Department of Energy
Albuquerque Field Office
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JUL 01 1993

Ms. Judith Espinosa, Secretary
New Mexico Environment Department
P.O. Box 3149
1190 St. Francis Drive
Santa Fe, NM 87504-0968

Dear Secretary Espinosa:

Enclosed please find three additional documents which should be added to the Environmental Protection Agency supplemental detailed documentation delivered to your office on June 22, 1993.

The additional documentation consists of the following:

1. Quality Assurance Program Plan for the Waste Isolation Pilot Plant Experimental Waste Characterization Program, Revision 2 (Draft), October 12, 1992; *w/ DOE/EM/48063-Revision 1

2. Waste Isolation Pilot Plant Analytical Laboratory Procedures Manual (Table of Contents only), WP 12-AL;

3. WIPP Analytical Laboratory Quality Assurance Plan, WP 12-13.

If you have any questions or require further information, please contact Patrick J. Higgins, of my staff at (505) 845-5914.

Mark L Matthews for

W. John Arthur, III
Project Director
WIPP Project Integration Office

3 Enclosures

930701

WIPP Library

QAPP

Quality Assurance Program Plan
for the Waste Isolation Pilot Plant
Experimental-Waste Characterization Program

Environmental Restoration and Waste Manageme

Waste Isolation Pilot Plan

Revision 1
July 1991

DOE/EM/48063-

AND REVISION 2 Attached

Controlled Copy Number 6
Assigned To

Patrick J. Higgins, Jr.

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Department of Energy
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United States Government **Department of Energy**

memorandum

DATE: JUL 23 1991

REPLY TO ATTN OF: EM-342

SUBJECT: Department of Energy Office of Environmental and Waste Management, Office of Waste Operations, Quality Assurance Program Plan for the Waste Isolation Pilot Plant Experimental-Waste Characterization Program, Revision 1.0

TO: Distribution

This memorandum transmits Revision 1 of the referenced Quality Assurance Program Plan (QAPP) to users noted on the controlled distribution list. Individuals with controlled copies are requested to replace the Revision 0 of the QAPP with the current attached Revision 1.

The QAPP supports the Waste Isolation Pilot Plant (WIPP) Experimental Waste Characterization Program by: (1) establishing the quality assurance and quality control (QA/QC) requirements for producing transuranic (TRU) waste characterization data at the Department of Energy Facilities; (2) establishing the QA/QC requirements for WIPP verification of TRU waste characterization data; (3) establishing the QA criteria for the preparation, review, and approval of site-specific Quality Assurance Project Plans; and (4) providing a guidance manual.

Revision 1 of the QAPP has been reviewed by the Environmental Protection Agency (EPA), and the contents of the document meets all verbal comments provided by EPA. If written comments from EPA are significantly different, another revision may be required, prior to shipment, to address these comments. Revision 1 of the QAPP contains additional requirements, criteria, and guidance on inner-bag sampling, and bin sampling for volatile organic compounds to address EPA's No-Migration Determination.

If you have any questions, please contact Mark Duff at 301-353-7206.

Jill E. Lytle
Associate Director
Office of Waste Operations
Environmental Restoration and
Waste Management

Attachment

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QUALITY ASSURANCE PROGRAM PLAN
FOR THE WASTE ISOLATION PILOT PLANT
EXPERIMENTAL-WASTE CHARACTERIZATION PROGRAM
Revision 1

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

July 15, 1991

Approved by _____ Date 7-23-91
Associate Director of the Office of
Waste Operations (EM-30)

Approved by _____ Date 7-19-91
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 experimental-waste characterization 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

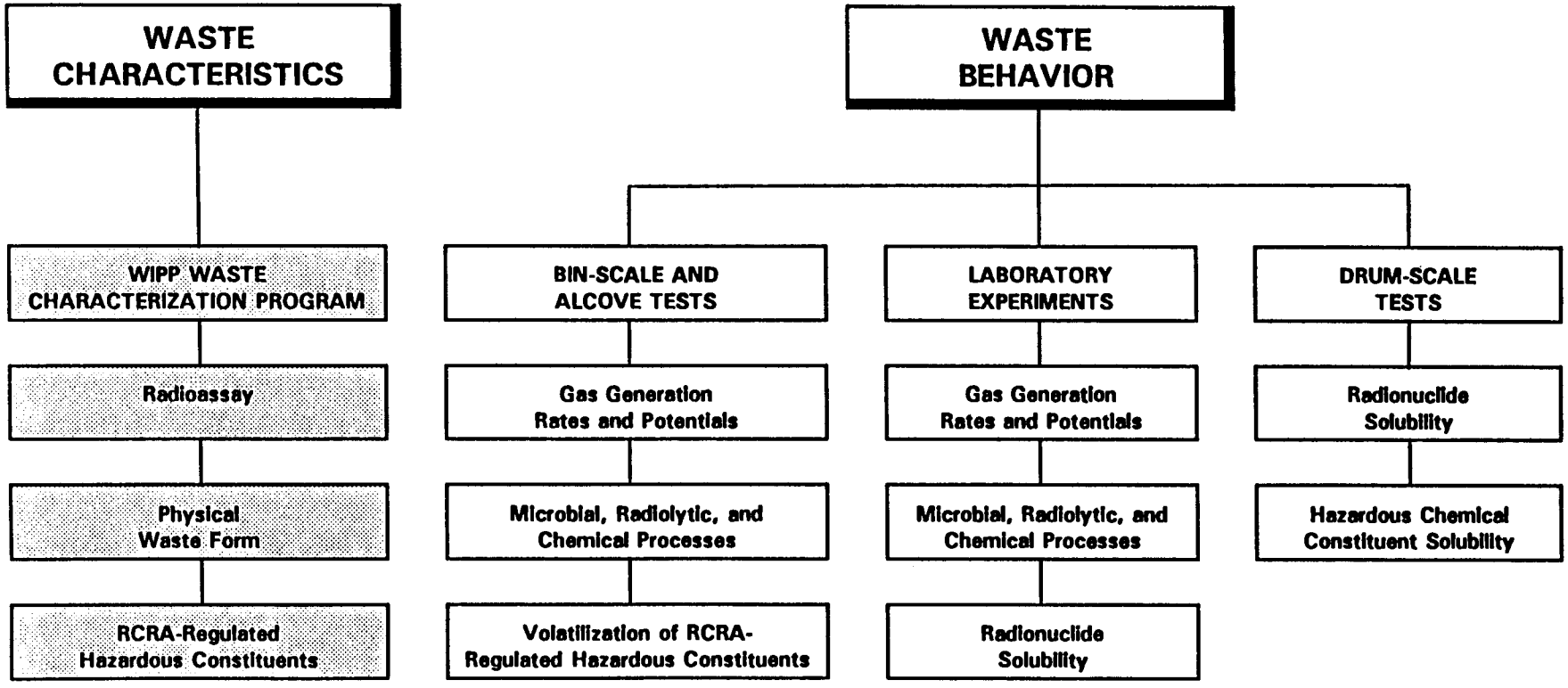


Figure 1-1. Aspects of WIPP Activities Related to Waste Characterization and Test Phase Programs

- 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 quality assurance activities and the requirement for those activities be identified in program plans. In the nuclear area, ASME NQA-1 is cited as the preferred standard for quality assurance. DOE Order 5820.2A (U.S. DOE, 1988) requires that DOE facilities managing nuclear materials comply with all applicable quality elements in ASME NQA-1. For the purposes of this document, "Element" refers to the specified Basic Requirement and its Supplement(s). 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

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.

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 WIPP test program are expected in 3 to 5 years after 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 EPA's conditional no-migration determination (NMD) (55 FR 47700), and includes quantification of hazardous constituents identified through the Program to support both compliance with the determination as well as future efforts by DOE to petition EPA for a long-term no-migration determination after completion of the Test Phase (Figure 1-2).

- *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.

From a technical perspective, then, this QAPP addresses the activities associated with TRU waste characterization efforts in general, and applies to all such activities conducted under the Program. All current 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).

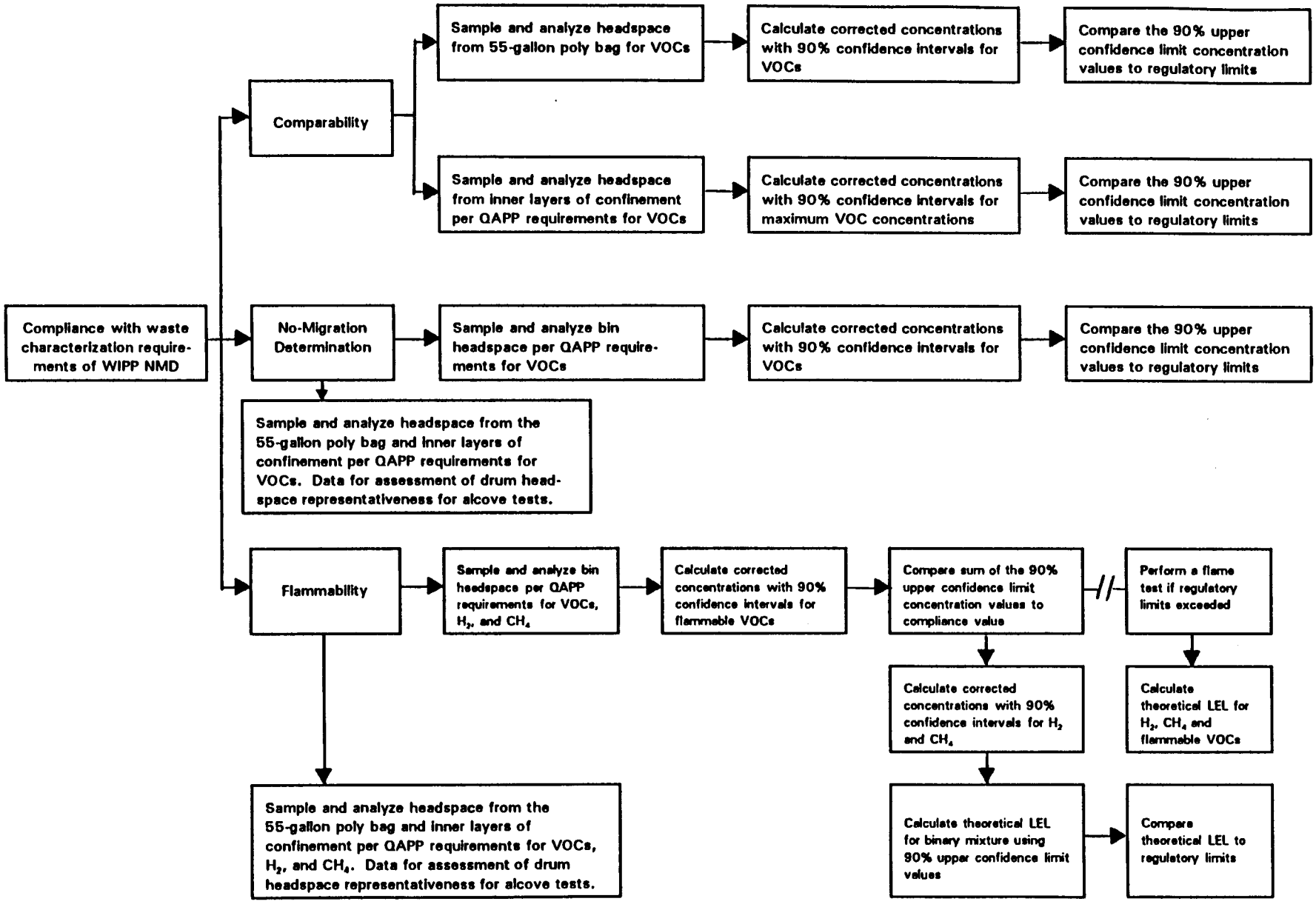


Figure 1-2. Overall Approach to Compliance With WIPP No-Migration Determination

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, E.N. Lorusso, and R.L. Hunter, 1991, "Rationale for Revised WIPP Bin-Scale Gas-Generation Tests with CH TRU Wastes at the Waste Isolation Pilot Plant," SAND90-2481, Sandia National Laboratories, Albuquerque, New Mexico.
- Department of Energy, 1991, "Waste Characterization Program Plan for WIPP Experimental Waste," DOE/WIPP 89-025, Current Revision, 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. A summary of the waste characterization requirements and the Program design is provided in Table 1-2.

