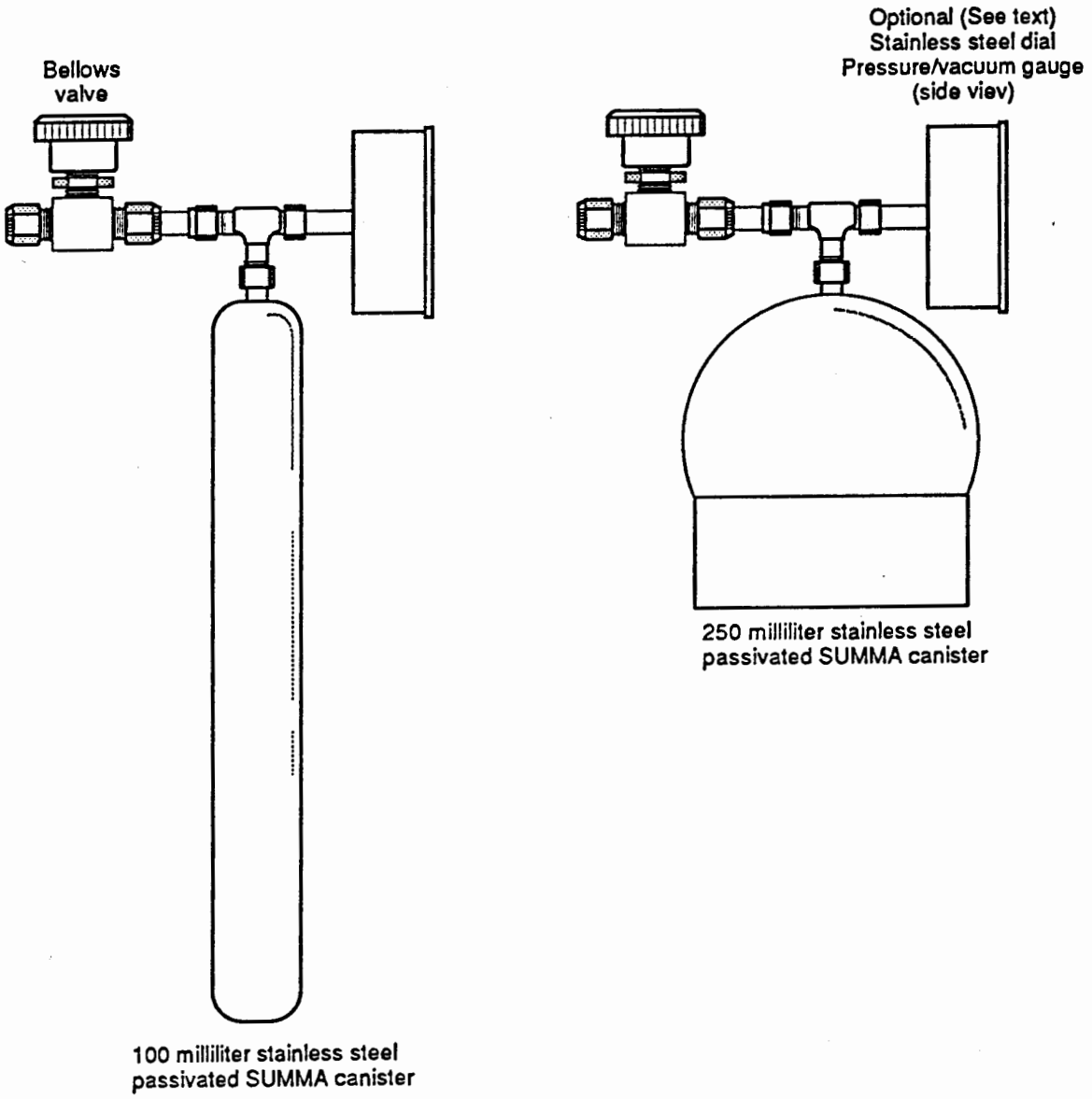

Figure 4-4. SUMMA® Canister Components Configuration (Not to Scale)

When a PID is not used with the headspace sampling manifold, a spiral bourdon-tube type dial pressure/vacuum gauge must be mounted on each headspace sample canister. This type of gauge must be helium leak tested to 1.5×10^{-7} standard cc/sec, have all stainless steel construction, and be capable of tolerating temperatures to 125°C. The gauge range must be able to indicate from 100 psig pressure to a vacuum of 30 inches mercury.

Vacuum/pressure gauges are not required on headspace sample canisters if a PID is attached to the headspace sampling manifold. The PID may be used along with confirmatory headspace sampling manifold blanks to check for headspace sampling manifold cleanliness. If a PID is not used to check for manifold cleanliness, each headspace sample canister must be equipped with a vacuum/pressure gauge. The vacuum pressure gauge will indicate if a headspace sample canister has a substantial leak. The vacuum/pressure gauge will therefore ensure that a clean headspace sampling manifold is not exposed to a headspace sample canister that has leaked.

4.2 Headspace Sampling Manifold Cleaning

The headspace sampling manifold must be properly cleaned prior to headspace sampling. Guidance procedures for cleaning and preparing the headspace sampling manifold are provided in the Guidance Manual. Cleaning protocols are presented below.

4.2.1 Initial Headspace Sampling Manifold Cleaning and Leak-Check

The surfaces of all headspace sampling manifold components that will come into contact with headspace gases must be thoroughly inspected and cleaned prior to headspace sampling manifold assembly. The headspace sampling manifold must be purged with zero air or helium and leak-checked after it has been constructed. This cleaning must be repeated if the headspace sampling manifold is contaminated to the extent that the routine system cleaning is inadequate.

4.2.2 Routine Headspace Sampling Manifold Cleaning and Leak-Check

The headspace sampling manifold must be cleaned and checked for leaks after headspace sample and duplicate collection, after field blank collection if the field blank is collected through the headspace sampling manifold, and after the additional cleaning required after field reference standard sample collection has been completed (see next section). The routine headspace sampling manifold cleaning and leak-check requires that headspace sample canisters be attached to the headspace sample canister ports, or that the ports be capped or closed by valves, and requires that the needle assembly be attached to the purge assembly. Zero air or helium, regulated through the purge assembly, must then be swept through the evacuated side of the headspace sampling manifold. In addition to sweeping the

evacuated side of the headspace sampling manifold, it is recommended that the headspace sampling manifold be heated and periodically evacuated and flushed with zero air or helium. Each cleaning must remove VOCs from the internal surfaces of the headspace sampling manifold to levels that are 50 percent or less of the PRQL of the analytes listed in Table 3-8, as determined by analysis of a sampling manifold blank. When a PID is used with the headspace sampling manifold, the PID must indicate 0.5 ppm or less. Sampling must be suspended and corrective actions must be taken when the analysis of a sampling manifold blank indicates these limits have been exceeded. The site Project Manager must insure that corrective action has been taken prior to resumption of sampling.

4.2.3 System Cleaning After Field Reference Standard Sample Collection

The headspace sampling system must be specially cleaned after a field reference standard has been collected because the field reference standard gases contaminate the pressurized side of the headspace sampling manifold when they are regulated through the purge assembly. This cleaning requires the installation of a gas-tight connector in place of the needle assembly, between the flexible hose and the purge assembly. This configuration allows both the evacuated and pressurized sides of the headspace sampling manifold to be flushed (evacuated and pressurized) with zero air or helium which, combined with heating the pneumatic lines, should sweep and adequately clean the system's internal surfaces. After this protocol has been completed and prior to collecting another sample, the routine system cleaning and leak-check (see previous section) must also be performed.

4.3 Headspace Sample Canister Cleaning

Headspace sample canisters used with these protocols must undergo a rigorous cleaning in accordance with the requirements specified in Section 7.4.1.

5.0 SAMPLE CUSTODY

In accordance with ASME NQA-1, Elements 8 and 13, this section provides identification and control of samples, drums, and bins to assure that only accepted items are used and that identification is maintained on the items or in documentation traceable to the items, and that the items are controlled to prevent damage or loss and to minimize deterioration.

In order to ensure that the data generated under this Program meet accepted standards for legal admissability, chain of custody (COC) must be maintained and documented for drums, samples, and bins throughout the waste characterization process. COC practices shall be documented in SOPs and must be in accordance with EPA guidelines as prescribed in National Enforcement Investigations Center (NEIC) Policies and Procedures, NEIC, Denver, Colorado (revised May 1986).

A waste drum, sample, or bin will be considered under effective custody control if:

- It is in your possession;
- It is in your view, after being in your possession;
- It was in your possession, and you controlled access by either locking it up or placing signed custody seals that prevent undetected access; or
- It is in a designated secure area. Examples include controlled access locations with complete documentation of personnel access and radiological containment areas (hot cells or glove boxes).

TRU waste drums and bins will be considered under effective custody control if they are sealed (i.e., unopened waste drums or sealed bins).

COC shall be maintained in accordance with SOPs that include the documentation requirements described in this section. Whenever a transfer of custody takes place, both parties must sign and date a COC form, with the relinquishing organization retaining a copy of the form. The party that accepts custody must inspect the custody form and all accompanying documentation (e.g., custody seals, sample tags, shipping forms, etc.) to ensure that the information is complete and accurate. The custodian must also inspect all drums, samples, or bins for signs of damage or tampering. Any discrepancies in information or signs of damage or tampering must be documented on the COC form by the receiving custodian. The original COC forms shall be included in the project file provided to WPO.

Each site Project Manager must implement a tracking system to monitor the location and status of drums, samples, and bins on a routine basis. Figure 5-1 is an example of a drum tracking logsheet which may be used for this purpose.

5.1 Drum COC

COC on individual drums shall be initiated at the time the drums are removed from stored inventories. Drum custody must be maintained until the drum's contents are placed in a bin or until the drum is removed from the test Program as a result of failing to meet program requirements. Custody records must document which drums were emplaced in each bin. An example drum COC form is provided in Figure 5-2. Site QAPjPs must include copies of forms used to document drum COC; these forms shall include provisions for each of the following:

- Signature of the individual initiating custody control, along with the date and time;
- Documentation of drum numbers for each drum under custody;
- Signatures of custodians relinquishing and receiving custody, along with the date and time of the transfer;
- Description of final drum disposition, along with signature of individual removing drum from custody; and
- Comment section.

5.2 Sample COC

COC on field samples (including field QC samples) shall be initiated immediately after sample collection or preparation. Sample custody must be maintained until the associated analyses are completed and the data have been validated at the project level (see Section 8.0). Alternatively, sample custody shall be maintained until the sample is expended or until the sample is removed from the Program. An example COC form for samples is provided in Figure 5-3. Site QAPjPs must include a copy of the form used to document COC for samples; this form shall include provisions for the following:

- Signature of individual initiating custody control, along with the date and time;
- Documentation of sample numbers for each sample under custody;
- Signatures of custodians relinquishing and receiving custody, along with date and time of the transfer; and
- Comment section.

WASTE CONTAINER TRACKING LOG SHEET

Waste Container Number: _____
Bin Number: _____

Operation	Date	Time	Initials
Removed from storage			
Transported to RTR			
RTR performed			
Transported to RA			
SGS performed			
PAN performed			
Transported to gas sampling			
Gas sampling performed			
Transported to visual			
Visual examination performed			
Drum contents packed in bin			
Transported to TRUPACT-II Facility			
TRUPACT-II loaded			
Transported to shipper			

Comments (note any discrepancies): _____

_____

Figure 5-1. Waste Container Tracking Log Sheet
EXAMPLE ONLY

WASTE CONTAINER CHAIN OF CUSTODY

Waste Container Number: _____
Bin Number: _____
Person attaching form: _____ Date: _____ Time: _____

Location	Relinquished by	Date	Time	Received by
Storage				
RTR				
RA				
Gas sampling				
Visual exam				

Comments (note any discrepancies):

_____

_____

Disposition: _____

_____

Completed by: _____ Date: _____ Time: _____

Figure 5-2. Waste Container Chain of Custody Form
EXAMPLE ONLY

SAMPLE CHAIN OF CUSTODY FORM

Sampler: _____

Destination Laboratory: _____

Number of Samples: _____

Field Sample Numbers

Location	Relinquished By	Date	Time	Received By

Comments (note any discrepancies): _____

_____

Completed by: _____ Date: _____ Time: _____

Figure 5-3. Sample Canister Shipment Chain of Custody Form
EXAMPLE ONLY

5.3 Bin COC

COC for bins shall be initiated upon introduction of drum wastes to the bin. Bin custody must be maintained until the bin is emplaced in WIPP or until the bin is removed from the test program as a result of failing to meet Program requirements. An example COC form for bins is provided in Figure 5-4. Site QAPjPs must include a copy of the form used to document COC for bins; these forms shall include provisions for the following:

- Signature of individual initiating custody control, along with the date and time;
- Documentation of bin numbers for each bin under custody;
- Signatures of custodians relinquishing and receiving custody, along with date and time of transfer;
- Descriptions of final bin disposition, including signature of individual removing bin from custody, date and time; and
- Comment section.

5.4 Sample Tag and Identification Numbers

Site QAPjPs must describe the conventions for assigning unique identification numbers to all drums, samples, and bins included in the Program. The site numbering conventions must comply with the requirements included in this section.

Each SUMMA® canister must be inscribed with a five-digit canister identification number that is unique to this program. This canister identification number must begin with two alpha characters that can be used to identify the laboratory that purchased the canister. These two alpha characters shall be followed by three numeric characters which may increase sequentially with each canister purchased.

The following laboratory identification codes should be used:

EI	EG&G Idaho VOC analytical laboratory
AW	Argonne National Laboratory - West gas analytical laboratory
AE	Argonne National Laboratory - East analytical laboratory
RI	EG&G Rocky Flats gas analytical laboratory
RO	EG&G Rocky Flats VOC analytical laboratory.

For example, EI001 would be the first canister identification number at the EG&G Idaho VOC analytical laboratory, while AE999 would be the last canister identification at the Argonne National Laboratory - East analytical laboratory.

BIN CHAIN OF CUSTODY FORM

Bin Number: _____

Person attaching form: _____ Date: _____ Time: _____

Location	Relinquished By	Date	Time	Received By

Comments (note any discrepancies): _____

_____

Completed by: _____ Date: _____ Time: _____

Figure 5-4. Bin Chain of Custody Form
EXAMPLE ONLY

5.4.1 Sample Tags

Sample tags shall be utilized to document the physical existence of a sample for the Program file. A removable sample tag must be securely attached to each field and field QC sample prior to shipment to the laboratory. The completed sample tag will be removed by the laboratory and placed in the site Project file. An example tag for samples is provided in Figure 5-5. Site QAPjPs shall include a copy of the sample tags used in the Program. These tags must include the following:

- Drum number
- Sample identification number (13 digits, as described later in this section)
- Sampler signature
- Ambient temperature and pressure during sample collection (°C and mm Hg, respectively)
- Comment section
- Requested analyses
- Sample matrix description
- Date and time of sample collection
- Designation of whether or not the sample is a blank.

The QAPjP must describe a system for documenting sampling and canister conditions as follows:

- After cleaning, canister pressure must be recorded by the certifying laboratory. The reported pressure must be obtained from a manifold gauge (or a canister gauge if the canisters have integral gauges).

- When canister gauges are used, canister pressure must be recorded in the field immediately prior to use.

- Sampling manifold pressure, ambient temperature (°C), and ambient pressure (mm Hg) must be recorded at the end of the sample collection.

- In the analytical laboratory, canister pressure must be recorded within 24 hours of validated time of sample receipt (VTSR) and immediately prior to analysis. Canisters must be thermally equilibrated to ambient temperature prior to measurement of their pressure.

- In the analytical laboratory, the temperature of the gas sample at the time of analysis must be recorded. Ambient temperature measurements may be used if SOPs require equilibration of samples prior to analysis.

- Date, time, and initials of the responsible individual must be documented for each of the above-mentioned measurements.

These documentation requirements may be met through the use of the example tag provided (Figure 5-5), or through other documentation as described in a site QAPjP.

FRONT:

Drum Number: _____

ID # [ ][ ] [ ][ ][ ][ ][ ][ ] [ ][ ][ ][ ][ ]

A A — Sampling Site ID; M M D D Y Y — DATE; A A X X X — Canister ID

Sampling Organization: _____ Sample Description: _____

LOCATION	C or M[a]	Ambient P and T[b]	Date[c]	Time[d]	Initials
Certifying Laboratory					
Field - Prior to Sample Collection					
Field - After Sample Collection					
Analytical Laboratory					

Blank Sample: Y / N

Remarks: _____

Notes:
[a] C = Canister pressure gauge reading (psig); M = Manifold pressure gauge reading (mm Hg)
[b] P = Pressure (mm Hg); T = Temperature (°C)
[c] Date: MMDDYY
[d] Time: 24 hour

BACK:

ANALYSIS *	
Gases: (Table 3-7 Analytes)	
VOCs: (Table 3-6 Analytes)	
pH	
Anions	
Cations	
Volatile organics	
Metals	
Semivolatile organics	
Other:	

* Report detected but unquantifiable analytes

Figure 5-5. Sample Canister Tag
EXAMPLE ONLY

5.4.2 Sample Identification Number

Each sample must be assigned a unique identification number. Thirteen-digit canister sample identification numbers shall be assigned in the following format:

AA	MMDDYY	AAXXX
sampling site ID	date	canister ID

where AA is a two-digit alpha character which designates the sampling site (e.g., ID for Idaho National Engineering Laboratory and RF for Rocky Flats), MMDDYY are numeric characters corresponding to the sampling date (in month-day-year format), and AAXXX is the alphanumeric canister identification number inscribed on the sample canister. For example, ID 031591 AW005 would uniquely specify a headspace sample collected at the Idaho National Engineering Laboratory on March 15, 1991, in SUMMA® canister number AW005.