1.3.1 Bin-Scale and Alcove Test Programs

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 a description of 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 containers of waste in accordance with SNL requirements from their inventory of TRU waste that can be 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. Figure 1-3 illustrates the sequence of events for the characterization of TRU waste to be included in the WIPP test program.

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 WIPP bin-scale and/or alcove test programs, and will be based on a statistically-valid sampling plan and results

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 Corroding Metals/Steel Corroding Metals/Aluminum Non-Corroding Metals Solid Inorganics Inorganic Sludges Cements/Additives Other organics (resins, oils, organic sludges, solvents)	Gases Argon, Oxygen Carbon Monoxide, Methane Carbon Dioxide, Ethane Hydrogen, Propane Nitrogen, Nitrogen Oxides Volatile Organic Compounds (VOCs) Acetone, Methanol Benzene, Methylene chloride Bromoform, 4-Methyl-2-pentanone Butanol, 1,1,2,2-Tetrachloroethane 2-Butanone, Tetrachloroethene Carbon tetrachloride, Toluene Chlorobenzene, 1,1,1-Trichloroethane Chloroform, Trichloroethylene Cyclohexane, 1,1,2-Trichloro-1,2,2-trifluoroethane 1,1-Dichloroethane, 1,3,5-Trimethylbenzene 1,2-Dichloroethane, 1,2,4-Trimethylbenzene 1,1-Dichloroethene, m-Xylene cis-1,2-Dichloroethene, o-Xylene Ethyl benzene, p-Xylene Ethyl ether
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 [d]	• 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.
[d] See Section 1.4 of the QAPP for a complete description of the DQOs.

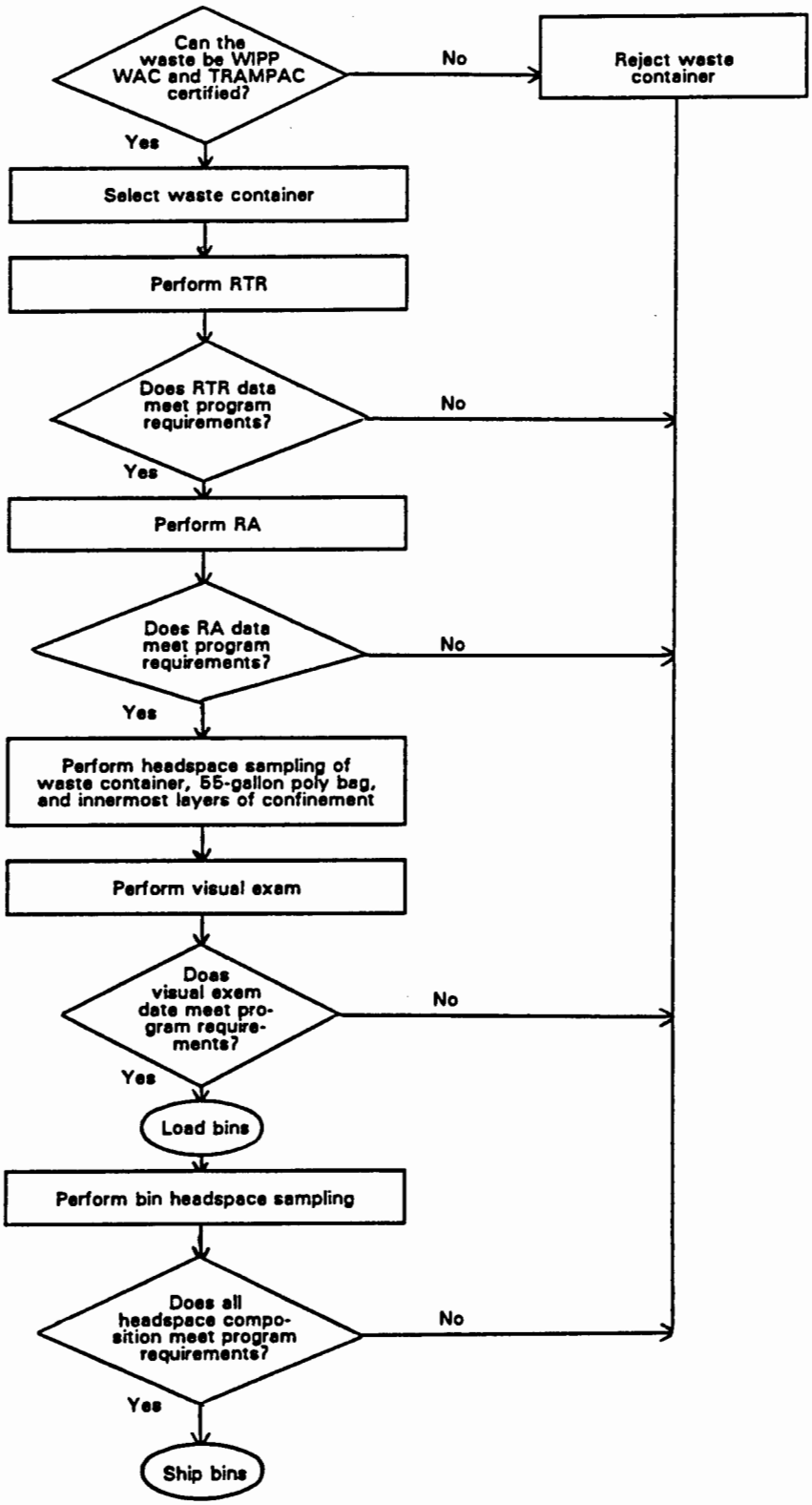


Figure 1-3. Sequence of Events for Waste Characterization

obtained from the experimental program. A detailed description of the methodology and the quantities and types of wastes that will be included in the WIPP test program is included in Lappin et al. (1991).

1.3.2 No-Migration Determination Waste Characterization Requirements

In response to the DOE's No-Migration Variance Petition (Petition) for the WIPP, EPA published its notice of the final conditional NMD for the WIPP on November 14, 1990 (USEPA, 1990). As a result of this NMD, DOE may place a limited amount of untreated TRU waste subject to the land disposal restrictions under RCRA in the WIPP for the purposes of testing and experimentation. The NMD imposes several conditions on such placement and is for a maximum of ten years. This QAPP includes the requirements for compliance with those conditions associated with the waste characterization requirements imposed by EPA. Compliance with the other conditions of the NMD (e.g., the air monitoring requirements at the WIPP) are addressed in the WIPP Part B permit application (USDOE, 1991b) and supporting WIPP documents. Figure 1-2 provides a schematic of the overall waste characterization approach for compliance with the NMD.

To comply with the requirements of the NMD associated with waste sampling and analysis, the following conditions must be met:

- *Headspace Sampling* - Obtain a representative headspace sample of gases and VOCs from the waste containers to be included in the WIPP test program. Within the constraints of the matrix to be sampled, samples must be collected from all layers of confinement that may contain sources of gases or VOCs.

- *Comparability* - Demonstrate, by waste type, that the composition of hazardous constituents in the headspace of containers is similar to those concentrations reported in the Petition. The headspace concentrations of the five major hazardous constituents reported by DOE must be less than or equal to the maximum allowable concentrations in the NMD (Table 1-3).

- *No-Migration Demonstration* - Demonstrate, by waste type, that the no-migration finding by EPA remains valid by demonstrating that for each container of waste to be sent to the WIPP (drum or bin), the mean headspace concentrations of three of the hazardous constituents reported in the Petition are less than or equal to the mean headspace concentrations included in the NMD (Table 1-4).

- *Flammability* - Demonstrate that each waste container emplaced underground at the WIPP has no layer of confinement containing flammable mixtures of gases (H_2 and CH_4) and VOCs (Table 1-5) or mixtures of gases and VOCs that could become flammable when mixed with air.

Table 1-3. Maximum Allowable Volatile Organic Compound Headspace Concentrations (Volume Percent) by Waste Type[a,b]

Constituent	Type I	Type II	Type III	Type IV
Carbon tetrachloride	0.08	0.18	0.58	8.18
Methylene chloride	0.44	0.84	0.50	1.42
1,1,1-Trichloroethane	1.88	5.68	2.12	14.96
Trichloroethylene	0.08	0.34	0.28	0.28
1,1,2-Trichloro-1,2,2-trifluoroethane	0.05	1.62	5.74	20.80

[a] Waste types are identified in USDOE, 1989.
[b] Concentration values from the No-Migration Determination (USEPA, 1990). These concentrations are obtained by multiplying the maximum VOC headspace concentrations reported in the Petition by two.

Table 1-4. Allowable Mean Volatile Organic Compound Headspace Concentrations (Volume Percent) by Waste Type[a,b]

Constituent	Type I	Type II	Type III	Type IV
Carbon tetrachloride	0.24	0.26	0.30	6.90
Methylene chloride	0.39	0.42	0.33	0.93
Trichloroethylene	0.25	0.28	0.29	0.38

[a] Waste types are identified in USDOE, 1989.
[b] Concentration values from the No-Migration Determination (USEPA, 1990). These concentration values are obtained by multiplying the mean VOC headspace concentrations reported in the Petition by ten.

Table 1-5. Flammable Volatile Organic Compounds[a] (VOCs) and their Lower Explosive Limit (LEL) Values[b,c]

VOCs	LEL (v/v%)
Acetone	2.5
Benzene	1.3
n-Butanol	1.4
2-Butanone	1.4
Chlorobenzene	1.3
Cyclohexane	1.3
1,1-Dichloroethane	5.6
1,2-Dichloroethane	6.2
1,1-Dichloroethene	6.5
cis-1,2-Dichloroethene	5.6
Ethyl benzene	1.0
Diethyl ether	1.9
Methanol	6.0
4-Methyl-2-pentanone	1.2
Toluene	1.2
1,2,4-Trimethylbenzene	0.9
1,3,5-Trimethylbenzene	1.4
o-Xylene	1.0
m-Xylene	1.1
p-Xylene	1.1

[a] VOCs identified as flammable by EPA for the NMD. EPA determined flammability by assessing potential fire hazards under WIPP conditions on a compound by compound basis.

[b] The lower explosive limit is also referred to as the lower flammable limit (ASTM, 1989 and NFPA, 1986).

[c] Only the most conservative lower explosive limit values are reprinted (NFPA, 1986 and De Renzo, 1986).

1.3.2.1 Headspace Sampling

Pretest waste characterization for the bin-scale tests will include sampling gases and VOCs from three areas within drums of TRU waste. These areas include (1) the drum headspace (i.e., the headspace directly under the drum lid), (2) the 55-gallon poly bag headspace, and (3) the innermost layers of confinement headspace. Drum headspace sampling will be included in Revision 2 of the QAPP to address future compliance during the alcove tests and operational phase. The 55-gallon poly bag and the innermost layers of confinement headspace sampling procedures are described in Sections 4.1 and 4.4, respectively. In addition, bin headspace samples shall be obtained (Section 4.5).

In order to demonstrate compliance in the future when drums could be directly emplaced in the WIPP, DOE must demonstrate to EPA's satisfaction that a drum headspace gas sample is representative of the gases and VOCs within the entire drum. Although compliance with specific conditions of the NMD during the bin-scale tests may be demonstrated based on the results of sampling and analyzing the headspace gases and VOCs in an experimental bin, samples must be obtained from all layers within the drums included in that bin. The data obtained from all the sampling locations will be evaluated by DOE to assess the need to obtain additional information on the concentrations of hazardous constituents within drums that may be emplaced in the WIPP during the alcove tests.

1.3.2.2 Comparability

To comply with the maximum concentration comparability condition of the NMD, headspace samples from within the 55-gallon poly bag and from the innermost layers of confinement shall be analyzed using Program-approved analytical procedures to determine the concentrations of carbon tetrachloride (CCl_4), methylene chloride (CH_2Cl_2), 1,1,1-trichloroethane (TCA), trichloroethylene (TCE), and 1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113) (Figure 1-4). The upper 90% confidence limit value of the highest measured concentration of each of these five major constituents found in the innermost layers of confinement shall be compared to the compliance concentrations listed in Table 1-3.

The upper 90% confidence limit value of the measured concentration of the five major hazardous constituents within the 55-gallon poly bag headspace shall also be compared to the allowable concentrations in Table 1-3. If this comparison and the one described above indicate that the highest concentrations of each of the five major hazardous constituents are less than the maximum allowable concentrations in Table 1-3, then DOE may place the contents of that drum into a bin for inclusion in the WIPP test program. If drums of waste are loaded into a bin prior to receipt of results from 55-gallon poly bag and innermost layers of confinement headspace sampling and analyses, the entire bin would be rejected if the data indicate that any drum is not in compliance with regard to the

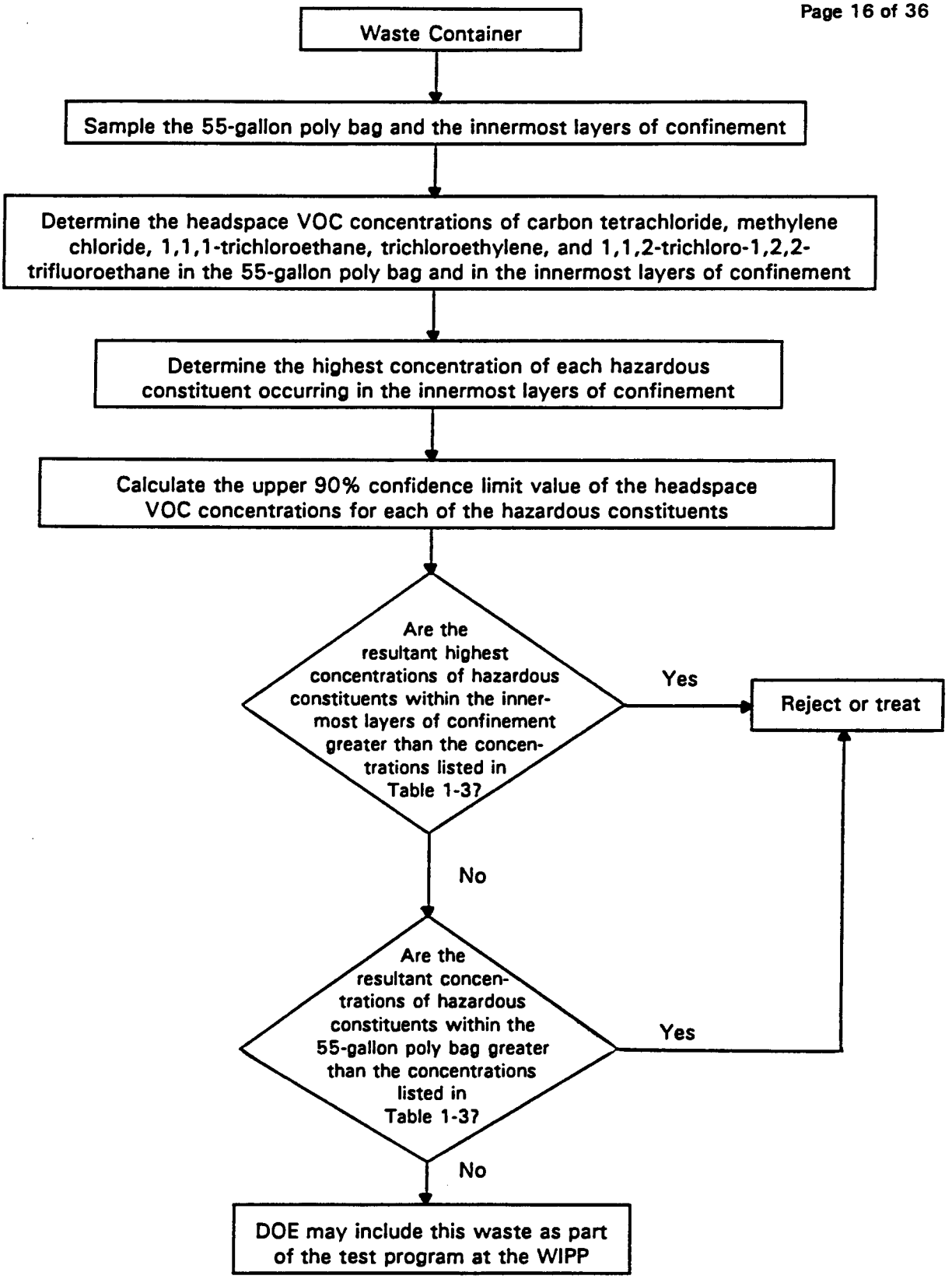


Figure 1-4. Comparability Flowchart

comparability criteria. Drum headspace sampling shall be included in Revision 2 of the QAPP. Results of drum headspace sampling will be compared to 55-gallon poly bag and innermost layers of confinement to assess demonstration of comparability.

1.3.2.3 No-Migration Demonstration

To comply with this condition of the NMD during the bin-scale tests, a headspace sample from within each experimental bin shall be collected (Section 4.5). Headspace samples from the experimental bins shall be analyzed using Program-approved analytical procedures to determine the concentrations of CCl_4, CH_2Cl_2, and TCE (Figure 1-5). Once determined, the upper 90% confidence limit value of the measured headspace concentrations of each of these hazardous constituents shall be compared (Section 12.6) to the allowable concentrations specified in Table 1-4. If this comparison indicates that the headspace concentrations of each of the three hazardous constituents are less than those specified, the bin may be emplaced in the WIPP as part of the WIPP test program.

1.3.2.4 Flammability

Figure 1-6 shows the overall approach to compliance with the flammability determination requirements. A representative sample shall be obtained from the bin headspace (Section 4.4) and analyzed for flammable VOCs (Table 1-5) using Program-approved procedures. Other headspace data obtained during waste characterization in support of the bin-scale tests shall be used to assess compliance for alcove tests.

Flammable VOCs

Bin headspace analytical results shall be evaluated to determine if significant levels of flammable VOCs are present in the bins included in the WIPP test program. Significant levels of flammable VOCs (Table 1-5) are defined as measured concentrations of 500 ppmv or greater. To perform this evaluation, the upper 90% confidence limit value of the measured concentrations (Section 12.6) of all analytes (Table 1-5) shall be summed. If the summed value exceeds 500 ppmv, a theoretical lower explosive limit (LEL) shall be calculated using the concentrations of the flammable VOCs plus hydrogen and methane. The flammability of the headspace sample shall be determined by using a modified version of ASTM Method E 681-85 (Section 7.6). For purposes of complying with the NMD, a headspace VOC mixture shall be considered flammable if, under the specified conditions of the test, the mixture propagates a flame from the ignition source to the test vessel walls.