5.5 QC Sample Record

Each site QAPjP must describe a system for documenting the correlation of field QC samples with their associated field samples, and for controlling this information in such a manner that field duplicates and field reference standards are submitted blind to the analytical laboratory. An example of a QC sample record form is provided in Figure 5-6. Site QAPjPs must include a copy of the form used to record QC samples, and the form must include the following information:

- Sample numbers of each sample collected from each drum, where drums are identified by drum number;
- Date and time of collection for each field and field QC sample;
- Correlation between QC sample numbers and the type of QC sample (e.g., field duplicate) they represent;
- Signature of the individual completing the form; and
- Comment section.

The QC sample record form must be completed at the time samples are generated and shall be submitted to the site Project QA Officer on completion.

FIELD QC SAMPLE RECORD FORM

Date: _____
Bin Number: _____
Sampler: _____

	DRUM NO.		DRUM NO.		DRUM NO.	
SAMPLE TYPE	Sample No.	Time	Sample No.	Time	Sample No.	Time
S_O						
S_I						
S_N						
D_O						
D_I						
D_N						
MB_O						
MB_I						
MB_N						
FB_O						
FB_I						
FB_N						
FS_O						
FS_I						

Comments (note any discrepancies): _____

_____

Legend
Sx - samples
Dx - duplicate samples
MBx - manifold blanks
FBx - field blanks
FSx - field standards

Where x = O for organic; I for inorganic; and N for oxides of nitrogen

Figure 5-6. QC Sample Record Form
EXAMPLE ONLY

6.0 CALIBRATION PROCEDURES AND FREQUENCIES

Test and measurement equipment used in the field and laboratory shall be controlled by a formal calibration program in accordance with ASME NQA-1, Element 12. The program requires equipment of the proper type, range, accuracy, and precision. Calibration of test and measurement equipment may be performed either internally, using in-house reference standards, or externally by agencies or manufacturers. All reference standards must have valid relationships to nationally recognized standards; if no national standards exist, the basis for calibration must be documented. In accordance with ASME NQA-1, Element 14, controls must be established and implemented in site QAPjPs and SOPs, resectively, the ensure that items which have not passed the required inspections and tests are not installed, used, or operated.

Documented and approved procedures must be used for the calibration of measurement and test equipment. Whenever possible, industry standard procedures should be adopted, such as those published by the ASTM or EPA, or procedures provided by manufacturers in equipment manuals.

Calibrated equipment must be uniquely identified by using either the manufacturer's serial number, a calibration system identification number, or other means. This identification, along with a label/record identifying when the next calibration is due (only for equipment not requiring calibration prior to use), must be attached or traceable to the equipment for ready access. Personnel must check the calibration status of equipment prior to use.

Measurement and test equipment must be calibrated at prescribed intervals as part of operational use. The frequency of calibration shall be based on the type of equipment, inherent stability, manufacturers' recommendations, values given in national standards, intended use, and experience.

Reference standards (physical and chemical) must be used for calibration. Physical standards must be stored separately from working measurement and test equipment, where possible. Equipment which cannot be calibrated or becomes inoperable during use must be removed from service and set aside to prevent inadvertent use, or it must be tagged to indicate that it is out of calibration. Such equipment must be repaired and recalibrated to the satisfaction of project requirements before it can be used again.

Instrument manuals must be kept on file for reference purposes. Records must be prepared and maintained for each piece of calibrated measurement and test equipment to indicate that established

calibration procedures have been followed. These records, which must be kept in the project files, shall include:

- Equipment identification/serial number
- Name of device
- Calibration and/or maintenance schedule
- Procedure(s) for calibration and/or maintenance
- Date and results of last calibration with signature of person calibrating
- Date for next scheduled calibration
- Facility or organization performing calibration
- Calibration procedure revision number
- Nonconforming conditions related to the equipment.

Any piece of equipment that fails to meet continuing calibration requirements must be recalibrated, and all affected measurements, assays, or examinations made since the last calibration of that piece of equipment must be rerun.

6.1 Real-Time Radiography

RTR equipment must be calibrated and maintained in accordance with site SOPs. These procedures must address X-ray beam focal area, intensity, symmetry, and energy. Documentation of equipment calibrations must be kept with the equipment as part of the maintenance record. When RTR equipment is in use, operational checks must be conducted at the beginning of each work shift. These checks shall include observation of a test pattern to ensure that the RTR system has adequate video quality.

6.2 Radioassay

The RA equipment must be calibrated at least twice each shift. This must be done at the beginning of the work shift prior to work and a second time at the end of the work shift.

6.2.1 Segmented Gamma Scanning (SGS)

The DOE facility standard guide used for preparation of SGS standards is ANSI N-15.35 (ANSI, 1983). The DOE facility standard for use of these standards is ANSI N-15.20 (ANSI, 1975). ANSI N-15.20 calls for the preparation of calibration material using intimate and stable mixtures of the TRU isotopes with matrix material and for preparation of a suitable number of calibration standards to cover the anticipated isotopic loading. In the case of Pu-239, this range is typically 5 to 200 g for 208 L drums. These guidelines should be followed.

When establishing a calibration curve for the SGS instrument, at least two calibration standards must be used for each Content Code. One standards drum must contain a TRU isotopic mass near the low

end of the expected isotopic loading, and the other standards drum must contain a TRU isotopic mass near the high end of the expected isotopic loading. Both drums must contain waste stream matrix mixtures and densities designed to simulate the waste streams.

6.2.2 Passive-Active Neutron (PAN) Assay System

Calibration of PAN units must include a thorough initial calibration after fabrication and routine calibrations using secondary "pink drum" standards and background drums.

Los Alamos Formal Report LA-10774-MS (LANL, 1986) lists the calibration standards used in all the current PAN units that have NIST-traceable or NIST-referenceable origins. Each PAN unit has been provided with a set of secondary standards (placed in pink drums for conspicuous identification) consisting of standard, NIST-referenceable Cf-252 passive assay and U-235 active assay materials. A baseline reference data set for both passive and active assays has been obtained for each PAN unit with these unique pink drum standards. Each unit must perform standard pink drum assays prior to and after each day of PAN waste drum assays.

6.2.3 Passive Neutron Coincidence Counting (PNCC)

Calibration of PNCC instruments, similar to SGS, must be obtained by establishing a curve of instrument response versus isotopic mass from ANSI N15.20 (ANSI, 1975) and ASTM C 853 (ASTM, 1982). A minimum of four calibration points must be obtained over the expected isotopic mass range using standards that are representative of the materials being measured. Within each Content Code (or waste category) the variation, due to interference effects, must be measured. Calibration standards must be constructed using containers as similar as possible to those used for the actual waste, with contents that are representative of the range of matrix conditions to be encountered. The sites must not extrapolate beyond the calibration range established during instrument calibration. Encapsulated Cf-252 sources, such as those used at Oak Ridge National Laboratory for passive PAN calibration, should also be available for PNCC calibration purposes.

Acceptable ranges for calibration data must be specified in site SOPs. If assay measurement values fall outside the acceptable range, assay measurements must not be performed until the issue has been resolved.

6.3 Headspace Sampling

Calibration requirements described in this section pertain to the calibration of equipment and instrumentation used during headspace sampling activities. All information relating to the calibrations

of the pressure and temperature sensors must be recorded. Prior to its use for this program, all the equipment described below must be calibrated and/or recertified according to site SOPs. All recertifications and recalibrations must be documented.

6.3.1 Certification Check of the Sampling Area Pressure Sensors

The sampling area pressure sensors must be certified prior to initial use, then annually, using NIST standards. The pressure indicated by the pressure sensor must be temperature compensated. If the pressure sensor does not agree with calibration standards to within ±1 mm Hg, it must be repaired or replaced.

6.3.2 Calibration of Ambient Air Temperature Sensor

The ambient air temperature sensor must be certified prior to initial use, then annually, to NIST traceable temperature standards.

6.3.3 Calibration of the Organic Vapor Analyzer

Sampling sites choosing to use an organic vapor analyzer with a photoionization detector (PID) must calibrate the PID once per day, prior to first use, or as necessary according to the manufacturer's specifications. Calibration gases must be of the highest possible quality.

6.4 Visual Examination

The weighing system used during the visual examination must be calibrated using NIST standards. Mass balances, spanning a range of weights from 10 g to 450 kg (1000 lbs), must be recalibrated in accordance with manufacturers' written procedures annually and after major maintenance. Operational checks with a reference standard must be conducted at the beginning of each work shift.

The other equipment used during visual inspection includes video monitoring and audio recording apparatus. Checks of this equipment must include observation of test patterns, prior to each day's use, to ensure that the video picture and soundtrack meet minimum quality acceptance criteria. Maintenance of the visual inspection equipment is discussed in Section 11.0 of this document.

6.5 Determination of Waste Drum Headspace Gases

There are several methods for analysis of the waste drum headspace inorganic gases listed in Table 3-7 that are to be collected in SUMMA® passivated canisters. These methods can be found in the Guidance Manual and include gas mass spectrometry (MS), gas chromatography (GC), and spectrophotometry. MS and GC methods may be applied to some or all gases on the list, while

spectrophotometry is applicable only to the determination of NO_x. Calibration procedures for these methods are derived from instrument manufacturers' recommendations and ASTM Methods 1946, 2650, and 3608. All laboratories shall maintain detailed SOPs covering all aspects of calibration described in this section and instrument run logs so as to enable reconstruction of the calibration sequence and frequency.

6.5.1 Mass Spectrometry

The calibration procedure for this method involves an initial instrument calibration to establish a mass pattern and sensitivity for each gas component. Calibrations shall be verified at the beginning and end of an analytical sequence using a certified standard.

Instrument Performance Criteria. The mass spectrometer must be capable of resolving all of the analytes listed in Table 3-7. This includes the triplet $N_2^+/CO^+/C_2H_4^+$ at mass 28, the doublet $NO^+/C_2H_6^+$ at mass 30, and the triplet $N_2O^+/CO_2^+/C_3H_8^+$ at mass 44. In some mass spectrometers, contribution from the tail of a neighboring peak may have to be compensated for, especially if a minor component is next to a major one (e.g., CO in N_2). In all cases, the mass spectrometer must be able to meet the program required detection limits (PRDLs) in Table 3-7.

6.5.1.1 Standards Preparation

Primary gas standards should be purchased from the best available source (Scott Specialty Gases or equivalent) for the analytes listed in Table 3-7 or for the analytes to be determined by GC. These standards must be certified by the manufacturer.

6.5.1.2 Calibration Procedure

Initial Calibration. Mass and sensitivity calibrations are done initially for all constituents to be measured. Additional calibrations should be performed as specified by the manufacturer or when standard recoveries will not meet the acceptance criteria of ±10% or closures will not meet ±3%.

Continuing Calibration. Continuing calibration checks may be done with the Laboratory Control Standard (LCS). If a gas other than the LCS is used for continuing calibration, then the LCS must be run as a sample during the analytical sequence. The continuing calibration gas must be from a separate source than used for the initial instrument calibration. The calibration check shall be run at the beginning and end of each analytical batch. The sensitivity of each of the analytes in the mixture must be measured. If the RPD between this sensitivity and the most recent valid calibration is less than 10% (50% for NO_x), then the mass spectrometer will be considered calibrated. Sensitivity

calibration must also be verified by showing that the sum of the partial pressure of the components of the components analyzed equals the total pressure of the introduced sample (±3%). If the partial pressure sum differs from the total sample pressure, the differences may be due to any of several problems (e.g., sensitivity errors, unaccounted components of the sample) and must be assessed.

6.5.2 Gas Chromatography

A multipoint calibration curve, consisting of a blank and three standards, will be generated for all analytes using gas standards. This calibration curve will be run daily and when indicated as a QC action.

6.5.2.1 Standards Preparation

Primary gas standards should be purchased from the best available source (Scott Specialty Gases or equivalent) for the analytes listed in Table 3-7 or for the analytes to be determined by GC. These standards must be certified by the manufacturer.

6.5.2.2 Calibration Procedures

Calibration procedures involve generating a multipoint initial calibration. The low standard shall be at the LOQ or the PRDL (Table 3-7), whichever is higher, and the high standard shall be such that it brackets the expected sample concentrations and remains in the linear range of the instrument. Continuing calibration checks must be run at the beginning and end of each analytical batch and at a frequency of 10% during the analytical sequence. The response factor of the continuing calibration check shall be compared to the corresponding average response factor from the most recent valid calibration. If the RPD between the average response factor and the continuing calibration check is less than 10%, the GC system will be considered calibrated. If NO_x is done by GC, the calibration check must be ±50% of the certified value.

6.5.3 Spectrophotometry

A multipoint calibration curve, consisting of a blank and three standards, shall be generated for the NO_x determination using aqueous standards.

6.5.3.1 Standards Preparation

Calibration standards are prepared by dissolving solid $NaNO_2$ in water. The curve is made on the assumption that 0.82 moles of $NaNO_2$ is equivalent to 1 mole of NO_2 gas.

6.5.3.2 Calibration Procedures

Calibration procedures involve generating a multipoint initial calibration. The low standard shall be at the LOQ or PRDL (Table 3-7), whichever is higher, and the high standard shall be such that it brackets the expected sample concentrations and remains in the linear range of the instrument. Continuing calibration checks must be run at the beginning and end of each analytical batch and at a frequency of 10% during the analytical sequence. The response factor of the continuing calibration check shall be compared to the corresponding average response factor from the most recent valid calibration. If the RPD between the average response factor and the continuing calibration check is less than 50%, the system will be considered calibrated.

6.6 Organic Gases

There are several Gas Chromatography/Mass Spectrometry (GCMS) and GC methods (see Guidance Manual) that are appropriate for the analysis of waste drum headspace samples for the VOCs listed in Table 3-8 that were collected in SUMMA® passivated canisters. The differences between these methods are the manner in which the gas sample is introduced to the GCMS or GC and the procedure used to calibrate the system. These methods use commercially available purge and trap systems, gas injection valve with sample loop, or hardware specified in *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air* for gas sample introduction to the GCMS. Calibration procedures used for the analysis of VOC canister samples by these methods are derived from EPA Compendium Method TO-14 and the third edition of EPA SW-846, Method 8240. The GCMS and GC calibration requirements are summarized in Table 6-1. All laboratories shall maintain detailed SOPs covering all aspects of calibration described in this section and instrument run logs so as to enable a reconstruction of calibration sequence and frequency.

6.6.1 Gas Chromatography/Mass Spectrometry Methods

The calibration procedure for GCMS methods involves preparing gas standards or gas/liquid standards spiked with internal standards. Multipoint initial calibration curves must be generated and verified using continuing calibration standards every 12 hours of operation.

6.6.1.1 Standards Preparation

Primary gas standards may be purchased from the best available source (Scott Specialty Gases or equivalent) for the target analytes specified in Table 3-8. Alternatively, primary gas standards may be prepared for the target analytes specified. Analytes that are gases at standard temperature and pressure shall be prepared in a static dilution bottle. For all other analytes, a mixture shall be prepared and loaded directly into a standard cylinder. Secondary analytical standards containing internal

Table 6-1. VOCs Summary Table - GCMS and GC Calibration Requirements

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
GCMS	BFB Tune	Every 12 hours	Table 6-2
GCMS	5-pt initial calibration	Initally and as needed	%RSD all compounds <35%
GCMS	Continuing calibration	Every 12 hours	RPD for all compounds within 35% of initial calibration
GC	3-pt initial calibration	Initially and as needed	%RSD all compounds <30%
GC	Continuing calibration	Every 12 hours	RPD for all compounds within 30% of initial calibration, RRTs within 3 standard deviations of initial calibration

standards shall be prepared from dilution of the primary standards. Reference standards must be EPA traceable, and primary standards should be checked against EPA air audit cylinders to verify the accuracy of their concentrations. Laboratory SOPs must specify detailed requirements for the preparation of gas standards.

Primary liquid standards shall be purchased from the best available source (Supelco or equivalent) for the target analytes specified in Table 3-8. The determination of methanol requires these standards to be prepared in a solvent other than methanol. Alternatively, primary liquid standards may be prepared from pure compounds. Commercially purchased primary standards must be certified by the manufacturer. Secondary analytical standards shall be prepared from the primary standards. Primary standards shall be checked against EPA reference standards to verify the accuracy of their concentrations. Laboratory standards will specify detailed requirements for the preparation of liquid standards.

6.6.1.2 Calibration Procedures

Calibration procedures involve satisfying instrument performance criteria in addition to calibration linearity requirements.

Instrument Performance Criteria. Prior to the analysis of any samples, it must be demonstrated that the GCMS system meets the 4-bromofluorobenzene criteria specified in Table 6-2. Instrument tuning is performed using perfluorotributylamine (PFTBA). Instrument performance criteria shall be checked by putting 50 ng of bromofluorobenzene (BFB) on column. Instrument performance criteria specified in Table 6-2 must be met at the beginning of each 12 hours of operation and prior to the analysis of any standards or samples.

Initial Calibration. After instrument performance criteria have been satisfied, a multipoint internal standard or external standard calibration shall be performed. The multipoint calibration must consist of five secondary analytical standards that define the linear range of the instrument for the analytes listed in Table 3-8. One of the standards must be at an amount (nanograms) equivalent to the PRQLs specified in Table 3-8.

Relative response factors (internal standard) or response factors (external standard) shall be generated for each specified target analyte. For the initial five-point calibration curve to be valid, the percent relative standard deviation (%RSD) of each relative response factor or response factor must be less than 35%. Alternatively, if linearity is not demonstrated (%RSD greater than 35%), then the GCMS

Table 6-2. 4-Bromofluorobenzene Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak, 100% relative abundance
96	5 to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5 to 9% of mass 174
176	Greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

data system may be used to generate a second or third order regression calibration curve. The method used for quantitation must be reported with the results.

Continuing Calibration. At the beginning of every 12 hours of operation, a midpoint concentration calibration standard shall be analyzed. Prior to the analysis of this midpoint standard, the specified instrument performance criteria using 50 ng of BFB must be satisfied.

The relative response factors or response factors for the continuing calibration standard shall be compared to the corresponding average response factor from the most recent valid five-point calibration. If the RPD between the average response factor and the continuing midpoint response factor is less than 35%, then the GCMS system shall be considered calibrated. For those analytes where a second or third order regression curve is used, the point from the continuing calibration standard for the analyte must fall within 35% of the curve value from the initial calibration. If the continuing calibration standard does not satisfy the calibration linearity requirement, a new five-point initial calibration curve must be generated. Sample analysis cannot proceed until the GCMS system has satisfied the calibration linearity requirement.

6.6.2 Gas Chromatography Methods

Gas chromatography may be used for the determination of methanol, butanol, acetone, 2-butanone, and 4-methyl-2-pentanone. The calibration procedure involves injecting gas standards directly on column by using a thermostated gas sample loop and injection valve. A multipoint initial external calibration curve shall be generated and verified using continuing calibration standard every 12 hours of operation.

6.6.2.1 Standards Preparation

Primary gas standards may be purchased from the best available source (Scott Specialty Gases or equivalent) for the alcohols and ketones specified in Table 3-8. Alternatively, primary gas standards may be prepared for the target analytes specified. Analytes that are gases at standard temperature and pressure shall be prepared in a static dilution bottle. For all other analytes, a mixture may be prepared and loaded directly into a standard cylinder. Secondary analytical standards containing internal standards shall be prepared from dilution of the primary standards. Reference standards must be EPA traceable, and primary standards must be checked against EPA air audit cylinders to verify the accuracy of their concentrations. Laboratory SOPs must specify detailed requirements for the preparation of gas standards.

6.6.2.2 Calibration Procedures

Calibration procedures involve generating a multipoint initial calibration curve followed by daily verification of the linearity of the initial calibration curve.

Initial Calibration. A multipoint external standard calibration shall be performed. The multipoint calibration must consist of three secondary analytical standards that define the linear range of the instrument for the alcohols and ketones listed in Table 3-8. One of the standards must be at an amount (nanograms) equivalent to the PRQLs specified in Table 3-8.

Response factors shall be generated for each specified target analyte. For the initial three-point calibration curve to be valid, the %RSD of each response factor must be less than 30%. Alternatively, if linearity is not demonstrated (%RSD greater than 30%), then the GC data system may be used to generate a second or third order regression calibration curve. The method used for quantitation must be reported with the results.

Continuing Calibration. At the beginning of each 12 hours of operation, a midpoint concentration calibration standard shall be analyzed. The response factors for the continuing calibration standard shall be compared to the corresponding average response factor from the most recent valid three-point calibration. If the RPD between the average response factor and the continuing midpoint response factor is less than 30%, then the GC system shall be considered calibrated. For those analytes where a second or third order regression curve is used, the point from the continuing calibration standard for the analyte must fall within 30% of the curve value from the initial calibration. The retention time of each analyte must fall within the retention time window. The retention time window shall be calculated as the mean plus or minus three times the standard deviation of the individual retention times for each initial calibration standard. If the continuing calibration standard does not meet these requirements, a new three-point initial calibration curve must be generated. Sample analysis cannot proceed until the GC system has satisfied the calibration linearity and retention time requirements.

7.0 ANALYTICAL PROCEDURES

Analytical procedures for the Program activities are addressed in this section. In accordance with ASME NQA-1, Element 9, these processes shall be controlled, and special processes such as those used in nondestructive examination shall be performed by qualified personnel using qualified procedures in accordance with specified requirements. Procedures for the operation of equipment and facilities used in RTR, RA, visual examination, gas, and VOC determinations can be found in the Guidance Manual. A protocol covering headspace sampling activities can be found in Section 4.0, and a procedure for headspace sampling can be found in the Guidance Manual.

7.1 Real-Time Radiography

RTR can be used to investigate the following waste characteristics:

- Waste Content Code
- Waste packaging configuration
- Waste inventory
- Quantity of free liquids.

RTR is a nondestructive testing method that allows the operator to examine the contents of a waste container without opening it. The examination method utilizes X-rays to inspect the waste container contents and allows the operator to view events in progress (real time) such as wave motion of free liquids. An RTR system must consist of:

1. An X-ray-producing device
2. An imaging system
3. An enclosure for radiation protection
4. A waste container handling system
5. An operator control station.

The X-ray-producing device must have controls which allow the operator to vary the voltage, thereby controlling image quality. It should be possible to vary the voltage, typically between 150-400 kV, to provide an optimum degree of penetration through the waste. For example, high-density material should be examined with the X-ray device set on the maximum voltage. This ensures maximum penetration through the waste container. Low-density material should be examined at lower voltage settings to improve contrast and image definition. The imaging system typically utilizes a fluorescent screen and a low light television camera.

7.2 Radioassay

Three types of radioassay procedures (SGS, PAN, and PNCC) may be used to meet the requirements of the Program. Procedures applicable to each of these assays are described below. Performance of software for controlling the measurement process and analyzing data shall be demonstrated and documented in accordance with ASME NQA-1, Element 11, Supplement 11S-2. Performance may be demonstrated by the use of test problems and/or in the context of testing the performance of the entire measurement system with quality control samples. Testing must cover the full range of expected applications of the system.

7.2.1 Segmented Gamma Scanning (SGS) Procedure

The assay procedures cited in ASTM C 853 (ASTM, 1982) are recommended for use at all DOE facilities. These procedures require the use of proper calibration standards, proper equipment and equipment setup, avoidance of practices (such as misalignment of the waste package) known to result in inaccurate assays, attention to proper record-keeping and equipment maintenance, and safe operation of the equipment.

7.2.2 Passive/Active Neutron (PAN) Assay Procedure

ASTM and American National Standards Institute (ANSI) standards have not been developed for the active assay portion of the PAN systems. However, the passive coincidence portion of PAN is similar to the PNCC assay technique and, therefore, PNCC, ASTM, ANSI, and Nuclear Regulatory Commission (NRC) standard practices and guidelines should be followed for that portion of the PAN system.

PAN SOPs must instruct operators to acquire a background and a "pink drum" data set before any assays on waste containers are performed. These data sets must be checked for consistency and, if the results fall outside a predetermined acceptance window, remedial action must be taken. Each site must determine and record this acceptance window. The remedial action may include a repetition of the background and/or standards measurements. No CH TRU waste drum assays shall be performed until the problem is satisfactorily resolved.

The assay procedure for PAN units specifies the use of a computerized data acquisition system. An operator must insert a waste drum into the PAN unit and enter all drum identification information via interactive software. Once the software has checked the information for correct format, the assay record and programmable electronics hardware must be properly indexed and switches set. The software then sends a message to the operator that the system is ready to begin an assay.

At this point, the PAN active assay begins. At the conclusion of the active assay, the software automatically records all data and initiates the PAN passive assay. At the conclusion of the PAN passive assay, all data must be recorded, analyzed, and printed out.

7.2.3 Passive Neutron Coincidence Counting (PNCC) Procedure

The assay procedures cited in ASTM C 853 (ASTM, 1983) are recommended for use at all DOE facilities. These procedures require the use of proper calibration standards, proper equipment and equipment setup, avoidance of practices (such as misalignment of the waste package) known to result in inaccurate assays, attention to proper record-keeping and equipment maintenance, and safe operation of the equipment.

7.3 Visual Examination

All CH TRU waste for the bin-scale test and a representative population of waste for the alcove test must be characterized through visual examination of the waste contents. This process must be recorded on audio/videotape and documented as follows:

A description of the waste items in the drum must be recorded on a data form as described in Section 8.0. The description can be brief, but it must clearly identify all discernable waste items and residual materials so that these can be classified according to the waste material categories listed in Table 3-5. Individual bags (or packages) and, if permissible, the contents of each bag within the drum must be weighed, and the weights must be recorded. In cases where bags are not opened and their contents weighed, a brief written description of the contents of the bags must contain an estimate of the amount of each constituent in the bags (e.g., paper, 20 weight percent; rubber gloves, 30 weight percent; etc.). In order to standardize these estimates, an operator training program has been developed (Guidance Manual), and each visual examination operator must participate in the training program. The written records of visual waste examination must be supplemented with the audio/video recording. The overall programmatic approach to visual characterization of the waste is outlined in Figure 7-1.

Figure 7-1 illustrates a two-track approach to the visual examination of waste. Subsequent to RTR and after the drum has been opened, an expert must decide about the extent of waste segregation and weighing that will be required to achieve the program objectives. If the waste is homogeneous, the expert may decide that a limited visual examination involving a confirmation of the RTR data and weighing bags is appropriate. If the waste is heterogeneous, the expert may decide a full visual examination involving determination of a waste item inventory by opening bags, segregating waste, and

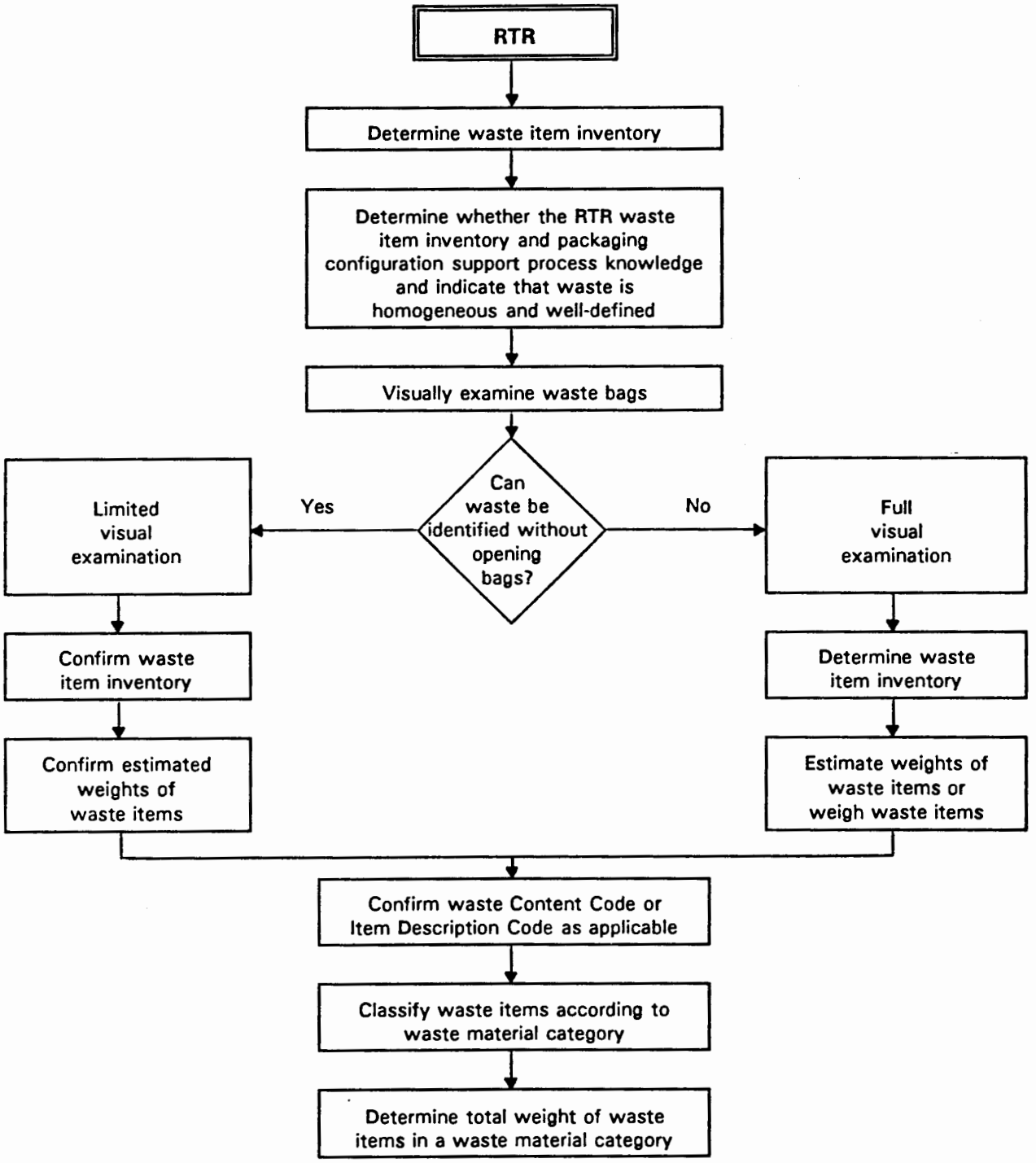

Figure 7-1. Overall Programmatic Approach to Visual Characterization of the Waste

weighing is warranted. Various degrees of segregation and weighing are possible based on the expert's judgment. In all cases, SOPs must be developed to support the visual examination process, and the basis for the expert's decisions must be documented.

7.4 Determination of Waste Drum Headspace Gases

Analytical methods for the analysis of inorganic gases collected using SUMMA® passivated canisters are derived from ASTM Methods 1946, 2650, and 3608. An overview of the methods will be given in this section. Detailed procedures for analysis of analytes listed in Table 3-7 are provided in the Guidance Manual. Laboratory SOPs must specify detailed requirements for implementation of these methods.

7.4.1 SUMMA® Canister Preparation

SUMMA® canisters used in these methods shall undergo a rigorous cleaning and certification procedure prior to their use in the collection of any samples. Guidance for the development of this procedure has been derived from Method TO-14 (USEPA, 1988) and can be found in the Guidance Manual. Specific details shall be given in laboratory SOPs for the cleaning and certification of canisters.

Canisters shall be cleaned and certified on a batch basis. A cleaning system, capable of processing several canisters at a time, composed of an oven and a cryogenically trapped vacuum manifold (Scientific Instrument Specialists or equivalent) shall be used to clean SUMMA® canisters. Prior to cleaning, a 24-hour leak test shall be performed on all canisters. Any canister that fails must be checked for leaks and reprocessed. One canister per batch shall be filled with humid zero air or humid ultra pure N_2 and analyzed for VOCs. The batch of canisters shall be considered clean if there are no VOCs above 50% of the PRQLs listed in Table 3-6. After the canisters have been certified against leaks and background contamination, they shall be evacuated to 0.05 mm Hg or less for storage prior to shipment. Each laboratory shall be required to maintain canister certification documentation.

7.4.2 Inorganic Gases Analytical Methods

Analytical procedures employing gas mass spectroscopy, gas chromatography, or spectrophotometric methods should be employed for the analysis of headspace gas collected in SUMMA® canisters. All of these methods meet the data quality assurance objectives listed in Table 3-7. The method or methods selected for the analysis of headspace inorganic gases shall be the decision of each participating laboratory, provided the requirements of Table 3-7 and the WIPP Performance Demonstration Program (Section 10.2) are satisfied.

7.4.2.1 Mass Spectrometry

The mass spectrometric method (Guidance Manual) is based on ASTM Method 2650 and covers the quantitative analysis of gases containing various combinations of the following components: hydrogen, nitrogen, nitrogen oxides (combined), oxygen, argon, carbon monoxide, carbon dioxide, methane, ethane, and propane.

The samples must be sent to the laboratory in SUMMA® canisters. The SUMMA® canisters should be mounted to the mass spectrometer via appropriate fittings (Swagelock or equivalent). The lines between the canisters and MS inlet system are then evacuated. Gas from the SUMMA® canister must be expanded into a known volume and temperature, at a measured pressure, for introduction into the mass spectrometer. The molecular species which make up a gaseous mixture must be ionized by electron bombardment. The positive ions of the different masses thus formed are accelerated in an electrostatic field and separated in a magnetic field. The abundance of each mass present is recorded. The mixture spectrum obtained must be resolved into individual constituents by means of simultaneous equations or other computer-generated algorithms.

7.4.2.2 Gas Chromatography

The gas chromatography method (Guidance Manual) is based on ASTM Method 1946 and the determination of the following components: hydrogen, oxygen, nitrogen, argon, carbon monoxide, carbon dioxide, methane, ethane, and propane. NO_x composition is not currently determined by this method.