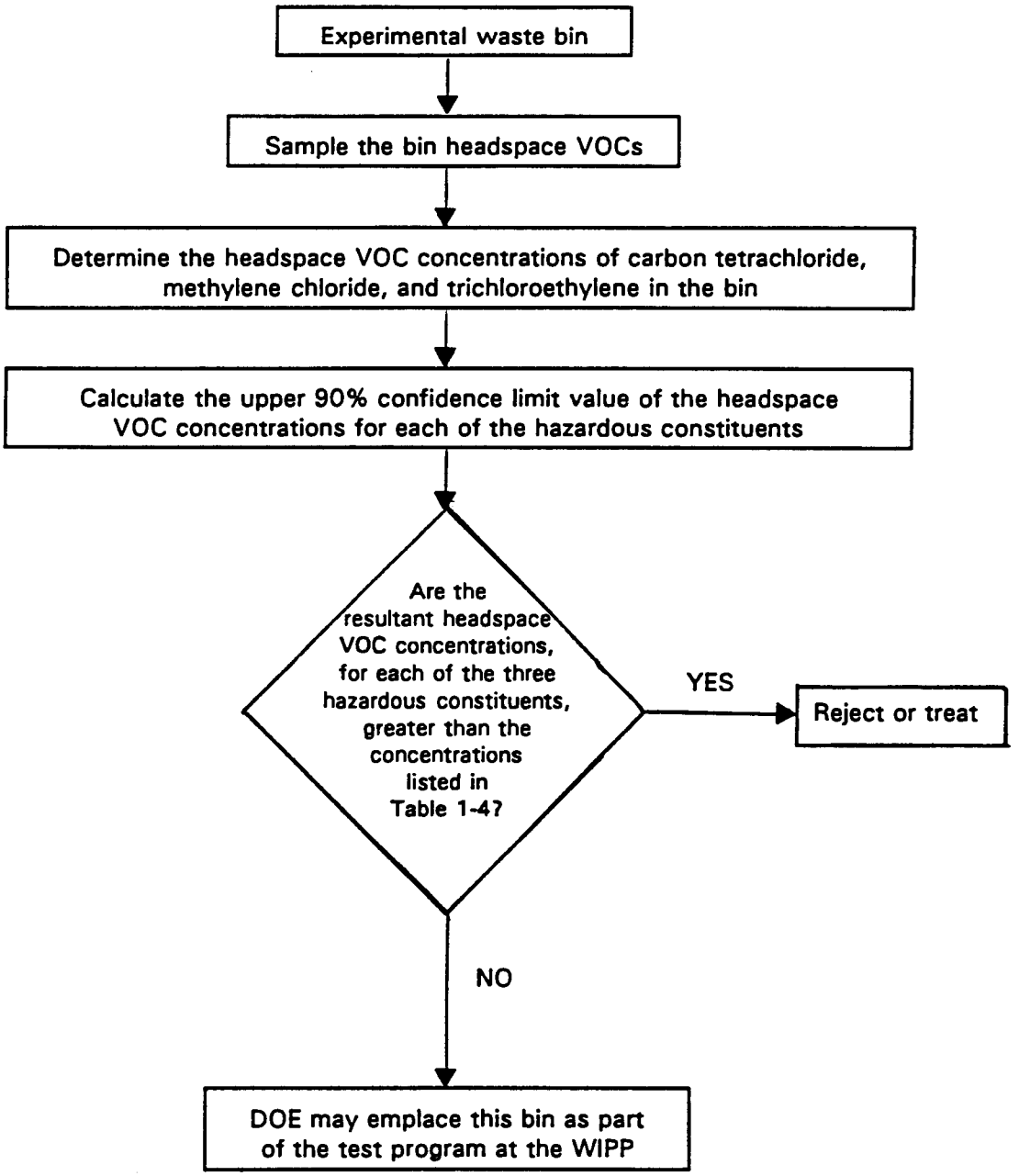


Figure 1-5. No-Migration Demonstration Flowchart

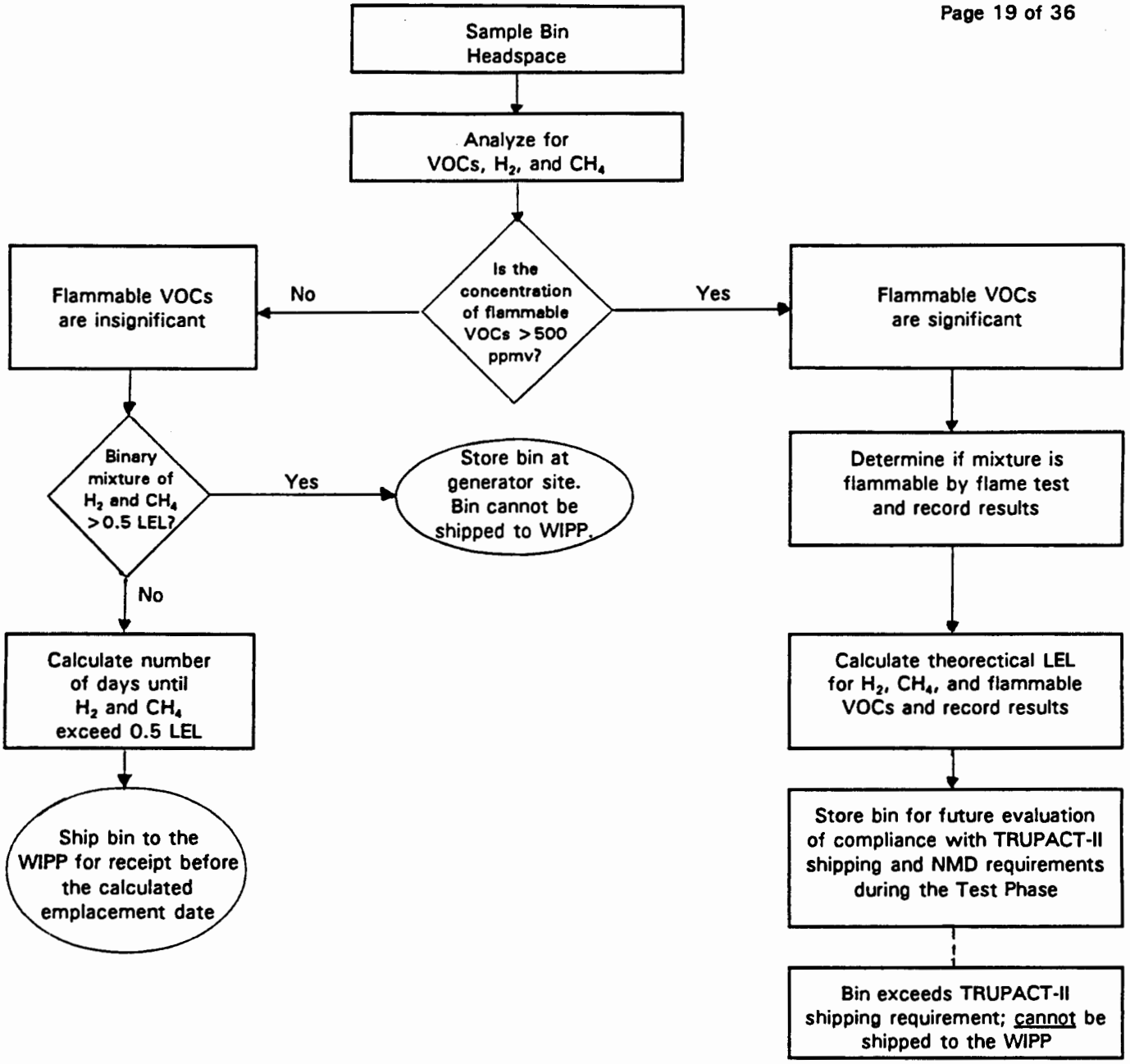


Figure 1-6. Compliance With Flammability Testing Requirements

If the headspace concentrations of flammable VOCs exceeds 500 ppmv, this bin currently may not be transported in the TRUPACT-II. Therefore, this bin must remain in storage at the generator/storage site until further assessment and resolution by DOE.

Le Chatelier Formula

For bins of waste that contain headspace concentrations of flammable VOCs less than 500 ppmv, the Le Chatelier formula (Coward and Jones, 1952) shall be used to determine if a headspace gas mixture exceeds 50% of the lower explosive limit (LEL) for binary mixtures of hydrogen and methane. The Le Chatelier formula is generated when the reciprocal of the LEL is substituted for the independent variable "a" in Equation 12-9 (Section 12.6). To perform the Le Chatelier determination:

1. Correct the measured analyte concentrations for recovery using Equation 12-10, Section 12.6.

2. Calculate analyte standard deviations using the measured analyte concentrations and analyte relative standard deviation values using Equation 12-11, Section 12.6.

3. Calculate the percentage of the theoretical LEL using the Le Chatelier formula expressed as Equation 12-9, Section 12.6, using the upper 90% confidence limit value of the H_2 and CH_4 concentrations. The mixture exceeds 50% of the LEL if the result of this computation exceeds 50%.

Emplacement Time Determination

The length of time during waste handling activities in which the mixture of H_2 and CH_4 will remain below 50% of the theoretical LEL shall be determined, and the bin shall be emplaced prior to exceeding that time limit. During the course of the bin-scale tests, theoretically determined hydrogen concentrations (Table 12-2) shall be verified by sampling the headspace gases.

The procedure is summarized as follows:

- An initial value for bin headspace hydrogen and methane concentrations is obtained.

- Based on the initial hydrogen and methane concentrations, the number of days remaining until 50% of the LEL is exceeded is determined from Tables 12-3 to 12-7 (Section 12.6). The times reported in Tables 12-3 to 12-7 include an allowance for a three-day shipping period.

- If the bin is not sampled, Table 12-2 (Section 12.6) shall be used to determine the remaining emplacement time, given knowledge of the time since the bin was loaded.

Bin sampling and analysis allows for maximization of the amount of time prior to emplacement.

1.3.3 General RCRA Waste Analysis Requirements

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. To verify waste characterization information that is based on knowledge of materials and processes generating TRU mixed waste to be emplaced in the WIPP during the Test Phase, site-specific waste profile plans for TRU waste will be prepared by each generator/storage facility and will be subject to approval by DOE/WPO (USDOE, 1991b). Waste profile plans will provide detailed information on waste characterization activities to be performed for specific categories of wastes. In addition, data obtained pursuant to the methods described in the waste category specific waste profile plans will be reported in a waste profile form that will be sent to DOE/WPO prior to shipment of the waste to the WIPP. 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 waste profile plan, that identifies the specific wastes and the number of containers to 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 WIPP test 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:

Radioassay (RA)

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.

Real-time Radiography (RTR) and Visual Examination

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.

Gas Sampling and Analyses

Determine the concentrations of gases (Table 3-6) 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.

Data on the concentrations of hydrogen and methane also will be used to demonstrate that binary mixtures of these gases do not exceed 50% of the Le Chatelier theoretical lower explosive limit (LEL) in waste containers and bins to be emplaced in the WIPP. In addition, based on the data collected or the time since bin loading (i.e., the waste has been loaded in the bin and the bin closed and placed in the standard waste box) at the generator/storage site, the number of days remaining until 50% of the LEL is exceeded will be determined (Section 12.6) and documented.

Volatile Organic Compound (VOC) Sampling and Analyses

Determine the concentrations of VOCs in the headspace of waste containers and bins at the DOE generator/storage facilities. These data shall be used to demonstrate compliance with the conditions of the no-migration determination requirements associated with waste characterization.

The specific DQOs for demonstrating compliance with the waste characterization requirements in the NMD are as follows:

Comparability - To demonstrate that the upper 90% confidence limit values of the measured headspace concentrations of volatile organic compounds listed in Table 1-3 do not exceed two times the maximum concentrations reported in DOE's Petition (USDOE, 1990b).

No-Migration Demonstration - To demonstrate that the upper 90% confidence limit values of the measured headspace concentrations of VOCs listed in Table 1-4 do not exceed ten times the mean concentrations reported in DOE's Petition.

Flammability - To demonstrate that (1) the upper 90% confidence limit values of the measured headspace concentrations of flammable VOCs listed in Table 1-5 do not exceed 500 ppmv, and (2) if

a mixture of flammable VOCs exceeds 500 ppmv, then to demonstrate, using an explicit "flame test," that the mixture is nonflammable.

Because wastes that contain headspace concentrations of flammable VOCs that exceed 500 ppmv currently may not be shipped in the TRUPACT-II, they will not be included as part of the WIPP test program unless DOE obtains an amendment to the TRUPACT-II Safety Analysis Report from the Nuclear Regulatory Commission to transport these wastes. Headspace concentrations of flammable VOCs less than 500 ppmv are considered by EPA to not significantly contribute to the potential flammability of the waste, therefore, as described under headspace sampling and analysis DQOs, the calculated theoretical LEL will be determined based on a binary mixture of hydrogen and methane.

RCRA-Regulated Hazardous Waste Characterization

Verify existing waste characterization information that is based on knowledge of the materials and processes that generate the waste using the information obtained from the Program.

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 1-6 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 all quality documents for the Program shall be accomplished 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) 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, the WIPP Waste Acceptance Criteria Certification Committee (WACCC) Chairperson, 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

Table 1-6. 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	Site Project Manager	Site Project Quality Assurance Officer
QAPP								
Review		X		X	X	X		
Review/Approval	X		X					
Implementation[a]				X				
Change Approval	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.

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 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 left-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. No changes that can affect the performance

criteria or data quality shall be made without prior approval of the DOE Operations Office, 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.

Each DOE generator/storage site shall maintain in its project files the original or a copy, as appropriate, of all QA/QC waste characterization data for waste that is sent to the WIPP. In addition, all QA/QC records associated with TRU wastes that are characterized but not sent to the WIPP because of noncompliance with the current programmatic or regulatory waste characterization requirements shall be maintained, for future evaluation, at the DOE generator/storage sites in accordance with the recording keeping requirements specified in this section.

The data/records management systems shall provide adequate control and retention for all the Program-related information. All waste characterization data and related QA/QC records that are to be transferred to DOE/WPO 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," (USDOE, 1991c), 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 the 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 an original hard copy 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 specified by DOE/WPO. 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 1-7 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 Investigation Center (NEIC) guidelines (USEPA, 1986a).

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. The site Project Manager is responsible for verifying supplier compliance with the applicable QA/QC requirements.

Table 1-7. 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.

1.7.2 Control of Subcontractors

Performance requirements must be communicated to all subcontractors involved in the Program. Performance requirements and compliance with this QAPP shall be part of subcontractor agreements. DOE operating contractors shall perform and document the results of 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 the 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 Sections 10.0 and 13.0.

All subcontractors providing analytical services in support of the Program must participate in the Performance Demonstration Program (PDP) as described in Section 1.11. Each subcontractor for analytical services shall demonstrate their ability to meet the QA objectives for the Program by successfully completing the requirements of the PDP prior to sample analysis. DOE/WPO shall review and approve the results of the PDP prior to the initiation of work by the subcontractor for analytical services. DOE/WPO, as the administrator of the PDP, 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. 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. 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 identification and control 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 identification and traceability of items may include item identification from initial receipt up to and including installation and use, physical identification, clear and legible marking, or a combination thereof. Items having limited calendar or operating life shall be identified and controlled to preclude use of items whose shelf life or operating life has expired.

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 approved 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 and documented at periodic intervals not to exceed three years.

Training may not be waived for visual examination of waste (Section 7.3). 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 1-8. 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.

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

Table 1-8. Minimum Training and Qualifications Requirements

Personnel	Requirements
Gas Chromatograph Analysts [a] 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 1 year independent spectral interpretation or demonstrated expertise
Gas Mass Spectrometer Analysts [a] Gas Chromatograph-Mass Spectrometer Analysts [a]	B.S. or equivalent experience and 1 year applicable experience
UV-VIS Spectrophotometer Analysts [a]	3 months applicable experience
Flammability Apparatus Analysts [a]/Interpreter	3 months applicable experience

[a] Analysts are those persons responsible for the overall technical operation and development of a specific laboratory technique.

- 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 WIPP 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. 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 Plan

Each analytical laboratory participating in the Program must have in place a documented laboratory QA plan 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 PDP and demonstrate conformance to the QA objectives for the Program. Each laboratory, through participation in the PDP, will demonstrate and document its performance characteristics. The PDP Plan (USDOE, 1991a) includes a detailed description of the requirements of the PDP.

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, 1986) 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 the 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.