The samples must be sent to the laboratory in SUMMA® canisters. Upon receipt at the laboratory, the pressure in the canister should be measured and recorded. At this point, the laboratory has the option of pressurizing the canister or leaving it at the pressure attained during field sampling. The sample should be injected directly on a column using a thermostated gas injection valve and sample loop (Valco Instruments #EC6UWE or equivalent). If the sample canister is pressurized, the sample can be delivered to the sample loop by using the positive pressure of the sample within the canister.

Components in a sample of headspace gas must be physically separated by the gas chromatograph and compared to corresponding components of a reference standard separated under the same operating conditions. The composition of the headspace gas must be calculated by comparing either the peak height or area response of each component with the corresponding value of that component in a reference standard mixture of known composition.

7.4.2.3 Spectrophotometry

The spectrophotometric method (Guidance Manual) is based on ASTM Method 3608 and covers the determination of the combined nitrogen dioxide (NO_2) and nitric oxide (NO) content in headspace gas in the range from 10 to 1000 ppm.

Samples shall be sent to the laboratory in SUMMA® canisters. The sample introduction apparatus must allow an airtight seal of the canister to the apparatus; evacuation of connecting lines; expansion of sample gas into a known volume, temperature, and pressure; and, finally, the controlled flow of the sample through a frit bubbler in an absorbing solution. An example of this type of apparatus is shown in the Guidance Manual.

7.5 Organic Gases

Analytical methods for the analysis of VOCs collected using SUMMA® passivated canisters should be derived from EPA SW-846 Method 8240/8260 (USEPA, 1986) and/or *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air* (USEPA, 1988). Detailed guidance procedures for the analysis of volatile organic compounds listed in Table 3-8 are included in the Guidance Manual. Site QAPjPs and laboratory SOPs must specify detailed requirements for methods implemented for the analysis of VOCs listed in Table 3-8.

7.5.1 VOC Analytical Methods

Analytical procedures must use GCMS for the analysis of VOCs listed in Table 3-8. Alcohols and ketones may be analyzed by GC-FID if the GCMS methods cannot meet the criteria specified in Tables 3-6 and 3-8. The method or methods selected for the analysis of headspace VOCs shall be the decision of each participating laboratory provided the requirements of Tables 3-6 and 3-8, the WIPP Performance Demonstration (Section 10.2), and all other requirements of this QAPP are satisfied. Samples and calibration standards must be analyzed at the same temperature ($\pm 2°C$).

7.5.1.1 Gas Chromatography/Mass Spectrometry Methods

Analytical procedures employing solid sorbent trapping or cryotrapping followed by thermal desorption and analysis by GCMS or direct on-column gas injection followed by GC-FID analysis shall be employed for the analysis of headspace VOCs collected in SUMMA® canisters. All gas chromatographs shall be equipped with wide-bore capillary columns and have sub-ambient capabilities. The GCMS shall be operated in the full scan mode. This will allow the detection and quantitation of all compounds listed in Table 3-8 and identification of compounds not listed in Table 3-8. These non-target compounds shall be reported as tentatively identified compounds (TICs); the reported concentrations will have a

higher uncertainty associated with them than the reported target analyte concentrations. For samples containing components not listed in Table 3-8, with total ion current peaks greater than 10% of the nearest (retention time) internal standard, a forward search of the latest NIST mass spectral library must be performed. With external standard quantitation, unknown components with total ion current peak areas greater than 10% of the largest target analyte identified or ten times greater than the standard deviation of the background, whichever is less, must be searched.

7.5.1.2 Gas Chromatography Methods

GC-FID methods must be used for the analysis of methanol, butanol, acetone, 2-butanone, and 4-methyl-2-pentanone when analysis of these compounds by GCMS fails to meet the criteria specified in Tables 3-6 and 3-8. Sample introduction shall be by thermostated gas injection valves with sample loops. The method must use two different wide-bore capillary columns. Positive analyte identification shall be achieved by retention time confirmation on both columns. The sample component peak must fall within the retention time window (Section 6.6.2.2) for a given analyte for positive identification. Quantitation of a given analyte shall be performed on one of the two columns. The column used for quantitation must be interferant free in the retention time window corresponding to the analyte.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

In accordance with ASME-NQA-1, Element 17, waste characterization analytical and test results which comply with the data reduction, validation, and reporting requirements of this QAPP section are records that furnish documentary evidence of quality. General guidelines for the data reduction, validation, and reporting of analytical and test results generated in support of this program are outlined in this section. To prevent the introduction of errors or the loss or misinterpretation of the data, adequate precautions must be taken. The requirements discussed here should be considered minimum and will be expanded on throughout the subsections herein regarding specific requirements of the individual analytical or test techniques. Site QAPjPs must delineate how the requirements within this section will be implemented. Implementation detail should be restricted to the SOPs that support the QAPjPs.

For the purpose of data reduction, validation, and reporting the following requirements should be considered minimum:

- All raw data shall be signed and dated in ink.
- All data must be recorded clearly and accurately in field logbooks and laboratory records (bench sheets and/or logbooks).
- All changes to original data must be lined out, initialed and dated by the individual making the change.
- All data must be transferred and reduced from field logbooks and laboratory records completely and accurately.
- All field and laboratory records must be maintained in permanent files according to NEIC guidelines.
- Data must be organized into standard format for reporting purposes, as outlined further in subsequent subsections.
- All electronic or video data must be stored appropriately to ensure that sample and associated QC data are readily retrievable.
- An analytical batch, for the laboratory, will be defined as a suite of samples of similar matrix processed as a unit. This unit must not exceed 20 samples.

<u>Data Reduction</u>

Data reduction must be performed at the site of data generation such that all the requirements outlined in subsequent subsections and/or within the method used are met. These requirements shall include but not be limited to the following:

- The techniques of data reduction used must be consistent with the applicable analytical/test technique and procedures described in subsequent subsections.
- The reporting units must be consistent with the appropriate method used and the requirements described in subsequent subsections.
- All analytical data must be reported to the significant figures specified in subsequent subsections and appropriate to the technique used.
- All data must receive independent technical review to ensure that the reduction is appropriate.

Data Validation

The data review process shall require scrutiny at three levels. The first level of review is conducted on each analytical batch in the laboratory or on each drum for the other characterization tests. Next, the site Project Manager shall review data associated with each bin (bin case). Finally, DOE/WPO will review data at the programmatic level.

The validation process must ensure that all data receives 100% review by qualified individual(s) independent of the original data generator, herein referred to as independent technical review. Validation shall be spread over the three levels of review and must be documented. The site QAPjPs and implementing SOPs must detail the data validation process and associated documentation.

Data Validation - Data Generation Level

The first level of review will ensure that the data have received scrutiny from qualified independent reviewers and signature release from the supervisor(s) and a laboratory QA Officer (as appropriate). The review process will be accomplished by meeting the following minimum requirements:

1. Independent technical review - 100% of the data must receive independent technical review. This reviewer(s) must be a qualified individual other than the data generator. The reviewer(s) must signature release the data and as a consequence ensure that:
    - The data were reduced according to the appropriate method and the reporting units reflect this.
    - Data generation and reduction were conducted in a technically correct manner in accordance with the methods used.
    - Calculations have been verified by a valid calculation program, a spot check of verified calculation programs, and/or 100% check of all hand calculations.
    - All variances from an accepted method have been documented to include the rationale for the variations.
    - All calculation requirements have been met.

- The data have been reviewed for transcription errors.
- The data are complete and include raw data, calculation records, and COCs as appropriate.
- QC sample results are within established control limits, and if not, the data have been appropriately qualified.
- Analytical sample holding times have been met, or exceptions documented.
- RTR and Visual tapes shall be reviewed, on a periodic basis, against the reported data to ensure that the data is correct and complete.

2. One hundred percent of the data must receive technical supervisory signature release on a per analytical batch or drum basis. This release must ensure that:

   - The data are technically reasonable.
   - All data have received independent technical review with the exception of RTR tapes, which shall receive periodic technical review.
   - The data package (analytical batch or per-drum data) is complete and includes raw data, data forms, calculation records, QC summaries, narrative, COCs, and sample tags as appropriate.
   - Analytical sample holding times were met, or exceptions documented.

3. One hundred percent of the data must receive QA signature release. This signature release must ensure the following:

   - Verification that analytical sample holding time requirements were met, or exceptions documented.
   - The data package is complete as appropriate for the point of data generation (i.e., analytical laboratory vs. Visual, RTR, and RA).
   - QC practices were documented and all QC criteria were met.
   - Validation has occurred and is documented.

Data Validation - Project Level

The second level of data review shall occur at the project level on a per-bin basis (bin case).

1. One hundred percent of the bin cases (characterization data generated in support of packing a bin) must have the signature release of the site Project Manager. This signature release must ensure the following:

   - The bin case documentation is complete.
   - Analytical sample holding time criteria have been met, or exceptions documented.

- Verification that data generation level, supervisory, and QA validation and signature releases have occurred. Project level verification should include some repetition of the previous level's validation process.

- Field QC samples have been reviewed for compliance with the criteria set forth within the QAPP. Blank contamination must be noted, duplicate precision reported, and reference samples evaluated for analyte recoveries.

2. One hundred percent of the bin case data must receive signature release from the site Project QA Officer. This signature release must at a mimumum ensure that the data have been evaluated regarding:

   - Field QC checks to include field blank, field duplicate, and field reference standard compliance to criteria within the QAPP.

   - Evaluation of compliance of the data with data quality objectives (DQOs).

   - Evaluation of compliance with applicable regulatory criteria.

   - Validation has occurred and is documented.

Once the data for a bin have received project level data validation and the data are considered acceptable, the site Project Manager must ensure that the laboratory is notified. Samples must be retained by the laboratory until this notification is received. Canisters may then be released from storage for cleaning and subsequent reuse. Sample tags must be removed prior to recycling the canisters and forwarded to the site Project Manager for inclusion in the bin case records.

Procedures For Assessing Compliance With Data Quality Objectives

Random sampling and normal distribution of results shall be assumed when evaluating data to determine compliance with DQOs. Accuracy and precision data generated as a result of the WIPP QAPP Performance Demonstration Program must be used to calculate standardized values in the absence of replicate data on real samples.

Data Evaluation With Replicate Results. The t-test shall be used to calculate the upper confidence limit (UL) at the confidence level specified for a given DQO. If a set of results is being compared to the action limit specified by the DQO to determine if it exceeds the action limit at a given confidence level, the upper confidence limit shall be calculated as follows:

$$UL = \overline{X} + t_{\alpha,\, n-1} \; (\; s/\sqrt{n} \;)$$

X = the mean value of the replicate measurements

$t_{\alpha,n-1}$ = t-value at the desired confidence level

s = calculated standard deviation of the results

n = number of replicates.

The measured result will exceed the action limit with the confidence specified if UL is greater than the action limit value.

Data Evaluation With a Single Result. Precision and accuracy data resulting from the Performance Demonstration Program and method performance testing shall be used to evaluate single result data. If the distribution of results is assumed to be normal, the probability that the actual sample result exceeds the action limit value specified by the DQO can be determined. Tables of the normal distribution (Abramowitz, 1972) give the probability of exceeding a series of standardized variables.

To utilize normal probability function tables, the reported analytical values need to be standardized (EPA, 1987). The analytical result is standardized using the method performance data and the action limit value specified by the DQO as follows:

$$Z = \frac{X_{AL} - C_{MC}}{s} \ , \ C_{MC} = \frac{C_M}{\%R}$$

Z = standardized value

X_{AL} = action limit value

C_{MC} = analytical result corrected for accuracy

C_M = analytical result

%R = percent recovery for a given analyte (from method performance data)

s = standard deviation of measured result for a given analyte (from method performance data).

To determine if the action limit value has been exceeded by the measured value at a specified confidence level, the calculated standardized Z-value is looked up in a normal probability function table. The probability of exceeding the action limit value is expressed as P(x) in the normal probability table. If the P(x) value corresponding to the calculated Z-value is less than the confidence level specified by the DQO for the action limit value, then the analytical result exceeds the action limit value. As an example, if a standardized value of 1.2 is calculated and a 90% confidence level was specified, then the analytical result would exceed the action limit value at the 90% confidence level.

Data Validation - DOE/WPO Level

The third and final level of data review shall occur at DOE/WPO and must, at a minumum, consist of the following:

1. One hundred percent check of the bin case reports for completeness and correctness.
2. One hundred percent verification that project level validation has occurred.
   - Data generation level validation documentation and signature releases.
   - Project level validation documentation to include field QC evaluation, compliance of the data with DQOs, and signature releases.
3. Procedures have been applied for the determination of data outliers detailed below.

Procedures For Determining Data Outliers

The statistical Q-test shall be used for rejection of a suspect result from a set of replicate measurements on a sample from a single source. The Q-test involves calculating a quotient, Q, by dividing the difference between the suspect result and its nearest neighbor by the range.

$$Q = |\text{suspect value} - \text{nearest value}|/(\text{largest value} - \text{smallest value})$$

The suspect result shall be rejected if the calculated Q value is greater than the established rejection Q value for the number of replicates involved. The Q-test shall be performed at the 90% confidence level. Specific rejection criteria are given in Table 8-1.

Data Reporting

Data reporting requirements are detailed throughout subsequent subsections. Each site generating data for this program shall be required to submit a data package encompassing all phases of the testing described in this QAPP.

Data Reporting - Data Generation Level

The laboratories producing analytical data for this program shall submit an analytical batch report/package to the site project manager within 40 days of VTSR of the first sample in an analytical batch. Analytical batch report requirements are identified in subsequent subsections. The minimum information required on reporting forms is identified in the text of the subsections. This information is specified by analytical technique or test method. The format of the information is presented on "Example Only" forms for guidance purposes. Site QAPjPs must include the forms that are intended for reporting use. These site forms must receive DOE/WPO approval prior to their use in support of this Program.

Table 8-1. Q-Test for Rejection of a Suspect Result

No. of Results	Rejection Quotient $Q_{0.90}$
3	0.94
4	0.76
5	0.64
6	0.56
7	0.51
8	0.47
9	0.44
10	0.41

RTR, RA, and Visual data must appear on forms which meet the requirements identified in subsequent subsections. A copy of these forms must be forwarded to the site Project Manager as soon as possible after the data is generated. The original data forms must travel with the waste container through the characterization steps.

Data Reporting - Project Level

The flow of data documentation is represented in Figure 8-1. The site Project Manager will receive waste container information throughout the testing process. It is the responsibility of the site Project Manager to ensure the compilation of all pertinent information regarding a bin with the appropriate analytical and waste container data into one comprehensive report. This report shall entail combining the analytical data report/package with data generated from RTR, RA, headspace sampling and visual analyses, as appropriate, on a per-bin basis. The site Project Manager shall then submit a validated bin case report to DOE/WPO within two weeks of receipt of all the necessary bin case data. The final bin case report submitted to DOE/WPO must, at a minimum, consist of the following:

- A cover page with the site name, project identification, and approval/release signatures of the site Project Manager and site Project QA Officer.
- A table listing the field sample numbers, associated laboratory sample numbers, waste container numbers, and the identity of the bin.
- A narrative describing any problems encountered throughout the entire scope of tests performed for this program (i.e., Visual, RTR, RA, and laboratory analysis), to include any deviations from the methods used.
- A result summary for each characterization technique.
- All raw data.

The site Project Manager must maintain a record of transmittal regarding the submission of the bin case report to the DOE/WPO. The process by which data are compiled, reviewed, and then shipped to the DOE/WPO must be formalized in a site-specific procedure.

8.1 Real-Time Radiography

The results of the RTR examination for each waste container must be documented and available to the data users. Videotapes must be stored by the data generating facility, as specified on page 8-1.