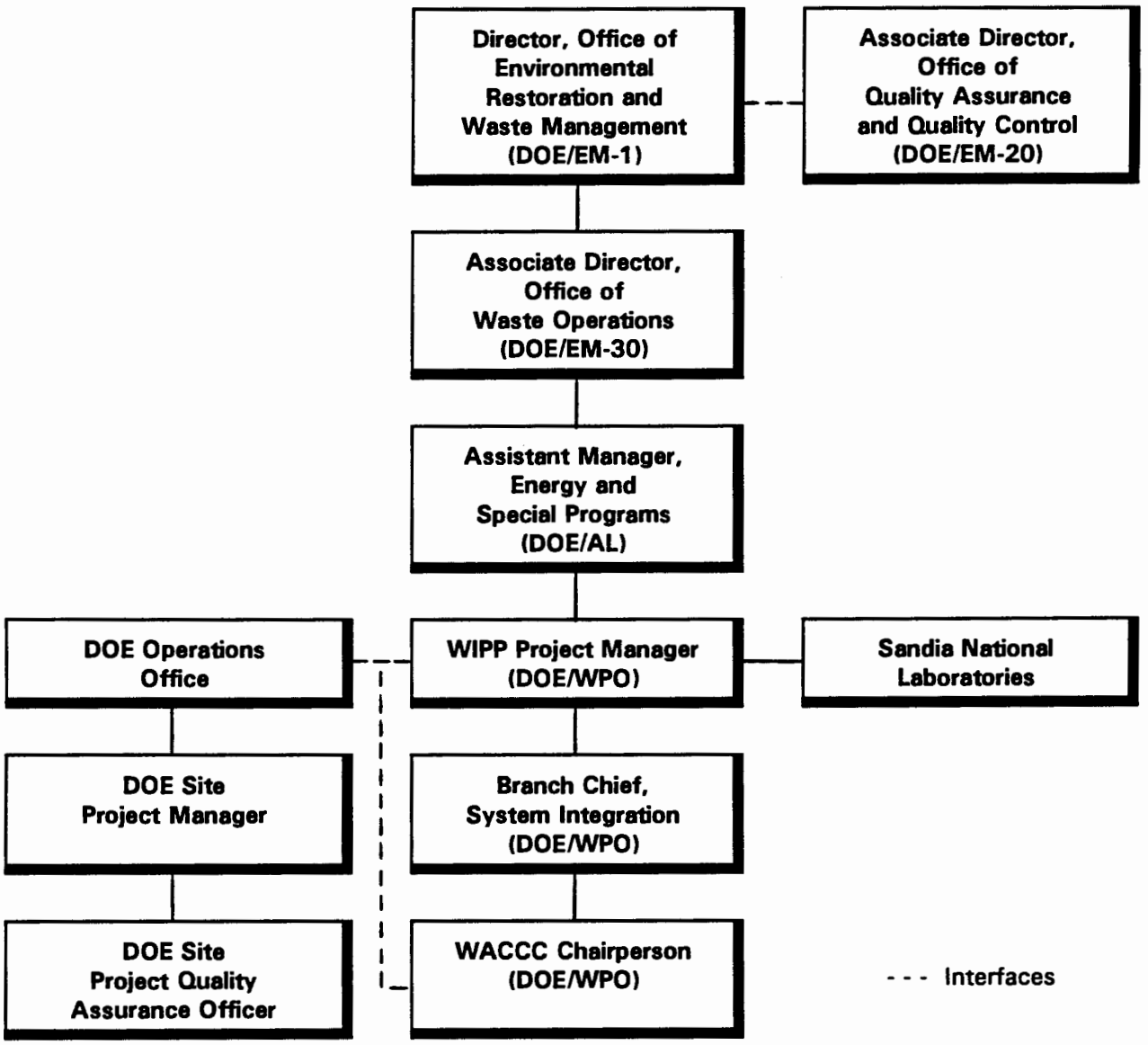


Figure 2-1. QAPP Functional Organization Chart

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

The Associate 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 Associate Director provides interpretations of requirements and interfaces and coordinates with other federal agencies for programs and activities managed by EM.

The Associate Director develops and oversees the implementation of the EM QA Program based on DOE Orders, national standards, EPA requirements, and EM program requirements. The Associate Director shall provide an independent oversight function regarding the requirements of this QAPP and shall review this QAPP for consistency with all applicable regulations and requirements. The Associate Director may participate in selected field organization audits/appraisals of contractor facilities/operations that may be scheduled at least annually by the field organizations or EM-30.

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 1-3). 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 PDP (Section 1.11). In this role, the WIPP Project Manager shall be responsible for coordinating implementation of the PDP 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 PDP, 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 the 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 test 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 QAPP 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 of the 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

As a part of the Program, each DOE Operations Office will review and approve the site QAPjPs for the facility under that office's responsibility. The Operations Offices 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 Offices prior to implementation of the change. The DOE Operations Offices 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 1-3). 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 the 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 1-3). 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 designed to obtain data to support the data quality objectives, as described in Section 1.4.

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. Six waste characterization techniques must be used:

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

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 and semi-quantitative 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.

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
Flammability testing	NR	NR	NR	YES	YES

NR = characterization not required to meet objective.

The objectives of RTR for the Program are the following:

1. If applicable, verify waste Content Code (Table 3-2) and site-specific Item Description Code (IDC), as defined in Section 15.0.

2. Where applicable, provide an estimated inventory of the waste items, residual materials, and packaging materials and/or waste material categories.

3. If present, estimate the quantity of free liquids.

4. Provide the information necessary for making an estimate of waste items' residual and packaging materials weight for homogeneous 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 WIPP bin-scale and alcove test phase (Molecke, 1990a, 1990b), 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, packaging materials, and residual materials to estimate weights of the waste material categories specified in Table 3-3. These data on standard weights should include weights of packaging and residual materials as well as the weights of standard waste items such as HEPA filters, Raschig rings, and Fulflo® filters. These data should also include information on the proportion of materials in composites such as leaded rubber, cemented process liquids, and sludges.

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.

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 estimated inventories of waste items and residual 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. The sites should document how accurately an operator can make these determinations. The precision and accuracy of weight data estimates shall be enhanced by utilizing standardized RTR procedures and operator qualifications.

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

*1XX is used to designate the Content Code of newly generated waste. 2XX is used to designate the Content Code of retrievably stored waste.

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

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

3.1.2 Completeness

An audio/videotape of the RTR examination and an RTR data form, validated according to the requirements in Section 8.0, must be obtained for 100% of the waste containers used in the 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 homogeneous waste for the WIPP bin-scale and alcove tests.

3.2 Radioassay

The following three RA 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 indicates 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 WIPP 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 (Molecke, 1990a; Molecke and Lappin, 1990).

Each generator/storage facility must use one or more RA techniques. Each facility shall demonstrate and technically justify that any RA techniques used are appropriate for their specific installation. The basis for using a specific assay technique should be the physical form of the waste, the radionuclide content, and the 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 TRU content, and its distribution.

The accuracy, precision, and completeness quality assurance objectives to be used for the three identified RA techniques are summarized in Table 3-4. These objectives apply to wastes with homogeneous distributions, low neutron absorber and moderator content, and little self-shielding of the fissile sources. They are expected to be achievable in the presence of backgrounds generated by

Table 3-4. Quality Assurance Objectives for Radioassays

	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

alpha and gamma emitting sources and in the presence of quantities of neutron and gamma absorbing and moderating material, as is the case for most waste encountered in this program. 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 a total error of ±0.5 g (95% confidence level) per waste container for waste containers containing approximately 1 g of weapons grade plutonium (Lappin et al., 1990).

3.2.1 Precision and Accuracy

The precision and accuracy of the measurement technique must be determined through replicate processing and comparison with calibration standards, 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

RA data shall be obtained for 100% of the waste containers used in the Program. RA data shall be validated according to the requirements in Section 8.0.

3.2.3 Comparability

If multiple systems are planned for use in this program, the DOE sites should perform multiple independent assays of a sample of waste containers. For example, these independent assays may include a passive gamma assay such as SGS followed by 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 TRU wastes to be emplaced in WIPP as part of the Test Phase are packaged in 55-gallon drums and can contain waste individually packaged in several layers of plastic confinement. Pretest waste characterization for the bin-scale tests will include sampling and analysis of headspace gas from three areas within drums of TRU waste (see Figure 3-1): (1) the drum headspace (i.e., the headspace directly under the drum lid), (2) the 55-gallon poly bag headspace, and (3) the innermost layers of confinement headspace. Drum headspace sampling will be included in Revision 2 of the QAPP. The results of analyses of headspace samples from the 55-gallon poly bag and innermost layers of confinement must be used to demonstrate compliance with the comparability requirements of the NMD. Bin headspace samples must also be obtained. The results of analyses of headspace samples from within experimental bins must be used to demonstrate compliance with the no-migration finding

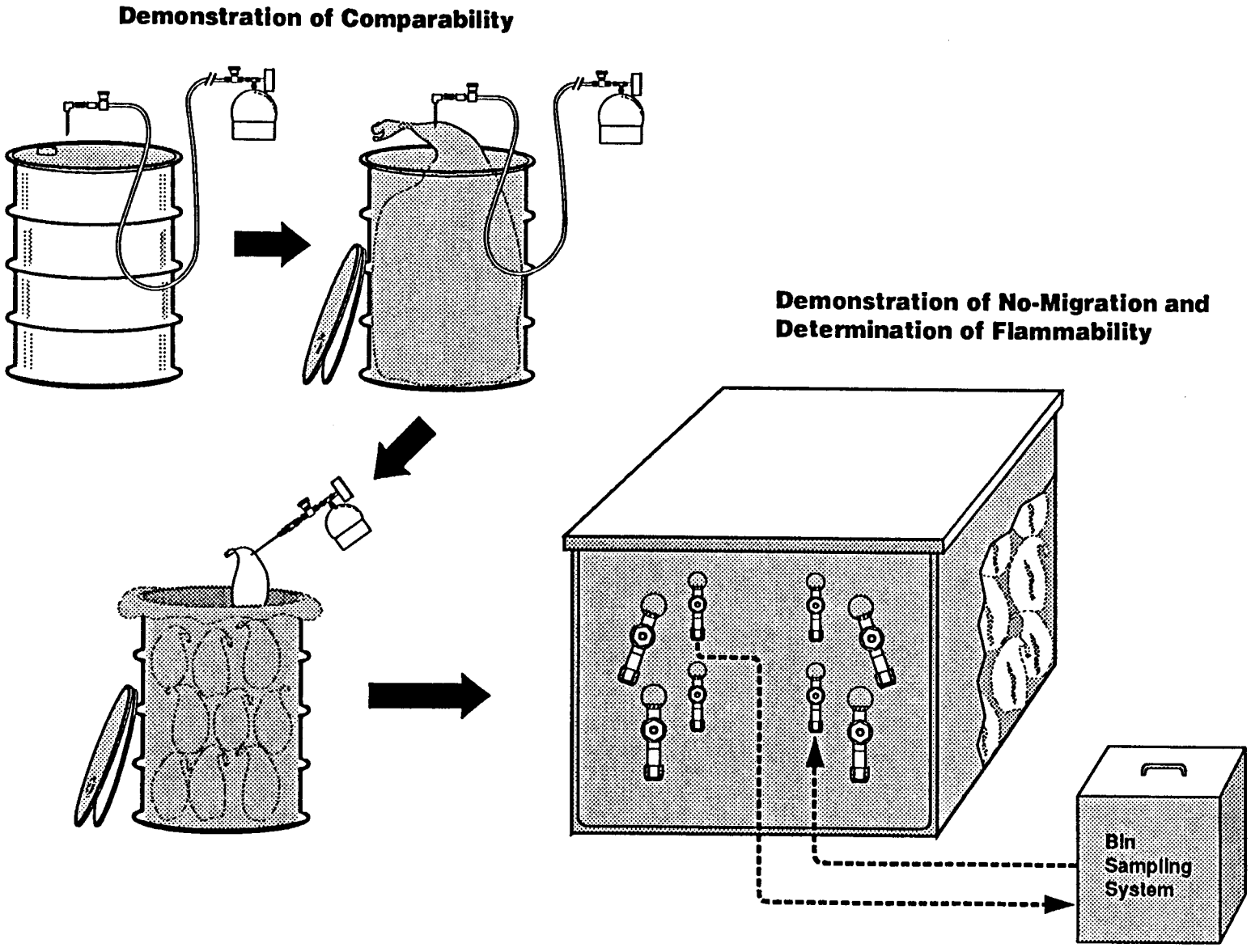


Figure 3-1. Overall Sampling Compliance Scheme

and flammability testing requirements of the NMD. The following sections detail quality assurance objectives for headspace sampling operations.

3.3.1 55-Gallon Poly Bag Headspace Sampling

The precision and accuracy of 55-gallon poly bag headspace sampling operations must be assessed by analyzing field QC headspace samples. These 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.