8.1.1 RTR Data Reduction

The identity of waste items and residual material documented on an RTR data form must be used in conjunction with site-specific data on standard weights of waste items and residual material to classify

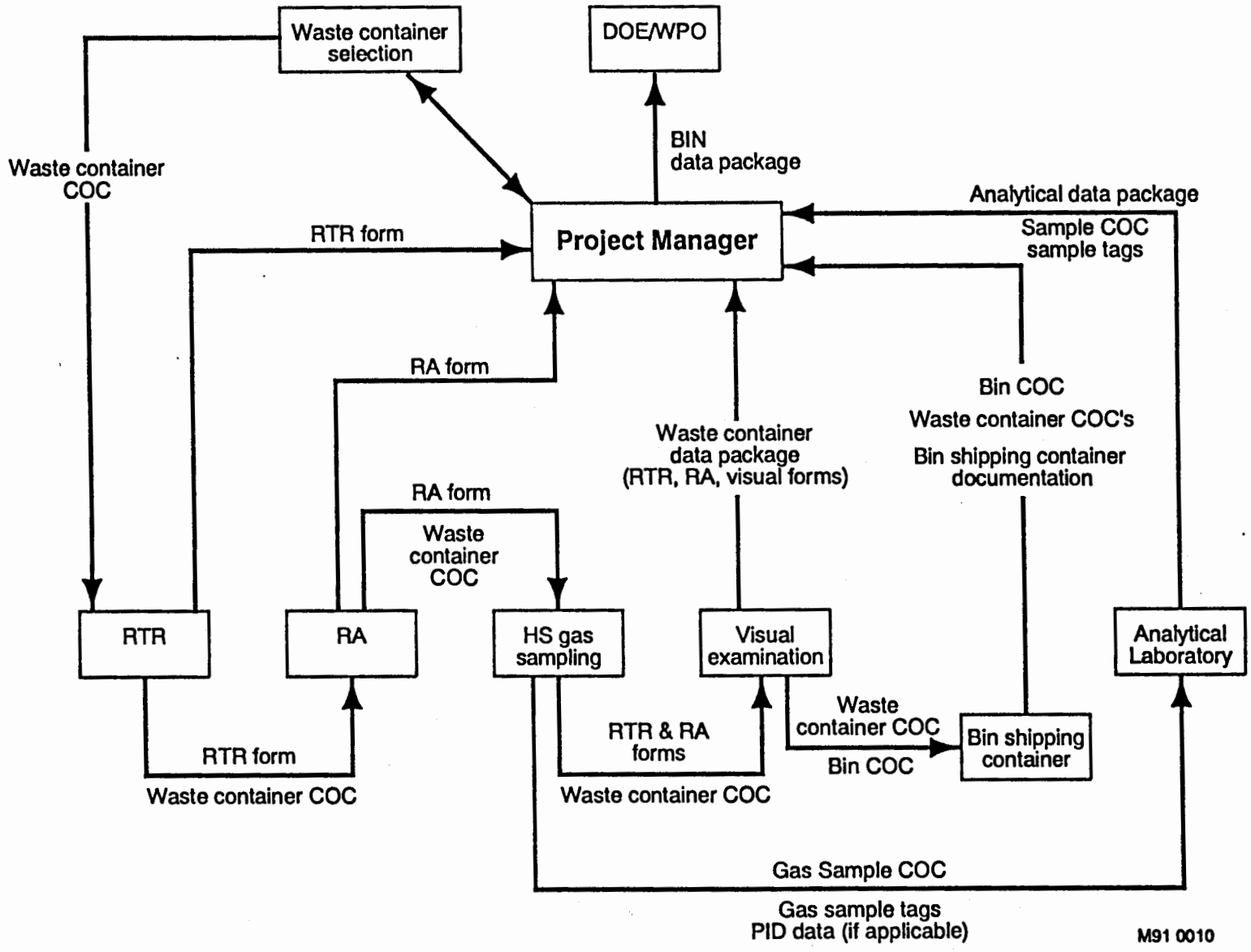

Figure 8-1. Overall Data Reporting Flowchart

waste items into the waste material categories of interest (Table 3-3) and to estimate the total weight of waste material in these categories. Site QAPjPs must specify procedures for this classification and estimation activity and for evaluating the error of the weight estimates. Error calculation procedures must be developed with the concurrence of Sandia National Laboratories.

8.1.2 RTR Data Reporting

The data reporting requirements for RTR are:

- Examining site
- Waste container identification number
- Content Code or IDC, as applicable
- Any changes made to Content Code or IDC
- Date of RTR examination
- Operator signature/date
- Reviewer signature/date
- Presence/absence of waste container liner (yes/no)
- Inventory of waste container contents, e.g., number/count of a particular waste item
- Description of contents packaging materials
- Estimated volume of free liquid, if present (ml)
- Audio/videotape identification number
- QC replicate (yes/no); if yes, brief description of comparison results.

Figure 8-2 or a similar form containing all the information specified above must be completely filled out and signed. Figure 8-1 indicates how the RTR data form should travel through the waste characterization process.

8.2 Radioassay

The results of RA for each waste container must be documented and available to the data user.

8.2.1 Data Reduction

The reduction of RA data may be accomplished using computer software that is specifically designed for the particular assay being performed. This software may vary from site to site. This software and/or other data reduction procedures must be specified in site QAPjPs and supporting SOPs.

8.2.2 Radioassay Data Reporting

The data reporting requirements for RA are:

- Examining site
- Type(s) of RA: PAN/SGS/PNCC (circle)
- Waste container identification number
- Content Code or IDC, as applicable
- Date of RA examination

RTR DATA FORM

Page 1 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

1. Is this a single component waste form? _____ Yes _____ No

2. Brief description of waste container contents:

_____

_____

_____

3. Do you recommend the TRUCON Content Code or the IDC be changed? _____ Yes _____ No

   If yes, what is the recommended TRUCON Content Code? _____ IDC? _____

4. Have liquids been detected? _____ Yes _____ No

   If yes, describe the container type, location, etc. where liquids were detected and estimate the volume of liquid.

_____

_____

_____

   Estimated volume of liquids _____(ml)

5. Waste container fill percentage: _____ %.

6. Waste container liner present? _____ Yes _____ No

Figure 8-2. RTR Data Form
EXAMPLE ONLY

RTR DATA FORM (continued)

Page 2 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

7. Inventory of Waste Container Contents

WASTE ITEM(S) AND RESIDUAL MATERIALS			PACKAGING MATERIAL		
Description	Quantity	Est. Weight*	Description	Quantity	Est. Weight*

Figure 8-2. RTR Data Form
(Continued)
EXAMPLE ONLY

RTR DATA FORM (continued)

Page 3 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

8. QC replicate? _____ Yes _____ No

If yes, brief description of comparison results:

_____

_____

_____

9. **Comments**

_____

_____

_____

_____

10. Operator Name(s)/Date | Operator Employee Number

_____ | _____

_____ | _____

_____ | _____

11. Data Recorder Name(s)/Date (if applicable) | Recorder Employee Number

_____ | _____

_____ | _____

12. Reviewer/Supervisor Name/Date | Reviewer/Supervisor Employee Number

_____ | _____

* Estimated weights will be determined using the RTR derived waste inventory along with manufacturers' information and process knowledge.

Figure 8-2. RTR Data Form
(Continued)
EXAMPLE ONLY

RTR SUMMARY DATA FORM Page 4 of 4

			Initials	Date
Examining Site:	_____			
Waste Container ID Number:	_____	Operator	_____	_____
TRUCON Content Code:	_____	Recorder	_____	_____
IDC:	_____	Reviewer	_____	_____

WASTE TYPE	Estimated Weight (kg)	Error (kg)
CELLULOSICS Paper, plywood, wood, cloth, HEPA filters	_____	_____
Other organics Resins, oils, solvents, organic sludges	_____	_____
Plastics Bags, liners, waste, benelex, plexiglas	_____	_____
Rubber Gloves, aprons	_____	_____
Corroding metal - steel Drums, wastes, including stainless	_____	_____
Corroding metal - aluminum Wastes	_____	_____
Noncorroding metal Lead, tantalum, copper, etc.	_____	_____
Solid inorganic wastes Glass, ceramics, graphite, etc.	_____	_____
Inorganic sludges	_____	_____
Cements	_____	_____
Totals	_____	_____

Comments (note any descrepancies):

_____

_____

_____

Figure 8-2. RTR Data Form

(Continued)

EXAMPLE ONLY

- Operator signature/date
- Reviewer signature/date
- Total Pu-239 fissile gram equivalents (gm) and associated error
- Total alpha activity (curies) and associated error (curies)
- Reference standard calibration performed (yes/no)
- QC replicate (yes/no); if yes, brief description of comparison results.

Figure 8-3 is an example RA data form. Figure 8-1 indicates how the RA data form should travel through the waste characterization process.

8.3 Visual Examination

The results of the visual examination must be documented and available for each waste container.

8.3.1 Visual Examination Data Reduction

The visual examination data reduction requirements are generally the same as those for RTR, Section 8.1.1.

8.3.2 Visual Examination Data Reporting

Reporting for visual waste characterization must include:

- Examining site
- Waste container identification number
- Content Code or IDC, as applicable
- Any changes made to Content Code or IDC
- Date of visual examination
- Operator signature/date; second operator signature/date
- Reviewer signature/date
- Waste container's gross weight (kg)
- Empty waste container weight
- Each waste bag's measured or estimated weight (kg)
- Identity and estimated or measured weight of identical waste items present inside each bag (kg)
- Identity and total estimated or measured weight of identical residual material present inside each bag (kg)

RA DATA FORM

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

1. Assay method used: _____

2. Calibration (within established ranges): _____ Yes _____ No

3. Error analysis performed: _____ Yes _____ No

4. Total Pu-239 fissile gram equivalents (FGE): _____ g ± Error _____ g

5. Total alpha activity: _____ Ci ± Error _____ Ci

6. If this is not a new examination, record: _____ Yes _____ No
   Date of examination: _____/_____/_____
   Operator name: _____

7. QC replicate? _____ Yes _____ No
   If yes, brief description of comparison results: _____

8. Comments: _____

9. Operator Name/Date _____ Operator Employee Number _____

10. Data Recorder Name/Date (if different from operator) _____ Recorder Employee Number _____

11. Reviewer/Supervisor Name/Date _____ Reviewer/Supervisor Employee Number _____

Figure 8-3. RA Data Form
EXAMPLE ONLY

- Total number of inner waste bags in each waste container
- Identity and total estimated or measured weight of identical waste items and residual materials *outside* of the individual bags (kg)
- Estimated total water content of cements, if present (wt %), from process knowledge
- Suspected identity and estimated or measured weights of unknown waste items and residual materials (kg)
- Estimated volume of free liquid, if present (ml)
- Estimated or measured total weights per waste container for each of the waste material categories identified in Table 3-5 (kg)
- Mass balance reference standard calibration performed (yes/no)
- QC duplicate (yes/no); if yes, brief description of comparison results
- Expert's rationale for extent (full/limited) of visual examination
- Audio/videotape identification number.

Figure 8-4, or similar forms containing all the information specified above, must be completely filled out and signed. Figure 8-1 indicates how the visual examination data sheet should travel through the waste characterization process.

8.4 Gas Analysis

Required procedures for data reduction and reporting of gas analysis results are given in this section. Specific equations for data reduction must be detailed in site QAPjPs and/or implementing SOPs.

8.4.1 Data Reduction

All inorganic analyte concentrations determined by GC shall be quantified using average relative response factors obtained from certified calibration standards. Target compound concentrations must not be blank corrected. Results from laboratory blanks run in association with samples shall be reported separately from any target compounds detected. All results must be reported in volume percent and shall be limited to three significant figures, but no more than two decimal places. Guidance procedures for converting raw data to reportable results in volume percent can be found in the Guidance Manual. Site SOPs must detail procedures for reducing raw data to reportable results.

VISUAL DATA FORM

Page 1 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

1. Mass balance reference standard calibration performed? _____ Yes _____ No

2. Expert's rationale for extent (full/limited) of visual examination:

   _____

   _____

   _____

3. Do you recommend the TRUCON Content Code or IDC be changed? _____ Yes _____ No

   If yes, what is the recommended TRUCON Content Code? _____ IDC? _____

4. Are liquids present? _____ Yes _____ No

   If yes, describe the container type, location, etc. where liquids were found and estimate the volume of liquid.

   _____

   _____

   _____

   Estimated volume of liquids _____(ml)

5. Waste container fill percentage: _____ %

6. Gross weight of waste container: _____ kg

7. Empty waste container weight: _____ kg

8. Rigid waste container liner? _____ Yes _____ No
   If yes, weight of rigid waste container liner: _____ kg

9. Waste container liner bag(s)? _____ Yes _____ No

Liner Bag Type	Number of Liner Bags	Liner Bag Weight (kg)

Figure 8-4. Visual Data Form
EXAMPLE ONLY

VISUAL DATA FORM (continued)

Page 2 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

10. Inner waste container(s)		Waste Item(s) and Residual Material		
ID Number	Weight (kg)	Description of Contents and Packaging Material	Est. Weights	Actual Weights

11. Total number of inner waste containers: _____

12. Description of contents and packaging material of loose waste items or residual materials not contained in inner waste container(s)	Waste Item(s) and Residual Material	
	Est. Weights	Actual Weights

Figure 8-4. Visual Data Form
(Continued)
EXAMPLE ONLY

VISUAL DATA FORM (continued)

Page 3 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

13. **Comments** (Note any discrepancies):

_____

_____

_____

_____

14. QC duplicate? _____ Yes _____ No

If yes, brief description of comparison results:

_____

_____

_____

_____

15. Audio/videotape identification number: _____

16. Operator Name(s)/Date — Operator Employee Number

_____ _____

_____ _____

_____ _____

17. Data Recorder Name(s)/Date (if applicable) — Recorder Employee Number

_____ _____

_____ _____

18. Reviewer/Supervisor Name/Date — Reviewer/Supervisor Employee Number

_____ _____

Figure 8-4. Visual Data Form
(Continued)
EXAMPLE ONLY

VISUAL EXAMINATION SUMMARY DATA FORM Page 4 of 4

			Initials	Date
Examining Site:	_____			
Waste Container ID Number:	_____	Operator	_____	_____
TRUCON Content Code:	_____	Recorder	_____	_____
IDC:	_____	Reviewer	_____	_____

WASTE TYPE	Estimated Weight (kg)	Error (kg)
CELLULOSICS Paper, plywood, wood, cloth, HEPA filters	_____	_____
Other organics Resins, oils, solvents, organic sludges	_____	_____
Plastics Bags, liners, waste, benelex, plexiglas	_____	_____
Rubber Gloves, aprons	_____	_____
Corroding metal - steel Drums, wastes, including stainless	_____	_____
Corroding metal - aluminum Wastes	_____	_____
Noncorroding metal Lead, tantalum, copper, etc.	_____	_____
Solid inorganic wastes Glass, ceramics, graphite, etc.	_____	_____
Inorganic sludges	_____	_____
Cements/Estimated water content (wt%)	_____	_____
Totals	_____	_____

Comments (note any descrepancies):

_____

_____

_____

Figure 8-4. Visual Data Form
(Continued)
EXAMPLE ONLY

8.4.2 Data Validation

All gas analysis data must be reviewed and approved prior to being reported. This validation process will include three levels of review, outlined in greater detail in the introduction to Section 8.0. In addition to these requirements, review at the generation and site project levels shall verify that the criteria in Table 3-7 have been met. The laboratory data review process must be detailed in site QAPjPs and/or implementing SOPs.

8.4.3 Data Reporting

Each laboratory analyzing samples will be required to submit data reports for each analytical batch. Data must be reported on approved standard forms. Report forms that meet QAPP requirements are included in this subsection as examples only. These forms or approved equivalent forms may be used for reporting purposes. The text identifies the information that must appear on individual data forms. Reporting data forms must include the following:

1. Cover Page

   Cover page with laboratory name, project identification, and approval/release signatures of the laboratory Manager or laboratory Project Manager, and laboratory QA Officer. A table listing field sample numbers, associated laboratory sample numbers, laboratory QC samples analyzed in association with field samples, and the analytical method used. DOE-WIPP cover page form, Cover Page GA (Figure 8-5), may be used.

2. Case Narrative

   A case narrative describing any problems encountered during sample analysis, or deviations from the referenced procedures.

3. Reporting Forms

   a. Analysis data sheets listing the techniques used (MS, GC, or spectrophotometric) and the concentrations in volume percent of all target analytes in Table 3-7. For analytes not detected, the analyte Method Detection Limit (MDL) corrected for sample volume must be reported. Those target analytes detected in corresponding laboratory blanks must be flagged on the analysis data sheet. EPA Inorganic Analysis SOW data qualifying flags shall be used. These are as follows:

      C (Concentration) qualifier: Enter "B" if the reported value was obtained from a reading that was less than the Program Required Detection Limit (PRDL) but greater than or equal to the MDL. If the analyte was analyzed for but not detected, a "U" must be entered.

      Q qualifier: Specified entries and their meaning are as follows:

      E - the reported value is estimated because of the presence of interference. An explanatory note must be included.

      * - Duplicate analysis not within control limits.

DOE-WIPP
COVER PAGE - GAS ANALYSIS DATA PACKAGE

Lab Name: _____

Lab Code: _____ Analytical Batch No.: _____

DOE Sample No.	Lab Sample ID

Comments:

_____

_____

_____

_____

Release of the data contained in this hard copy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

Figure 8-5. Cover Page - Gas Analysis Data Package
EXAMPLE ONLY

The method qualifiers (M) are:
MS - Mass Spectrometry
GC - Gas Chromatography
C - Spectrophotometry

In addition, laboratory name, analytical batch number, field sample number, laboratory sample ID, date received, and date analyzed must be on the analysis data sheet. DOE-WIPP data Form 1, Gas Analysis Data Sheet (Figure 8-5a).

b. Calibration form listing the accepted and measured values of calibration verification standards and the calculated percent recovery for each analyte listed in Table 4-1. This form must also contain the laboratory name and code, analytical batch number, initial calibration verification (ICV) and continuing calibration verification (CCV) source, method identification, and calibration date and time. DOE-WIPP Form 2 (Figure 8-5b) may be used.

c. Laboratory blank summary form listing all associated laboratory blanks. This form must contain laboratory name, laboratory blank identification number, date and time blank was analyzed, field sample number, laboratory sample number and the time each sample was analyzed. Blanks with analyte levels greater than the PRDLs will be flagged. DOE-WIPP Form 3 (Figure 8-5c) may be used.

d. Duplicate sample results are entered along with the original sample results. The RPD between the two results are calculated and presented here. This form also contains the laboratory name, analytical batch number, sample and duplicate identification, and the date the sample and duplicate were run. DOE-WIPP Form 4 (Figure 8-5d) may be used.

e. Laboratory control standard results are entered along with the accepted value and the percent recovery. This form must contain the laboratory name, analytical batch number, and the date the gas LCS was analyzed. DOE-WIPP Form 5 (Figure 8-5e) may be used. If the LCS is run as the calibration verification, then this form is not required.

f. Copies of data system quantitation reports for all calibration standards, samples, and blanks.

g. Copies of all raw data, including chromatograms and standard preparation records.

4. Copies of all sample chain-of-custody forms.

8.5 Organic Gas Analysis

Required procedures for data reduction and reporting of organic gas analysis results are given in this section. Specific equations and procedures must be detailed in QAPjPs and SOPs, as appropriate.

DOE-WIPP
FORM 1
GAS ANALYSIS DATA SHEET

DOE SAMPLE NO.

Lab Name: _____

Lab Code: _____

Analytical Batch No.: _____

Lab Sample ID: _____

Date Received: _____

Analysis Date: _____

Concentration Units (Vol. %)

Analyte	Concentration	C	Q	M
Argon				
Carbon Dioxide				
Carbon Monoxide				
Hydrogen				
Nitrogen				
Nitrogen Oxides				
Oxygen				
Methane				
Ethane				
Propane				

Figure 8-5a. Gas Analysis Data Sheet
EXAMPLE ONLY

DOE-WIPP
FORM 2
CALIBRATION VERIFICATION

Lab Name: _____

Lab Code: _____ Analytical Batch No.: _____

ICV Source: _____

Analysis Date: _____ CCV Source: _____

Analyte	M	ICV True	ICV Found	ICV %R	CCV1 True	CCV1 Found	CCV1 %R	CCV2 True	CCV2 Found	CCV2 %R	CCV3 True	CCV3 Found	CCV3 %R
Argon													
Carbon Dioxide													
Carbon Monoxide													
Hydrogen													
Nitrogen													
Nitrogen Oxides													
Oxygen													
Methane													
Ethane													
Propane													

Figure 8-5b. Calibration Verification Form
EXAMPLE ONLY

DOE-WIPP
FORM 3
BLANKS

Lab Name: _____

Lab Code: _____

Analytical Batch No.: _____

Analysis Date: _____

Concentration Units (Vol. %)

Analyte	Laboratory Blanks			
	ICB	CCB1	CCB2	CCB3
Argon				
Carbon Dioxide				
Carbon Monoxide				
Hydrogen				
Nitrogen				
Nitrogen Oxides				
Oxygen				
Methane				
Ethane				
Propane				

Figure 8-5c. Laboratory Blanks Form
EXAMPLE ONLY

DOE-WIPP
FORM 4
DUPLICATES

DOE SAMPLE NO.

Lab Name: _____

Lab Code: _____

Analytical Batch No.: _____

Analysis Date: _____

Concentration Units (Vol. %)

Analyte	Sample	Duplicate	RPD
Argon			
Carbon Dioxide			
Carbon Monoxide			
Hydrogen			
Nitrogen			
Nigtrogen Oxides			
Oxygen			
Methane			
Ethane			
Propane			

Figure 8-5d. Laboratory Duplicates Form
EXAMPLE ONLY

DOE-WIPP
FORM 5
LABORATORY CONTROL SAMPLE

Lab Name: _____

Lab Code: _____ Analytical Batch No.: _____

Analysis Date: _____ Gas LCS Source: _____

Analyte	LCS Gas (Vol. %)		
	True	Found	%R
Argon			
Carbon Dioxide			
Carbone Monoxide			
Hydrogen			
Nitrogen			
Nitrogen Oxides			
Oxygen			
Methane			
Ethane			
Propane			

Figure 8-5e. Laboratory Control Sample Form
EXAMPLE ONLY

8.5.1 Data Reduction

All organic analyte concentrations shall be quantified using average relative response factors for internal standard quantitation and average response factors for external standard quantitation. Target compound concentrations shall not be blank corrected. Results from blanks run in association with samples shall be reported separately, flagging any target compounds detected. Forward library search of the latest NIST mass spectral data base for the identification of unknown peaks with a total ion current area greater than 10% of the nearest (retention time) internal standard for internal standard quantitation and 10% of the largest target analyte peak, or ten times greater than the standard deviation of the background for external standard quantitation, shall be performed. Compounds identified by forward library searching shall be reported as TICs. Concentrations for TICs shall be calculated assuming a relative response factor equal to one using the nearest internal standard; if external standard quantitation is used the response factor from a chemically similar compound shall be used. All results shall be reported in part per million on a volume/volume basis (ppmv) and shall be limited to two significant figures. Detailed procedures for converting raw data to reportable results in ppmv are given in the organic section of the Guidance Manual, and procedures used must be detailed in site laboratory SOPs.

8.5.2 Data Validation

Data review at the generation and site project levels shall verify that criteria specified in Tables 3-6, 6-1, and 6-2 have been satisfied. This shall be done in addition to the criteria set in the general introduction to Section 8.0.

8.5.3 Data Reporting

VOC data shall be recorded and submitted on approved standard forms. USEPA CLP forms, appropriately modified for this program, will meet QAPP requirements. The minimum information that must appear on the individual data forms is identified in the text of this subsection. The following data forms must be included in a batch report:

1. GCMS reporting forms:

   EPA Contract Laboratory Program (CLP) reporting forms, modified to accommodate the Table 3-8 analytes and the appropriate header information, or approved equivalent forms may be used. For individual forms, the minimum information required is as follows:

   a. Analysis data sheets (Modified EPA CLP Form I) listing concentrations in ppmv of all target analytes in Table 3-8. For analytes not detected, the analyte MDL corrected for sample volume must be reported. Those target analytes detected in laboratory blanks must be flagged on the analysis data sheet. EPA CLP data qualifying flags shall be used. In addition, laboratory name, field sample number, laboratory sample number, date received and date analyzed must be on the analysis data sheet. TICs

must be reported on forms equivalent to that used for target analytes. The laboratories also have the option to report more than one sample per analytical data sheet and to include TICs on the same form.

b. Laboratory blank summary form (Modified EPA CLP Form IV) listing all samples analyzed with the associated laboratory blank. This form must contain laboratory name, laboratory blank identification number, date and time blank was analyzed, field sample number, laboratory sample number and the time each sample was analyzed.

c. Instrument performance criteria form (Modified EPA CLP Form V) to report the results of instrument performance testing with BFB and to summarize the date and time of analysis of samples run in conjunction with the BFB sample. This form must contain the laboratory name, BFB laboratory file identification number, date and time of BFB injection, instrument identification, field sample number, laboratory sample number, and the date and time each sample was analyzed.

d. Initial calibration form (Modified EPA CLP Form VI) listing the response factor or relative response factor for the analytes listed in Table 3-8 at all five calibration points, the average response factor or relative response factor for each analyte, and the percent relative deviation for each analyte across the five calibration points. For those analytes where a higher order regression curve is used, this must be indicated on the form. This form must contain the laboratory name, instrument identification and calibration date.

e. Continuing calibration form (Modified EPA CLP Form VII) listing the daily response factor or the daily relative response factor and the average response factor or the average relative response factor for the most recent initial calibration along with the percent difference between the daily and average response factor for each analyte listed in Table 3-8. This form must also contain the laboratory name, instrument identification, calibration date and time, laboratory file identification and the corresponding initial calibration date.

f. Internal standard area summary form (Modified EPA CLP Form VIII) listing the extracted ion current areas for each internal standard from the daily calibration along with the upper (two times the daily standard area) limit and the lower (0.5 times the daily standard area) limit for each. The internal standard areas for each standard from every sample run during a working day must be listed along with the field sample number. This form must contain the laboratory name, laboratory continuing calibration file identification, instrument identification, and the date and time the continuing calibration sample was analyzed.

g. Laboratory control sample form (Figure 8-6) listing measured concentration (ppmv), known concentration (ppmv), and percent recovery of all analytes present in the LCS. This form must contain the laboratory name, laboratory sample number, laboratory file number, and date and time analyzed.

h. Laboratory duplicate form (Figure 8-7) listing the concentration (ppmv) of all target analytes in Table 3-8 detected in the sample and duplicate sample along with the RPD between the two measurements. For those analytes not detected, the MDL corrected for sample volume must be reported. This form must contain the field sample number, laboratory sample number, laboratory file number, laboratory duplicate file number, date and time sample was analyzed, and date and time duplicate was analyzed.

HEADSPACE VOC
LABORATORY CONTROL SAMPLE

Lab Name: _____ Lab Sample ID: _____

Lab File ID: _____ Date Analyzed: _____

Time Analyzed: _____

Compound	Concentration (ppmv)		% Recovery
	Measured	Known	

Figure 8-6. Headspace VOC Laboratory Control Sample Form
EXAMPLE ONLY

HEADSPACE VOC
LABORATORY DUPLICATE

Lab Name: _____ Field Sample No.: _____

Lab File ID: _____ Date/Time Analyzed: _____

Lab Duplicate File ID: _____ Date/Time Analyzed: _____

Compound	Concentration (ppmv)		RPD
	Sample	Duplicate	
Acetone			
Benzene			
Bromoform			
1-Butanol			
2-Butanone			
Carbon Tetrachloride			
Chlorobenzene			
Chloroform			
Cyclohexane			
1,1-Dichloroethane			
1,2-Dichloroethane			
1,1-Dichloroethene			
cis-1,2-Dichloroethene			
Ethyl Benzene			
Ethyl Ether			
Methanol			
Methylene Chloride			
4-Methyl-2-pentanone			
1,1,2,2-Tetrachloroethane			
Tetrachloroethene			
Toluene			
1,1,1-Trichloroethane			
Trichloroethene			
1,1,2-Trichloro-1,2,2-trifluoroethane			
1,3,5-Trimethylbenzene			
1,2,4-Trimethylbenzene			
m-Xylene			
o-Xylene			
p-Xylene			

Figure 8-7. Headspace VOC Laboratory Duplicate Form
EXAMPLE ONLY

i. Copies of data system quantitation reports for all calibration standards, samples and blanks.

j. Copies of mass spectra for all reported TICs.

2. GC reporting forms:

EPA CLP forms modified to list only methanol, butanol, acetone, butanone and 4-methyl-2-pentanone, as well as the appropriate header information, may be used.

a. Analysis data sheets (Modified EPA CLP Form I) listing concentrations in ppmv of all alcohols and ketones in Table 3-8. For analytes not detected, the analyte MDL corrected for sample volume must be reported. Those target analytes detected in corresponding laboratory blanks must be flagged on the analysis data sheet. EPA CLP data qualifying flags may be used. In addition, laboratory name, field sample number, laboratory sample number, date received, and date analyzed must be on the analysis data sheet. The laboratories also have the option to report more than one sample per analytical data sheet.

b. Laboratory blank summary form (Modified EPA CLP Form IV) listing all samples analyzed with the associated laboratory blank. This form must contain laboratory name, laboratory blank identification number, date and time blank was analyzed, field sample number, laboratory sample number, and the time each sample was analyzed.

c. Initial calibration form (Modified EPA CLP Form VI) listing the response factors for the alcohols and ketones listed in Table 3-8 at all five calibration points, the average response factor or for each analyte, and the percent relative deviation for each analyte across the five calibration points. This form must contain the laboratory name, instrument identification and calibration date.

d. Continuing calibration form (Modified EPA CLP Form VII) listing the daily response factor and the average response factor for the most recent initial calibration along with the percent difference between the daily and average response factor for each alcohol and ketone listed in Table 3-8. This form must also contain the laboratory name, instrument identification, calibration date and time, laboratory file identification, and the corresponding initial calibration date.

e. Analyte identification form (Modified EPA CLP Form X) listing the detected analyte, analyte retention time from both columns, analyte retention time window for both columns, which column quantitation was performed on, laboratory name, field sample number, laboratory sample number, data file identification number, instrument identification, and column identification numbers.

f. Copies of all data system quantitation reports for all calibration standards, samples and blanks.

9.0 INTERNAL QC CHECKS AND FREQUENCY

Internal quality controls for the Program are required for both field and laboratory activities. Control of these activities shall be in compliance with ASME NQA-1, Elements 9 and 11. Processes affecting quality shall be controlled through operator training and routine review of performance. Test control shall be accomplished through the use of QC tests including routine measurements of performance standards, replicates, and blanks. These QC activities are the subject of this section.

9.1 Field Operations

The internal QC checks and their frequencies for the various field operations performed during the Program are described below.

9.1.1 Real-Time Radiography

The RTR system involves qualitative and semi-quantitative evaluations of visual displays. Operator training and experience are the most important considerations for assuring quality controls in regard to the operation of the RTR system and for interpretation and disposition of RTR results. Only trained personnel shall be allowed to operate RTR equipment. Standardized training requirements for RTR operators must be based upon existing industry standard training requirements and shall comply with the training and qualification requirements of ASME NQA-1, Element 2, except for Supplement 2S-2. Requalification of operators must be based upon evidence of continued satisfactory performance (primarily tape reviews) and must be done at least every two years. Unsatisfactory performance shall result in disqualification. Retraining and demonstration of satisfactory performance are required before an operator is again qualified to operate the RTR system.

A training drum with various container sizes holding different amounts of liquid must be periodically scanned by each operator. The videotape must then be reviewed by supervision to ensure that operator's interpretations remain consistent and accurate. Imaging system characteristics of the monitoring system must be verified on a routine basis.

Oversight functions include periodic tape reviews of accepted waste containers and must be performed by personnel other than the operator who dispositioned the waste container.

Independent replicate scans and replicate observations of the video output of the RTR process must be done under uniform conditions and procedures. Independent replicate scans must be performed on one out of every 20 drums. Independent observations of one scan (not the replicate scan) must be

made once during each work shift. As an additional QC check, the RTR results must be verified directly by the visual examination data. The site Project QA Officer shall be responsible for monitoring the quality of the RTR data and calling for remedial action when necessary.

9.1.2 Radioassay

RA is a quantitative measurement of key radioactive constituents in a waste drum and its contents. This isotopic assay system must be checked through the use of calibration and background drums as well as replicate readings.

As discussed in Section 6, RA must be performed on calibration and background drums. One calibration drum and one blank drum must be assayed at the beginning and end of each work shift. This procedure verifies routine performance of the measurement system.

Independent replicate measurements must be performed on 10 percent of the waste drums. The site Project QA Officer shall be responsible for monitoring the quality of the RA data and calling for remedial action when necessary.

9.1.2.1 Radioassay Operator Training

The present-day RA units are highly automated, computer-based systems. The instruments are computer-controlled using interactive software. Only trained personnel shall be allowed to operate the assay equipment. Standardized training requirements for radioassay operators must be based upon existing industry standard training requirements and shall comply with the training and qualification requirements of ASME NQA-1, Element 2, with the exception of Supplement 2S-2. Requalification of operators must be based upon evidence of continued satisfactory performance and must be done at least every two years. Unsatisfactory performance shall result in disqualification. Retraining and demonstration of satisfactory performance are required before an operator is again qualified to operate an RA system.

9.1.3 Headspace Sampling

Field QC checks shall be accomplished by preparation and submission of control samples from the field. Control samples shall be used to check equipment and sampler operation in the field and monitor ambient air quality in the vicinity of the waste container prior to sampling. Required field QC sample frequencies are summarized in Tables 9-1 and 9-2 for initial and routine samples.

Table 9-1. Summary of the Number and Type of Field QC Headspace Samples Required for First Two Bins Prepared at Each Site

First Two Bins	Volatile Organic Compounds	Gases	NO_x
Field blanks	1/drum	2/bin	2/bin
Sampling manifold blanks[a]	1/day	1/day	1/day
Field reference standards	1/bin or 5% of all samples	1/bin or 5% of all samples	1/bin or 5% of all samples
Field duplicate samples	1/bin or 5% of all samples	1/bin or 5% of all samples	1/bin or 5% of all samples

[a] One manifold blank sample must be collected, analyzed, and demonstrated clean prior to first use (all analyses), then at the specified frequency thereafter.

Table 9-2. Summary of the Number and Type of Field QC Headspace Samples

Routine	Proposed		
	Volatile Organic Compounds	Gases	NO_X
Field blanks	1/day	1/week	1/week
Sampling manifold blanks[a]	1/day prior to work	1/day prior to work	1/day prior to work
Field reference standards	1/bin or 5% of all samples	1/bin or 5% of all samples	1/bin or 5% of all samples
Field duplicate samples	1/bin or 5% of all samples	1/bin or 5% of all samples	1/bin or 5% of all samples

[a] One manifold blank sample must be collected, analyzed, and demonstrated clean prior to first use (all analyses), then at the specified frequency thereafter.

Field Blanks (FBs). FBs shall be used to evaluate background levels. FBs must initially be collected at a frequency of one per drum for VOCs and two per bin for gases and NO_x for the first two bins, then one per day for VOCs and one per week for gases and NO_x thereafter. The site Project Manager shall use the FB data to assess its impact on the sample results.

Sampling Manifold Blanks (SMBs). SMBs shall be used to assess the cleanliness of the sampling manifold prior to first use of the manifold for sample collection. After the initial cleanliness check, SMBs must be collected at a frequency of one per day for VOCs, gases, and NO_x. The site Project Manager shall use the SMB data to assess its impact on the sample results and have the responsibility to ensure corrective action measures are taken when SMB data indicates manifold contamination.