3.3.1.1 Precision

The precision of the 55-gallon poly bag headspace sampling and analysis 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 ±25%; (2) for gases, the RPD exceeds ±20% for concentrations greater than 5X the Program Required Detection Limit (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 greater than ±PRDL for concentrations less than 2.5X the PRDL.

3.3.1.2 Accuracy

A field reference standard must be collected using the headspace sampling manifold to assess the relative percent accuracy (RPA) of the 55-gallon poly bag headspace sampling and analysis 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.

3.3.1.3 Representativeness

Specific headspace sampling steps to ensure the representativeness of 55-gallon poly bag headspace samples include:

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

- Use of a low internal volume headspace sampling manifold
- Small sample volume: low sample volume to available headspace volume ratio
- Careful pressure regulation
- Performance audits
- Collection of headspace sampling manifold blanks, field reference standards, field blanks, and field duplicates.

3.3.1.4 Completeness

The amount and type of data that may be lost during the 55-gallon poly bag 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 sufficient headspace samples are collected to meet the Program objectives.

3.3.1.5 Comparability

Consistent use and application of uniform procedures and equipment (in accordance with USDOE, 1991d) should ensure that 55-gallon poly bag headspace sampling operations are comparable at the different headspace sampling sites.

3.3.2 Innermost Layer of Confinement Headspace Sampling

The field QC headspace samples collected during innermost layer of confinement headspace sampling operations must include equipment blanks, field reference standards, and field blanks. When sufficient available headspace exists, field duplicates must be collected in accordance with the frequency specified in Section 9.1.4.

3.3.2.1 Precision

The precision of the innermost layer of confinement headspace sampling and analysis operation must be assessed by collecting field duplicates for VOC and gas determination. Remedial action must be taken if (1) for VOCs, the RPD exceeds ±25%; (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.

3.3.2.2 Accuracy

A field reference standard must be collected using the innermost layer of confinement sampling equipment to assess the RPA of the sampling and analysis 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.

3.3.2.3 Representativeness

Specific innermost layer of confinement headspace sampling measures to ensure the representativeness of headspace samples include:

- Sample canister cleaning and leak check
- Sampling equipment (needle and filter) cleaning or disposal after use
- Use of sample canisters with passivated internal surfaces
- Use of low internal volume sampling equipment (needle and filter)
- Collection of a small sample volume: low sample volume to available headspace volume ratio
- Performance audits
- Collection of sampling equipment blanks, field reference standards, and field blanks
- Needle and filter should be purged with zero air or helium and capped for storage prior to use to prevent sample contamination.

3.3.2.4 Completeness

One hundred percent of the innermost layers of confinement containing a minimum of 1 liter of available headspace must be sampled. This volume is dictated by the analytical sample size requirement of 100 ml and the objective of not removing more than 10% of the available headspace. The headspace within rigid innermost layers of confinement (e.g., glass, metal, and rigid plastic containers) will not be sampled, but a headspace sample will be collected from the closest non-rigid layer of confinement.

3.3.2.5 Comparability

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

3.3.3 Bin Headspace Sampling

The field QC samples collected during bin headspace sampling operations must include equipment blanks, reference standards, and field duplicates. Field blanks are not required during bin sampling operations since the bin sampling equipment is carefully sealed to the bin prior to sample collection, thus preventing ambient air intrusion.

3.3.3.1 Precision

The precision of the bin headspace sampling and analysis operation must be assessed by collecting field duplicates for VOC and gas determinations. Remedial action must be taken if (1) for VOCs, the RPD exceeds ±25%; (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.

3.3.3.2 Accuracy

A reference standard must be collected in the laboratory or field prior to bin headspace sample collection, through the bin sampling equipment to assess the RPA of the sampling and analysis 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.

3.3.3.3 Representativeness

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

- Recirculation of the headspace gases to achieve homogeneity prior to sampling
- Canister cleaning and leak check
- Equipment cleaning and leak check
- Use of sample canisters with passivated internal surfaces
- Collection of small volume samples: low sample volume to available headspace volume ratio
- Careful pressure regulation and flow control
- Performance audits
- Collection of bin headspace sampling equipment blanks, reference standards, and field duplicates.

3.3.3.4 Completeness

A bin headspace sample must be obtained from 100% of the bins to be used in the WIPP bin-scale test.

3.3.3.5 Comparability

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

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 (Lappin et al., 1991). 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, determined during the visual examination, will be used as input to the gas generation behavior model.

3.4.1 Precision and Accuracy

The precision and accuracy of weight estimates obtained from visual examination are unknown at the present time. However, the quality of the visual examination data shall be enhanced by the use of standardized procedures and operator qualifications. 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. The precision and accuracy with which weight estimates can be made must be evaluated and reported.

3.4.2 Completeness

An audio/videotape of the visual examination and a visual examination data form, validated according to the requirements in Section 8.0, must be obtained for 100% of the waste containers used in the WIPP bin-scale test and for a representative portion (to be determined by Sandia National Laboratories) of the waste containers used during the WIPP alcove test.

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 DQOs specifically for this program have resulted in the data quality assurance objectives listed in Tables 3-5 and 3-6. The specified quality assurance objectives represent the required quality of data necessary to draw valid conclusions regarding the objectives of this program. Program required limits, such as the PRDL associated with gas analysis and the Program Required Quantitation Limit (PRQL) associated with VOC analysis, are specified by the Program to ensure that the analytical data collected satisfy the requirements of all data users. 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 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 and field 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 3-5 and 3-6. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

3.5.2 Accuracy

Accuracy is the degree of agreement between a measured analyte concentration (or the average of replicate measurements of a single analyte concentration) and the true or known 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 3-5 and 3-6. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Table 3-5. VOC Quality Assurance Objectives

Compound	Precision [a] (%RSD or RPD)	Accuracy [a] (%R)	MDL (ng)	Completeness (%)
Benzene Bromoform 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, total number of nanograms delivered to the analytical system per sample)

[a] Criteria apply to concentrations listed in Table 3-7
[b] Estimate, to be determined

Table 3-6. Gas Analysis Quality Assurance Objectives

Analyte Gases	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[a] (combined)		±20%	50-150%	0.01	90%
a. Nitric Oxide	10102-43-9				
b. Nitrogen Dioxide	10102-44-0				

a = Analyses of Nitrogen Oxides are not required for innermost layers of confinement.
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
PRDL = Program required detection limit

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 MDL is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL will be expressed in nanograms for VOCs and volume percent for gases. The MDLs will be determined (Glaser, 1981) by replicate measurements of samples containing the analytes listed in Tables 3-6 and 3-7. Laboratory determined MDLs must be less than or equal to those listed in Table 3-5 and the PRDLs listed in Table 3-6. 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 the collection of sufficient numbers of samples using a clean sampling manifold that does not introduce sample bias.

3.6 Flammability Testing

The quality assurance objectives for flammability testing are summarized in Table 3-8. Flammability testing uses a modified version of ASTM Method E 681-85 as a flammable/nonflammable test to evaluate the flammability of bin headspace samples.

3.6.1 Precision and Accuracy

Flammability test results are considered qualitative. Since qualitative evaluations do not lend themselves to statistical analysis, precision will be enhanced by requiring that sites use a standardized flame test apparatus and a standardized analytical method in conjunction with operator training.

Table 3-7. VOC Headspace Target Compound List (TCL) and Program Required Quantitation Limit (PRQL)

Volatiles	CAS Number	PRQL [a] (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 [b]	108-38-3	1
28. o-Xylene	95-47-6	1
29. p-Xylene [b]	106-42-3	1

[a] Values based on delivering 10 mL to the analytical system.
[b] These xylene isomers cannot be resolved by the analytical methods employed in this program.

Table 3-8. Flammability Testing Quality Assurance Objectives

Evaluation	Accuracy[a] (%)	Completeness[b] (%)
Flammability	100	100

[a] Accuracy expressed as,
(Number of test mixtures whose flammability is correctly evaluated/Total number of test mixtures evaluated by flammability testing specialist)*100

[b] Completeness expressed as,
(Number of bin headspace samples evaluated with valid data/Number of bin headspace samples requiring flammability testing)*100

Duplicate samples will be collected for flame testing when sufficient bin headspace is available. The accuracy of each laboratory's flame test apparatus shall be assessed and qualified prior to the evaluation of samples and annually thereafter. A flame test apparatus is qualified when it successfully evaluates the flammability of each of the test mixtures (Table 6-3). Each flame test apparatus shall have an accuracy of 100% (Table 3-8).

3.6.2 Completeness

The measure of completeness for flammability testing shall be 100% (Table 3-8). Samples from all bins with headspace containing a summed concentration of flammable VOCs that cannot be shown to be below 500 ppmv shall be analyzed with valid results using the flame test apparatus.

3.6.3 Comparability

Comparability between sites shall be achieved by using a standardized flame test apparatus and a standardized analytical method. On a per sample basis, flame test results should be comparable to a theoretical flammability evaluation made using the Le Chatelier formula (Section 12.6). The procedure for performing a theoretical flammability evaluation is presented in Section 12-6 and detailed in ASTM E 681-85. Sites must provide this comparability data to DOE/WPO, even though the flame test is currently the only acceptable method of evaluating the flammability of bin headspace samples. DOE is collecting these data so that in the future, headspace flammability can be evaluated with a theoretical calculation.

3.6.4 Representativeness

The quality characteristic of representativeness as applied to flammability testing shall be achieved by the use of clean headspace sampling equipment that does not introduce sample bias and by proper sampling techniques.

4.0 SAMPLING PROCEDURES

The headspace sampling requirements described in this section must be followed to collect representative headspace samples from within TRU waste containers and bins. The headspace composition may indicate the production of headspace gases and VOCs 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 3-6, and "VOCs" is used to represent the analytes listed in Table 3-7 or a Program required subset of the listed analytes. "Headspace" should be interpreted to mean the VOCs and gas within a layer of confinement. "Protocol" material constitutes the absolute minimum requirements for compliance with this program. "Guidance" material consists of recommended practices. "Procedures" consist 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 55-Gallon Poly Bag Headspace Sampling Protocol

This section describes requirements for the collection of headspace samples from the 55-gallon poly bag within a TRU waste drum. 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 3-6 and 3-7 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.

Before collecting the first headspace sample, the headspace sampling manifold must be used to collect a sampling manifold blank and a field reference standard. The concentrations of analytes in these QC