Field Reference Standards (FRSs). FRSs shall be used to assess the accuracy with which the sampling manifold can collect VOC, gas, and NO_x samples into SUMMA® canisters. FRSs must be collected at a frequency of one per bin or 5% of all samples collected, whichever is greater. FRS results shall be acceptable if the criteria specified in Tables 3-6 and 3-7 are satisfied. The site Project QA Officer shall have the responsibility to monitor and document FRS results. The site Project Manager shall also have the responsibility to ensure the corrective action measures are taken when these criteria are not satisfied.

Field Sample Duplicates. Duplicate samples will be collected to assess the precision with which the sampling procedure can collect samples into SUMMA® canisters. Field duplicates must be collected at a frequency of one per bin or 5% of all samples collected, whichever is greater. Field duplicate results shall be acceptable if the criteria in Tables 3-6 and 3-7 are satisfied. The site Project QA Officer shall have the responsibility to monitor and document the field duplicate results. The site Project Manager shall have the responsibility to ensure corrective action measures are taken when these criteria are not satisfied.

9.1.4 Visual Examination

The visual examination test operation shall consist of a semi-quantitative and/or quantitative evaluation of the waste drum contents. The test shall include weighing some or all of the contents of the drum. This may involve sorting and weighing materials by waste material category (Section 7.3).

Replicate measurements must be performed. They shall be used as a measure of the precision of the visual examination process. One in twenty, or at least one package from each drum, must be reweighed after all other drum contents are weighed.

A training program using simulated and real waste shall be developed based on the site waste form and waste management operation (e.g., waste segregation and packaging). This training program must be used to assess operator performance before actual waste characterization by visual examination. The site QAPjPs and supporting SOPs shall specify the training requirements for visual examination.

The documented and audio/visual taped results of the RTR evaluation of a drum must be available to visual examination personnel prior to or during the visual examination. The concurrence of two visual operators on the examination results is required to complete the visual examination.

9.2 Laboratory Operations

To assure that data of known and documented quality will be generated, each participating laboratory shall implement an Analytical Laboratory Quality Assurance Plan (Section 1.10). Laboratory QA plans and/or SOPs must specify qualitative and quantitative acceptance criteria for the QC checks for this program and corrective action measures taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, method proficiency samples, and blind audit samples.

9.2.1 Gas Analysis

The daily quality of analytical data generated in the laboratories is controlled by the implementation of the QAPjP and an Analytical Laboratory Quality Assurance Plan. Laboratory QA plans and/or SOPs must specify qualitative and quantitative acceptance criteria for the QC checks for this program and corrective action measures taken when these criteria are not satisfied. The types of internal laboratory QC to be considered include replicates, blanks, QC samples, calibration standards, and calibration verification samples.

In addition, acceptable method performance must be demonstrated prior to the analysis of samples.

9.2.1.1 Initial and Continuing Method Demonstration

All laboratories using these methods must demonstrate acceptable performance prior to the analysis of any samples. Demonstration of acceptable performance shall be achieved by participation in the Performance Demonstration Program. This shall require analyzing standards which contain all of the analytes listed in Table 3-7. The analysis of replicate samples must meet the criteria specified for precision, accuracy and method detection limits in Table 3-7. Demonstration of acceptable method performance must be repeated at a minimum of every six months. It shall be the responsibility of the laboratory QA Officer to monitor and document method performance. The laboratory QA Officer and

technical supervisor shall have responsibility to implement corrective actions when acceptable method performance is not met.

Field Reference Samples (FRSs). Commercially purchased gas standards shall be used to prepare FRSs. FRSs shall be collected by attaching clean evacuated canisters to the sampling manifold and filling them with the gas standard. The gas standards used to make the FRSs must be independent of those used for instrument calibrations. These standards must contain at least five of the target analyte gases (TAGs).

Laboratory Control Samples (LCSs). Commercially purchased gas standards shall be used to prepare LCSs. The gas standard used to prepare the LCSs must be independent of those used for instrument calibration. These standards must contain some but not necessarily all of the analytes listed in Table 3-7. The concentration of analytes must be in the linear range of the analytical instrument.

LCS results shall be acceptable if the criteria in Table 3-7 are satisfied. LCSs must be analyzed at a frequency of 10% of all field samples or one per analytical batch, whichever is greater. It shall be the responsibility of the laboratory QA Officer to monitor and document the LCS results. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when unacceptable results are generated.

9.2.1.2 Blank Analyses

Several different types of blank samples including field blanks, laboratory blanks, and sample manifold blanks must be analyzed.

Sampling Manifold Blanks (SMBs). SUMMA® canisters shall be taken to the field to monitor the cleanliness of the sampling manifold. SMBs shall be collected by attaching a clean canister to the sampling manifold and filling it with ultra high purity nitrogen or helium (99.999% pure). The site Project Manager must be notified if any analytes (except nitrogen) listed in Table 3-7 are detected at levels exceeding the PRDLs. It shall be the responsibility of the site Project Manager to ensure that corrective action measures are taken.

Laboratory Blanks (LBs). SUMMA® canisters shall be filled with ultra high purity nitrogen (99.999% pure) in the laboratory using the sample preparation manifold. The same analytical procedure used to prepare the field samples for analysis shall be used to prepare the LBs. LB results will be acceptable

if the concentrations of the TAGs (except nitrogen) are within the QAOs. LBs will be analyzed daily during analytical operations.

9.2.1.3 Duplicate Analysis

Laboratory Duplicate. The laboratory shall analyze field samples in duplicate. Sample canisters shall be analyzed in duplicate at a frequency of 10% of all samples thereafter, or one per analytical batch, whichever is more frequent. The site Project QA Officer may request additional duplicate sample analysis.

Single canisters analyzed in duplicate shall be used to assess laboratory precision. Laboratory duplicate results shall be considered acceptable for gases if the RPD is less than ±10% for concentrations greater than 10 times the PRDL and the absolute difference is less than ±PRDL for concentrations less than 10 times the PRDL. Laboratory duplicates for NO_x shall be acceptable if the RPD is less than ±20% for concentrations greater than 5 times the PRDL and the absolute difference is less than ±PRDL for concentrations less than 5 times the PRDL. Duplicates which do not meet these criteria should be flagged (see Section 8.4.3 on Data Reporting). It will be the responsibility of the laboratory QA Officer to monitor and document the results.

9.2.2 Organic Gases

To assure that data of known and documented quality will be generated, each participating laboratory shall implement an Analytical Laboratory Quality Assurance Plan. Laboratory QA plans and/or SOPs must specify qualitative and quantitative acceptance criteria for the QC checks for this program and corrective action measures taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, method proficiency samples, and blind audit samples.

9.2.2.1 Initial and Continuing Method Demonstration

All laboratories analyzing samples for this program must demonstrate acceptable method performance prior to the analysis of any samples. Demonstration of acceptable performance shall be achieved by analyzing commercially available (Scott Specialty Gases or equivalent) gas standards. These standards must contain all of the analytes listed in Table 3-8 at concentrations appropriate (two to five times the MDLs) to determine the parameters specified in Table 3-6. The analysis of replicate samples must meet the criteria specified for precision, accuracy and method detection limits in Table 3-6. Demonstration of acceptable method performance shall be repeated at a minimum of every six months. It shall be the responsibility of the laboratory QA Officer to monitor and document method

performance. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when acceptable method performance is not met.

Laboratory Control Samples (LCSs). Commercially purchased gas standards shall be used to prepare laboratory control samples. The gas standard used to prepare the LCSs must be independent of those used for instrument calibration. These standards must contain some but not all of the analytes listed in Table 3-8. The concentration of analytes must be in the linear calibration range of the analytical instrument. LCS preparation details shall be given in laboratory SOPs.

LCS results shall be acceptable if the criteria in Table 3-6 are satisfied. LCSs must be analyzed at a frequency of 10% of all field samples or one per analytical batch, whichever is greater. It shall be the responsibility of the laboratory QA Officer to monitor and document the LCS results. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when unacceptable results are generated.

9.2.2.2 Blank Analyses

Several different types of blank samples including field blanks, laboratory blanks and sample manifold blanks must be analyzed.

Laboratory Blanks (LBs). SUMMA® canisters must be filled with zero air in the laboratory. The same analytical procedure used to prepare the field samples for analysis shall be used for the LBs. LB results shall be acceptable if the concentrations of analytes listed in Table 3-8 are less than 50% of the PRQLs. LBs must be analyzed daily before sample analyses during analytical operations. Laboratory SOPs shall give the details of blank preparation and analysis. The laboratory technical supervisor shall have responsibility to implement corrective actions when blanks do not satisfy criteria specified.

Sampling Manifold Blanks (SMBs). SMBs shall be collected by attaching a clean SUMMA® canister to the sampling manifold and filling it with ultra-high purity air. The site Project Manager must be notified if VOCs are detected at levels exceeding 50% of the PRQLs listed in Table 3-8. It shall be the responsibility of the site Project Manager to ensure that corrective action measures are taken.

9.2.2.3 Duplicate Analysis

Duplicate samples must be generated in the laboratory and analyzed.

Laboratory Duplicate. The laboratory must analyze individual field samples in duplicate. Field canisters shall be analyzed in duplicate at a frequency of 10% of all field samples or one per analytical batch, whichever is greater.

Field canisters analyzed in duplicate shall be used to assess laboratory precision. Laboratory duplicate results shall be acceptable if the RPD is less than ±25%. It shall be the responsibility of the laboratory QA Officer at each participating laboratory to monitor and document the results.

9.2.2.4 Internal Standards

Internal standard area counts must be monitored for all samples. The GCMS shall be considered in-control if the area counts are within the range of 50% to 200% of the average internal standard area counts from the most recent five-point calibration. It shall be the responsibility of the laboratory QA Officer and technical supervisor at each participating laboratory to maintain control charts to monitor internal standard area counts and to initiate corrective actions as required.

10.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

In compliance with ASME NQA-1, Element 18, this section includes a description of the required performance and system audits and their frequency. Planned and scheduled audits shall be performed to verify compliance with all aspects of the QA/QC requirements for this program and to determine its effectiveness.

This program includes two types of evaluations. To verify compliance with the QAPP requirements, the WACCC shall review and approve the site-specific QAPjPs for compliance with the QAPP, and then the WIPP WACCC Audit and Surveillance Group shall perform planned and documented system audits of program activities described in those QAPjPs.

In addition, overall analytical system performance shall be evaluated by each analytical laboratory's participation in the Performance Demonstration Program (Section 1.11). DOE/WPO shall be responsible for administering the Performance Demonstration Program.

10.1 Audit and Performance Demonstration Program Personnel

The WACCC audit team shall include persons with the necessary analytical expertise and knowledge of DOE operations to address all the requirements established by this QAPP. The WACCC Chairperson is responsible for the selection of the audit team members. All auditors shall be independent of any direct responsibility for performance of the activities which they will audit. The Lead Auditor shall be trained, qualified, and certified in accordance with the requirements specified in ASME NQA-1, Supplement 2S-3.

DOE/WPO shall designate an organization that shall provide independent technical oversight and coordination of the inter-laboratory demonstration program to determine the performance characteristics of the analytical methods and participating laboratories.

10.2 Scope and Frequency of Audits

After approval of the site QAPjPs by the WACCC, the WACCC Audit/Surveillance Group shall conduct the audits. The WACCC shall develop and document an audit plan that identifies the audit scope, requirements, audit personnel, activities to be audited, organizations to be notified, applicable documents, schedule, and written procedures or checklists. Audits shall be performed in accordance with the written procedures or checklists. Elements that have been selected for audit shall be evaluated against the specified requirements in the QAPjP for the DOE site that is audited. Formal

audits must include evaluations of the site-specific field and laboratory activities and analytical laboratory protocols specified in the QAPjPs.

Formal WACCC audits of site program activities shall be performed prior to shipment of waste from that site and on at least an annual basis thereafter. Audit results shall be documented by auditing personnel and reported by the Lead Auditor to the WACCC Chairperson. The audit report shall be signed by the Lead Auditor and shall include the following information:

- A description of the audit scope.
- Identification of the auditors.
- Identification of persons contacted during audit activities.
- A summary of audit results, including a statement on the effectiveness of the quality assurance program elements which were audited.
- A description of each reported adverse audit finding in sufficient detail to enable corrective action to be taken by the audited organization.

Prior to the initial waste shipment from each generator/storage facility, a final report that includes the status or resolution of all findings resulting from a formal WACCC audit must be provided by the Lead Auditor to the WACCC Chairperson. Based on the final report, the WACCC Chairperson shall provide written approval for shipment of waste(s) to the WIPP to the appropriate DOE Operations Office Manager.

Copies of all WACCC audit reports identifying any nonconformances shall be sent to the appropriate DOE Operations Office and DOE/WPO. When corrective actions are required, a schedule that details all follow-up activities and final resolution shall also be provided by the WACCC to DOE/WPO and DOE/EM-30.

It is the responsibility of the WACCC Chairperson to ensure that all audit findings are resolved and the appropriate corrective actions implemented in a timely manner. The WACCC Chairperson must report all significant findings (i.e., nonconformances that may impact the quality of the data) to the appropriate DOE Operations Office. Follow-up action shall be taken by the responsible management organization to verify that corrective action is accomplished as scheduled. The site QAPjPs shall include a description of the organization(s) and person(s) responsible for tracking corrective actions.

Audit records for internal and external audits shall include audit plans, audit reports, written replies, and the record of completion of corrective actions.

In addition, the site Project QA Officer at each site shall schedule and conduct internal audits of program activities at least twice per year. The results of these audits shall be reported to the site Project Manager. These internal audit reports shall be maintained as part of the project files. The QAPjPs shall include a description of the roles and responsibilities related to this internal audit requirement.

10.3 Scope and Frequency of Performance Demonstration Program

Each laboratory must demonstrate its ability to meet the data quality assurance objectives for the analytes of interest prior to receiving actual waste samples. Single blind audit cylinders shall be prepared and distributed to each of the laboratories participating in the program. The specific analytical criteria that analytical laboratories must meet prior to participation in the program are described in the Performance Demonstration Program Plan. The DOE-designated, independent organization shall compile, review, and report the results of the performance evaluation to DOE/WPO.

The initial analytical performance demonstration for the participant laboratories shall be conducted and a demonstration of their adequacy documented, prior to analyzing WIPP waste characterization samples. Thereafter, analytical laboratories shall be reevaluated semi-annually. DOE/WPO shall provide written notification of the adequacy of an analytical laboratory and approval of its participation in the program to the appropriate DOE Operations Office management.

11.0 PREVENTIVE MAINTENANCE

As specified by ASME NQA-1, Element 10, inspection required to verify comformance of a item or activity shall be planned, executed, and documented. Preventive maintenance must have two aspects: (1) a schedule of preventive maintenance activities to ensure accuracy of measurement system and minimize downtime; and (2) a collection of critical spare parts and backup systems and equipment.

In compliance with ASME NQA-1, Element 12, tools, gauges, instruments, and other measuring and test equipment used for activities affecting the quality of this program shall be controlled by proper handling and storage, and at specified periods calibrated and adjusted to maintain the necessary accuracy. Calibration requirements discussed in Section 6.0 describe how field and laboratory equipment and instrumentation shall be kept in working order. The manufacturer's operating procedures that have been developed for maintaining instruments, should be used for developing calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, and service arrangements for equipment. Calibration and maintenance of field and laboratory equipment and instrumentation shall be in accordance with manufacturers' specifications or applicable test specifications and shall be documented. The QAPjPs or other implementing documents shall include a description of how calibration and maintenance of field and laboratory equipment and instrumentation shall be documented.

Operational sites shall be responsible for performing routine maintenance and shall keep tools and spare parts available to conduct routine maintenance. Maintenance that cannot be performed by equipment managers shall be performed by a person certified or trained to repair the instrument. Verification of maintenance activities related to headspace gas sampling shall be performed in accordance with site-specific SOPs. Instruments shall be calibrated to proper specifications following maintenance to ensure proper completion of the maintenance procedure. The date of maintenance shall be recorded in master calibration/maintenance log books for the sampling manifold and associated instrumentation. Adequate spare parts shall be kept available to ensure that appropriate quality control measures are maintained. These parts and supplies are in addition to those that will be required in the normal course of events.

12.0 SPECIFIC AND ROUTINE PROCEDURES TO ASSESS DATA QUALITY

The precision and accuracy of all quantitative data generated during the characterization of waste to be sent to the WIPP must be routinely assessed. In accordance with ASME NQA-1, Element 11, characteristics to be tested and test methods to be employed shall be specified. Radioassay, head-space gas and VOC analyses, and portions of the visual examination yield quantitative data suitable for statistical analysis. The precision, accuracy, completeness, and comparability of these data shall be determined by conventional procedures as outlined below. Qualitative data, or descriptive information, generated by RTR and visual examination are not amenable to statistical analysis. However, these are complementary techniques yielding some similar data, and therefore visual examination results shall be used to assess the quality of RTR data when applicable.

The quality assurance objective for measurement data is to ensure that characterization data are of known and acceptable quality. Precision, accuracy, and completeness are measures essential to assessing the quality of the analysis data, and hence, to applying the data appropriately in the decision-making process.

The quality assurance objectives for analytical data from the samples collected shall include the descriptions for precision, accuracy, representativeness, comparability and completeness, and are discussed in Section 3.5. The specific equations to assess precision, accuracy, completeness, comparability, MDL, and LOQ for the program are as follows.

12.1 Precision

Precision is a either expressed as the RPD for duplicate measurements or as the RSD for three or more replicate measurements.

For duplicate measurements, the precision expressed as the RPD is calculated as shown below:

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

where C_1 and C_2 are the two values obtained by analyzing the duplicate samples. 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 shown below:

$$RSD = s / \bar{y} * 100$$

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

The standard deviation, s, is defined as follows:

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

where y_i is the measured value of the ith replicate sample analysis measurement, and n equals the number of replicate analyses.

12.2 Accuracy

Accuracy is expressed as the percent recovery (%R).

For situations where a standard reference material is used, the percent recovery (%R) is calculated as shown below:

$$\%R = \frac{C_m}{C_{srm}} * 100$$

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

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

$$\%R = \frac{S - U}{C_{sa}} * 100$$

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.

12.3 Completeness

Laboratory completeness, expressed as the percent complete (%C), is calculated as follows:

$$\%C = \frac{V}{n} * 100$$

where V is the number of valid analytical results obtained, and n is the total number of determinations required for the actual number of samples collected.

12.4 Comparability

Comparability is expressed as percent correlation. The formula for comparability is as follows:

$$Comparability = \frac{CC}{TD} * 100$$

where:

CC = total number of drums with correctly identified waste items

TD = total number of drums inspected by both procedures

12.5 Method Detection Limit

The MDL for all measurements is defined as follows:

$$MDL = t_{(\eta-1, 1-\alpha=.99)} * s$$

where $t_{(\eta-1,\ 1-\alpha\ =\ .99)}$ is the t distribution value appropriate to a 99% confidence level and a standard deviation estimate with η-1 degrees of freedom, and s is the standard deviation of replicate measurements.

12.6 Limit of Quantitation

The LOQ for all measurements is defined as follows:

$$LOQ = 3.3 * MDL$$

where the MDL is the method detection limit.

13.0 CORRECTIVE ACTIONS

In compliance with ASME NQA-1, Element 16, conditions adverse to the QA/QC requirements specified in this document shall be promptly identified and corrected as soon as practical. The cause of any adverse condition that affects compliance with the QA/QC requirements shall be determined and action taken to preclude its recurrence. The identification, cause, and corrective action(s) for conditions that do not comply with the quality requirements for this Program must be documented and reported to appropriate levels of management as indicated throughout this section.

The status of work and program activities at the DOE facilities shall be monitored and controlled by the site Project Manager and site Project QA Officer. This status shall include (1) nonconformance identification, documentation, and reporting, and (2) operational variance identification, documentation and reporting.

13.1 Nonconformance

Nonconformances are uncontrolled and unapproved deviations from an approved plan, procedure, or expected result. Nonconforming items and activities are those which do not meet the program requirements, procurement document criteria, or approved work procedures. In compliance with ASME NQA-1, Element 15, nonconforming items shall be identified and segregated, and the affected organization(s) notified. Examples of potential nonconforming items that shall be addressed in the QAPjPs include wastes that do not meet the experimental program requirements (Molecke, 1990a, 1990b; Lappin et al., 1991) or RCRA-regulated hazardous wastes that are mislabeled. Disposition of nonconforming wastes or other items shall be identified and documented. The QAPjPs shall identify the person(s) responsible for evaluation and disposition of nonconforming items, and include referenced procedures for controlling them.

Nonconformances may be detected and identified by:

- Project Staff - During the performance of field operations, supervision of subcontractors, and preparation and verification of numerical data validation
- Laboratory Staff - During the preparation for and performance of laboratory testing, calibration of equipment, quality control activities, and laboratory data validation
- Quality Assurance Personnel - During the performance of oversight or audits
- WIPP WACCC Audit and Surveillance Personnel - During the performance of audits or surveillance of program activities.

Each nonconformance shall be documented by the personnel identifying or originating it. For this purpose, a nonconformance report including, as appropriate, results of laboratory analysis, quality control tests, audit reports, internal memorandum, or letters, shall be prepared. The nonconformance report must provide the following information:

- Identification of the individual(s) identifying or originating the nonconformance
- Description of the nonconformance
- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted
- Schedule for completing the corrective action
- Any required approval signatures.

The site Project QA Officer shall be responsible for developing a plan to identify and track all nonconformances and report this information to the DOE Operations Office. Documentation of nonconformances shall be made available to the site Project Manager, who is responsible for notifying project personnel of the nonconformance. In addition, the site Project QA Officer shall prepare a written report regarding significant nonconformances which could impact the results of the work and indicate the planned or implemented corrective action to the DOE Operations Office. Completion of the corrective action for significant nonconformances must be verified by the site Project QA Officer and, if applicable, the WIPP WACCC Audit and Surveillance personnel during scheduled audits.

13.2 Site Operational Variances

Variances are approved and controlled changes to approved plans or procedures caused by unusual or nonroutine occurrences that affect operations but not the ability to achieve the performance standards or quality requirements specified in a site QAPjP. When a variance is required, the person identifying the need for the variation must notify the site Project Manager and site Project QA Officer. A Record of Variance (Figure 4-2) must be completed prior to initiation of the activity to document the variation from normal, approved procedures. The site Project QA Officer shall assess the significance of the variance and determine whether further notifications are required.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Program established in compliance with ASME NQA-1 requires routine reports to management on the status of program activities. This section identifies the required reports and frequencies. In compliance with ASME NQA-1, Element 2, the QAPjPs shall identify the responsible organization(s) and person(s) and describe procedure(s) for providing QA reports to management to assess the adequacy of the Program and ensure its effective implementation. Each DOE facility participating in the Program shall identify the individuals responsible for QA reports and describe the type and frequency of those reports in the site QAPjP. Pertinent QA/QC information shall be reported to the site Project Manager and the site Project QA Officer to allow assessment of the overall effectiveness of the Program.

The site Project QA Officer shall, at a minimum, summarize in a monthly report to the site Project Manager, who in turn shall report to the DOE Operations Office all relevant information on the QA/QC activities during the period. This report shall include the following applicable information:

- Changes in the QAPjP
- Significant QA/QC problems, recommended solutions, and corrective actions taken
- Assessment of QC data gathered over the period, the frequency of analyses repeated due to unacceptable QA performance, and, if available, the reason for the unacceptable performance and corrective action taken
- Discussion of whether the QA objectives have been met, and any resulting impact on decision making
- Limitations on the use of the measurement data
- Status of Performance Demonstration sample results
- Results of audits and surveillances.

In addition to this monthly reporting requirement, the site Project QA Officer shall report all nonconformances as described in Section 13.0 to the DOE Operations Office. In accordance with ASME NQA-1, Element 16, conditions adverse to quality shall be identified, documented, and reported to management, and all follow-up action tracked to final closure. The WACCC Chairperson shall report all audit or surveillance findings to the DOE Operations Office (Section 10.0). DOE/WPO shall provide the results of the Performance Demonstration Program and notification of the analytical laboratory's adequacy in meeting program requirements to the DOE Operations Office (Section 10.0).

15.0 DEFINITIONS

ABSOLUTE CANISTER PRESSURE - The sum of the pressure indicated on the canister pressure gauge and the ambient barometric pressure.

ACCURACY - The degree of agreement between a measured value and an accepted reference or true value.

ANALYSIS DATE/TIME - The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

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

ANALYTICAL BATCH - A suite of samples of samples of similar matrix processed as a unit. This unit must not exceed 20 samples.

ANALYTICAL METHOD - Defines the 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 media 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. Note the following are all defined as analytical samples: WIPP and non-WIPP samples, duplicate samples, laboratory control sample (LCS), and field and manifold blanks.

AUDIT/APPRAISAL - A planned and documented activity performed in accordance with procedures to determine, by examination and evaluation of objective evidence, the adequacy of and extent to which applicable elements of the quality assurance program have been developed, documented, and effectively implemented in accordance with specified requirements. Audits can be internal examinations of programs or activities under an organization's control and within its organizational structure or external examinations of programs or activities of another organization.

BIN CASE - A report of all the information compiled pertinent to a bin in support of the characterization of the bin contents and its shipment to WIPP. This report may include a cover sheet, case narrative, leak test data, RA and RTR forms, visual examination forms, analytical batch report(s), COCs, transportation records, and validation documentation.

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

CALIBRATION BLANK - A volume of ultra pure gas containing undetectable quantities of analytes.

CHAIN OF CUSTODY - A set of procedures established to ensure that sample data integrity is maintained.

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

COMPLETENESS - The percentage of measurements made which are judged to be valid measurements. The completeness goal is to generate a sufficient amount of valid data based on project needs.

CONDITION ADVERSE TO QUALITY - An 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 which, if uncorrected, could have a serious effect on safety or operability.

CONTINUING CALIBRATION - Analytical standards run periodically to verify the calibration of the analytical system.

CONTROL LIMITS - A 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.

CORRELATION COEFFICIENT - A number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are the closer the value to one. Determined on the basis of the least squares line.

DATA QUALITY OBJECTIVES (DQO) - Qualitative and quantitative statements that describe the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data. Data quality objectives are determined based on the end uses of the data to be collected.

DATA REDUCTION - Operations necessary to correct data from the raw form to a final form as required by the customer.

DAY - Unless otherwise specified, day shall mean calendar day.

DOE CONTRACTOR - Includes any prime contractor or subcontractor subject to the contractual provisions of 48 CFR Part 923.70, 48 CFR Part 970.23, or other contractual provisions where DOE has elected to enforce ES&H requirements by specific negotiated contract provisions.

DOE OPERATIONS - Those DOE managed, directed, or funded activities for which the Department has responsibility for Environment, Safety and Health (ES&H).

DOE PROGRAM - An organized set of activities within a resource area having common objectives based on strategy set forth to meet assigned goals.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

FIELD BLANKS (FB) - Field blanks are 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. Sample canisters connected to the manifold adjacent to one another will be filled simultaneously. Field duplicates are used to document the precision of the sampling and analysis process.

FIELD ORGANIZATION - The first line DOE field element that carries the organizational responsibility for (1) managing and executing assigned programs, (2) directing contractors who conduct the programs, and (3) assuring that environment, safety, and health are integral parts of each program.

FIELD REFERENCE SAMPLES - Standard samples containing known concentrations of target analytes introduced through the sampling manifold. They are used to identify any bias in the sampling process.

FIELD SAMPLE - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique DOE Sample Number.

FREQUENCY (10%) - A frequency specification during an analytical sequence allowing for no more than 10 analytical samples between required quality control measurements, as specified by the WIPP-QAPP.

GAUGE PRESSURE - The 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 constitutes a means of accomplishing a task which has been found acceptable to the responsible agency. The word "should" is used to denote guidance material.

PROTOCOL - Material that constitutes the absolute minimum requirements for compliance with a given program. The word "shall" is used to denote these requirements. Verbatim compliance with protocols is mandatory.

PURGE AND TRAP - An analytical technique used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by 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 (QA) - All those planned and systematic actions necessary to provide adequate confidence that a facility, structure, system, or component will perform satisfactorily and safely in service. The goal of quality assurance is to assure that: research, development, demonstration, scientific investigations, and production activities are performed in a controlled manner; that components, systems, and processes are designed, developed, constructed, tested, operated, and maintained according to engineering standards, quality practices, and Technical Specifications/Operational Safety Requirements; and that resulting technology data are valid and retrievable. Quality assurance includes quality control, which comprises all those actions necessary to control and verify the features and characteristics of a material, process, product, or service to specified requirements.

QUALITY ASSURANCE OBJECTIVES - The characteristics of data that are associated with its ability to satisfy a given purpose or objective. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

QUALITY ASSURANCE OVERVIEW - An organized set of activities performed as independent functions. Its purpose is to assure that all aspects of quality-related activities at the program, project, and contractor level of management are adequately addressed. Such activities include:

(1) Periodic and timely reviews of program/project documents, activities, actions and plans;

(2) Review of new major procurements and management and operating contracts;

(3) Review of completed packages for management and operating contracts; and

(4) Review of DOE Orders with relevance to the incorporation of the DOE quality assurance policy, where necessary.

QUALITY ASSURANCE PLAN - A document that contains or references the quality assurance elements established for an activity, group of activities, a scientific investigation or a project and describes how conformance with such requirements is to be assured for structures, systems, computer software, components, and their operation commensurate with (1) the scope, complexity, duration, and importance to satisfactory performance, (2) the potential impact on environment, safety and health, and (3) requirements for reliability and continuity of operation.

QUALITY CONTROL (QC) - A routine application of procedures for controlling the monitoring process. Quality Control is the responsibility of all those performing the hands-on operations in the field and in the laboratory.

RADIOASSAY (RA) - Assay methods used to identify and quantify radionuclides in TRU waste.

REAL-TIME RADIOGRAPHY (RTR) - A nondestructive testing method that utilizes X-rays to inspect the physical waste form.

RECOVERY - Usually expressed as a percent. The numerical ratio of the amount of analyte measured by the laboratory method divided by the known amount of analyte added to the matrix (i.e., spiked sample) to be analyzed.

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

RESIDUAL MATERIAL - Anything not characterized as a waste item (e.g, vermiculite).

RESOLUTION - Also termed separation, the separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RUN - A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the WIPP-QAPP.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLING MANIFOLD BLANKS - Samples of high purity gas used to purge sampling equipment. They are collected after the sampling manifold has been cleaned and prior to sampling. These blanks are useful in documenting adequate cleaning of sampling equipment.

SAMPLE NUMBER - A unique identification number that is designated for each sample. The Sample Number appears on all sample reports which document information on that sample.

STANDARD DEVIATION - The square root of the variance of a set of values.

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

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

WASTE ITEMS - Easily identifiable discrete pieces/chunks of waste (e.g, rashig rings).

WASTE MATERIAL CATEGORY - A type of material specified by Sandia as a controlling variable for gas generation.

WEAPONS GRADE PLUTONIUM - An isotopic mix of plutonium used in the fabrication of nuclear weapons and whose mass fraction is dominated by the fissile nuclide plutonium-239.

16.0 LIST OF ACRONYMS

AE	Argonne National Laboratory-East Analytical Laboratory
ALARA	As Low As Reasonably Achievable
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing & Materials
AW	Argonne National Laboratory-West Gas Analytical Laboratory
BFB	Bromofluorobenzene
CCV	Continuing Calibration Verification
CH TRU	Contact Handled Transuranic
CLP	Contract Laboratory Program
COC	Chain of Custody
DOE	Department of Energy
DOE/AL	DOE Albuerque Operations Office
DOE/WPO	DOE WIPP Project Manager
DOT	Department of Transportation
DQO	Data Quality Objectives
EH-1	Assistant Secretary for Environment, Safety, and Health
EI	EG&G, Idaho VOC Analytical Laboratory
EM-1	Director of Office of Environmental Restoration and Waste Management
EM-20	Director of the Office of Quality Assurance and Quality Control
EM-30	Associate Director of the Office of Waste Operations
EPA	Environmental Protection Agency
FB	Field Blanks
FID	Flame Ionization Detector
FRS	Field Reference Sample
GC	Gas Chromatography
GCMS	Gas Chromatography/Mass Spectrometry
ICV	Initial Calibration Verification
IDC	Item Description Code
IDL	Instrument Detection Limit
IN	Idaho National Engineering laboratory
INEL	Idaho National Engineering Laboratory
LB	Laboratory Blanks
LCS	Laboratory Control Samples
LOQ	Limit of Quantification
MDL	Method Detection Limit
MMDDYY	Month-Day-Year Format
MS	Mass Spectrometry
NDA	Nondestructive Assay

NEIC	National Enforcement Information Center
NIST	National Institute of Standards and Technology
NR	Characterization not required to meet objective
NRC	Nuclear Regulatory Commission
ORNL	Oak Ridge National Laboratory
ORP	Office of Radiation Programs
P	Pressure
PAN	Passive/Active Neutron Counting
PRSD	Percent Relative Standard Deviation
PFTBA	Perfluorotributylamine
PID	Photoionization Detector
PNCC	Passive Neutron Coincidence Counting
ppmv	Parts per million by volume
PRDL	Program Required Detection Limit
PRQL	Program Required Quantitation Limit
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QC	Quality Control
RA	Radioassay
RCRA	Resource Conservation and Recovery Act
RFP	Rocky Flats Plant
RI	EG&G, Rocky Flats Plant Gas Analytical Laboratory
RO	EG&G, Rocky Flats Plan VOC Analytical Laboratory
ROI	Range of Interest
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
RTR	Real-time Radiography
SDG	Sample Delivery Group
SGS	Segmented Gamma Scan Counting
SMB	Sampling Manifold Blank
SNL	Sandia National Laboratories
SOP	Standard Operating Procedures
T	Temperature
TAL	Target Analyte List
TCL	Target Compound List
TIC	Tentatively Identified Compounds
TRAMPAC	TRUPACT-II Authorized Methods for Payload Control
TRAMPAC	Authorized Methods for Payload Control
TRU	Transuranic
TRUCON	TRUPACT-II Content Codes

TRUPACT-II	Transuranic Package Transporter
VOC	Volatile Organic Chemicals
VTSR	Validated Time of Sample Receipt
WAC	Waste Acceptance Criteria
WACCC	Waste Acceptance Criteria Certification Committee
WIPP	Waste Isolation Pilot Plant
WPO	WIPP Project Office

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