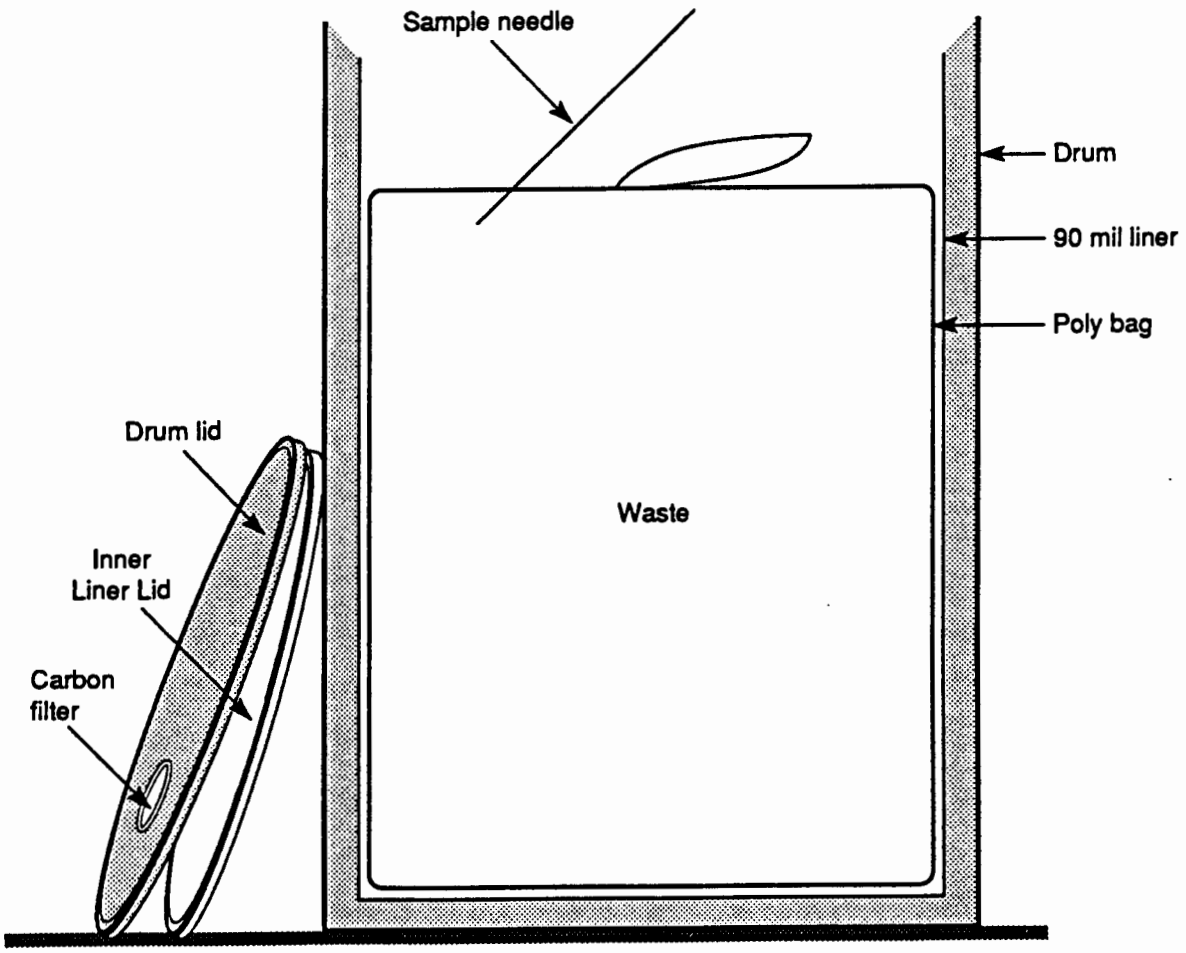


Figure 4-1. Schematic Diagram of Waste Drum with Minimum Layers of Containment and Sampling Location (Not to Scale)

samples must be determined in order to evaluate whether the headspace sampling manifold can meet the quality assurance objectives specified in Section 3.3.

4.1.1 Scope and Application

This protocol for sampling of the 55-gallon poly bag headspace within TRU 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 in TRU waste drums containing different waste types. 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. Drums and their contents 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 drums 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 a representative sample is withdrawn from within the poly bag. When an estimate of the available headspace volume can be made, less than 10% of that volume should be withdrawn. An assessment of the void volume can be made from knowledge of the Content Code. A determination of the headspace sampling manifold internal volume must be made and documented. The total volume of headspace gases collected during each headspace sampling operation can be determined by adding the combined volume of the sample canisters attached to the headspace sampling manifold to the internal volume of the headspace sampling manifold. Site SOPs must address documenting any non-routine events or occurrences that may affect the quality of the headspace sample collected.

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.

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

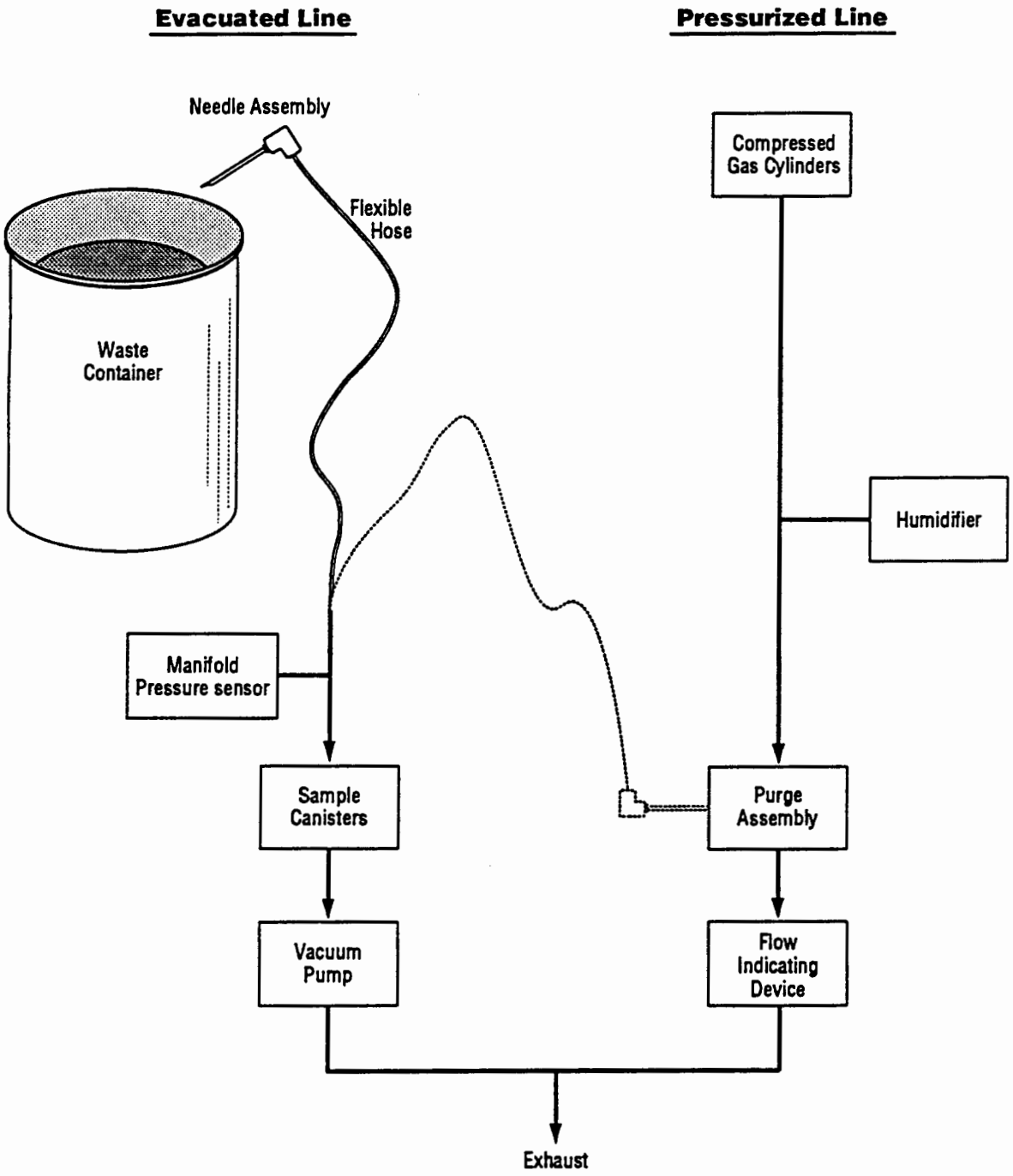


Figure 4-3. Headspace Sampling Manifold Schematic

- Headspace sampling manifold pressure sensor(s) that must be pneumatically connected to the headspace sampling manifold. The headspace sampling manifold pressure sensor(s) must be able to measure absolute pressure in the range from 0.05 mm Hg to 1000 mm Hg. Its resolution must be ±0.005 mm Hg at 0.05 mm of Hg. The headspace sampling manifold pressure sensor(s) must have an operating range from approximately 15°C to 40°C.

- 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.

In addition to the above required components, sites may choose to outfit their headspace sampling manifold with an organic vapor analyzer which includes a PID capable of detecting all of the VOCs, listed in Table 3-7.

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 at least 500 to 800 mm Hg and must be kept in the sampling area during sampling operations. Its resolution must be 1.5 mm Hg and calibration shall be based on NIST standards. A temperature sensor should be near the ambient pressure sensor 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. Based on data obtained on ambient air samples and mixed gas standards, it is anticipated that headspace samples will be stable longer than the specified holding times. The programmatic 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. NO_x and gas samples may be kept 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.

Table 4-1. Headspace Sample Containers and Holding Times

Parameter	Container	Headspace Sample Minimum Volume	Holding Temperatures	Field Holding Time[a]	Laboratory Holding Time[b]
Gases	SUMMA® Canister	100 milliliters	Ambient	4 days	28 days
VOCs	SUMMA® Canister	250 milliliters[c]	0-40°C	4 days	28 days
NO_x[d]	SUMMA® Canister	100 milliliters	Ambient	4 days	28 days

[a] From time of headspace sample collection to shipment.
[b] This is a programmatic-based maximum holding time. Holding time begins at VTSR.
[c] Alternatively, if available headspace is limited, 100 ml samples may be collected for determination of volatiles.
[d] A separate headspace sample will not be required if mass spectrometry is used for inorganic gas analysis.

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. All sample canisters should have VCR® fittings for connection to sampling and analytical equipment. 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). If the available headspace is limited, 100 mL samples may be collected for the determination of VOCs.

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

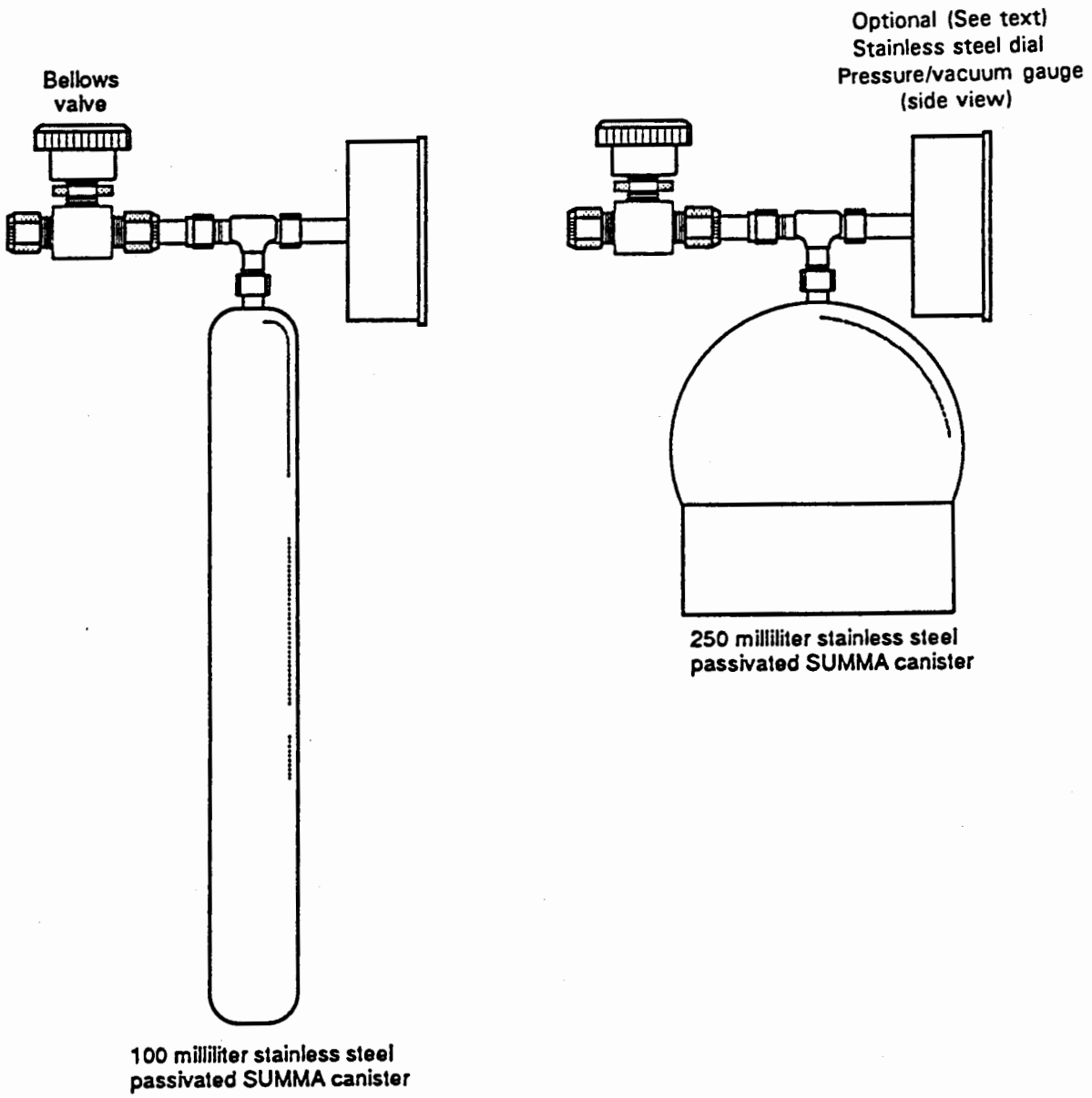


Figure 4-4. SUMMA® Canister Components Configuration
(Not to Scale)

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 for 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 3-7, 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.

4.4 Innermost Layer of Confinement Headspace Sampling Protocol

This section describes requirements for the collection of headspace samples from innermost layers of confinement within a TRU waste drum. An innermost layer of confinement is defined as the poly bag layer closest to the waste. This protocol is designed to ensure that representative headspace samples, including quality control samples, are consistently collected. The innermost layer of confinement samples must include:

- Headspace sample from within innermost layers of confinement that meet the minimum criteria discussed in Section 4.4.1.
- Field duplicates
- Field blanks
- Sampling equipment blanks
- Field reference standards.

4.4.1 Scope and Application

The TRU wastes to be sent to the WIPP as part of the Test Phase are packaged in 55-gallon drums and can contain waste items, residual materials, and/or packaging materials confined by several layers of plastic bags. Given this waste packaging heterogeneity, the EPA is requiring that the headspace sample which the DOE collects from within 55-gallon drums be representative of the entire headspace of the drum. EPA expects that all innermost layers of confinement in a drum will have to be sampled until DOE can demonstrate to the EPA, based on the data collected, that sampling of all innermost layers is either unnecessary or can be safely reduced.

DOE will address EPA's requirement that headspace sampling of waste drums be representative of the entire headspace within a drum by sampling all of the innermost layers of confinement that meet the following minimum criteria:

- The innermost layer of confinement must have a minimum of 1 liter of headspace from which a representative 100 ml sample must be withdrawn. A 250 ml sample should be collected if there is enough available headspace gas (2.5 liter, minimum). When duplicates are collected, twice the available headspace shall be present.
- Headspace contained within rigid innermost layers of confinement (e.g., glass, metal, and rigid plastic containers) will not be sampled, but the poly bag closest to the rigid container must be sampled if a minimum of 1 liter of headspace is available.

4.4.2 Method Summary

The innermost layer of confinement sampling protocol described here employs either a low internal volume sampling manifold or a direct canister sampling system to collect headspace samples from within innermost layers of confinement within TRU waste drums. Sampling equipment must be leak checked and cleaned prior to use.

Headspace samples must be collected from within innermost layers of confinement which meet the criteria specified in Section 4.4.1. The sample canisters must be evacuated to 0.05 mm Hg prior to sample collection.

The sampling operator must determine and document, in accordance with site SOPs, which innermost layers of confinement are eligible for headspace sampling. This documentation must include the innermost layer of confinement identification number and estimated available headspace volume (less than 1 liter, greater than 1 liter, or greater than 2.5 liters). If the estimated available headspace volume is less than 1 liter and therefore ineligible for headspace sample collection, then site SOPs must address documenting this information and any other non-routine events or occurrences that may affect the quality of the headspace sample collected.

The operator must determine whether to use a 100 ml or a 250 ml sample canister. If the innermost layer of confinement to be sampled contains at least 1 liter but not more than 2.5 liters of available headspace, then a 100 ml sample canister must be used. If the innermost layer of confinement to be sampled contains greater than 2.5 liters of available headspace, then a 250 ml sample canister should be used. In all cases, the maximum allowable sample volume should be collected. Sample holding times and storage conditions must conform to the requirements specified in Table 4-1.

Once the appropriate sample canister size has been determined, the operator must locate an area within the innermost layer of confinement from which headspace sample can be withdrawn. Care should be taken to prevent placing the tip of the clean needle in an area where solid material or particulate matter might fill or clog the needle.

The operator must press the needle through the innermost layer of confinement and into the headspace. Holding the needle in the innermost layer of confinement headspace, the operator must then open the inlet valve on the low internal volume manifold or the sample canister to allow headspace to expand into the evacuated sample canister. The pressure vacuum gauge on the low internal volume manifold or the sample canister will indicate when the canister pressure equilibrates

with ambient pressure, indicating that the canister has filled. Once the canister has filled, the operator must close the inlet valve and prepare the sample canister for shipment.

4.4.3 Sampling Apparatus

To accomplish innermost layer of confinement headspace sampling, two SUMMA® canister-based headspace sampling systems have been developed. The first of these, a low internal volume headspace sampling manifold, is similar to the headspace sampling manifold described for 55-gallon poly bag sampling in Section 4.1. The second, a direct canister headspace sampling system, employs a needle and filter attached directly to the SUMMA® sample canister. Site QAPjPs must specify which apparatus is used.

One advantage of the low internal volume headspace sampling manifold is that it can be evacuated from the tip of the sampling needle to the sample canister, thus minimizing the possibility of contamination by ambient gases trapped in the needle and filter. A disadvantage, however, is that even the lowest internal volume headspace sampling manifold will withdraw more headspace from within an innermost layer of confinement than will be withdrawn using the direct canister sampling system.

4.4.3.1 Low Internal Volume Headspace Sampling Manifold

The equipment and operational requirements for low volume manifold sampling are the same as those for 55-gallon poly bag sampling as described in Sections 4.1 to 4.3. However, modifications to the 55-gallon poly bag sampling equipment design as described in the Guidance Manual are necessary to reduce its overall internal volume. These modifications include reducing the length and possibly the inner diameter of most pneumatic lines and fittings as well as reducing the number of canister ports from six to one.

4.4.3.2 Direct Canister

Figure 4-5 illustrates the direct canister sampling equipment for innermost bag sampling. Figure 4-5 shows a stainless steel needle fitted into a reducer, a 0.5 μm filter, and a port connector that allows the filter to be connected to the 1/4-inch port of the SUMMA® canister's valve. Both the port connector and the reducer are made of stainless steel and have internal volumes of approximately 0.5 ml.

The needle, used to puncture the innermost layer of confinement during headspace sampling, is 1/8-inch stainless steel tubing, approximately 5 inches long and sharpened at the end. It has an inner

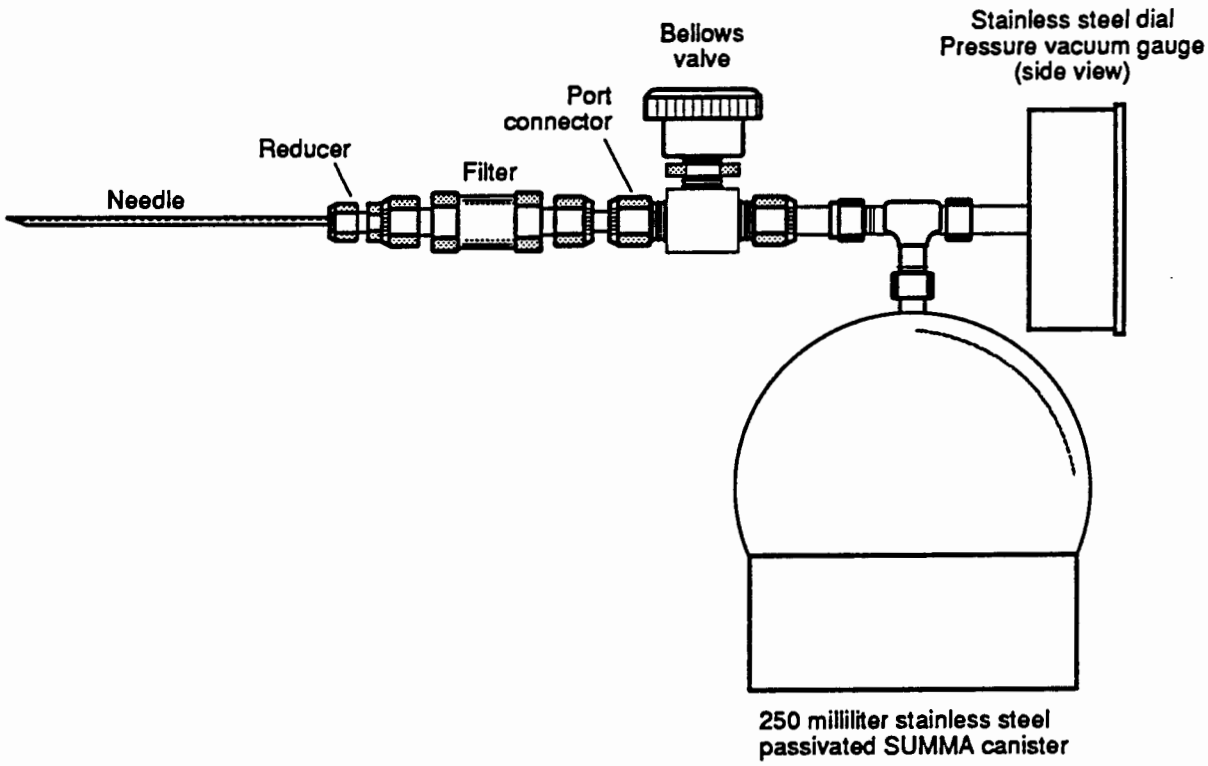


Figure 4-5. Schematic Diagram of Direct Canister Sampling Equipment

diameter of approximately 0.1 inch with an estimated internal volume of approximately 0.2 ml. The filter is a particulate filter containing a 0.5 μm sintered stainless steel fritt and has 1/4-inch fittings on both its inlet and outlet. The filter housing is made of stainless steel and is estimated to have an internal volume of approximately 4 ml. The combined internal volume of these components is approximately 5 ml. This low internal volume, approximately 2 percent of the sample volume, should maximize the representativeness of the headspace sample being collected.

4.4.4 Quality Control Measures

To prevent cross contamination, the needle, adapters and filter of the direct canister apparatus must be disposed of or, if reused, cleaned between sample collections. As a further QC measure the needle and filter, after cleaning, should be purged with zero air, nitrogen, or helium and capped for storage to prevent sample contamination by VOCs present in the ambient air. Field duplicates must be collected to assess sampling and analytical precision. Field blanks must be collected in the area where the innermost layer of confinement sampling is conducted. Equipment blanks and field reference standards may be collected, when using the direct canister sampling system, by adapting the purge assembly on the 55-gallon poly bag sampling manifold to accommodate the 1/8-inch needle of the direct canister sampling system. If the needle assemblies are the same as for the headspace sampling manifold, then the equipment blanks collected using the manifold are a sufficient quality control measure.

The QA/QC requirements for the low internal volume headspace sampling manifold are the same as those for the 55-gallon bag sampling in Sections 4.1 and 4.2.

4.5 Bin Headspace Sampling Protocol

This section describes the requirements for the collection of headspace samples from a bin containing TRU wastes. This protocol is designed to ensure that representative headspace samples, including quality control samples, are consistently collected. The bin samples must include the following:

- Headspace samples from within the bin
- Field Duplicates
- Sampling Equipment Blanks
- Field Reference Standards.

4.5.1 Scope and Application

During the WIPP Test Phase, DOE Plans to conduct bin-scale experiments in the WIPP to assess the rates and potential for gas generation that may influence the long-term integrity of the repository. At the generator/storage facilities, four to six drums of TRU wastes will be emptied into each test bin and

placed in a WIPP standard waste box (SWB). The experimental bin must be sampled at the generator/storage sites to demonstrate compliance with conditions specified in the NMD as described in Section 1.3. Bin sampling for assessment of compliance with the NMD requirements shall not be done until all drum data has been validated and approved by DOE/WPO (Section 8). This sample must be collected within 20 working days prior to TRUPACT-II loading.

4.5.2 Method Summary

The bin sampling system must be used to recirculate and homogenize bin headspace gas at a preset flow rate for a predetermined length of time, thereby making the collection of a representative headspace sample possible. The flow rate and time will be a function of waste type and packaging configuration as prescribed by Sandia National Laboratories.

Six liter bin headspace sample(s) must be collected in evacuated (0.05 mmHg) SUMMA® passivated canister(s) after the recirculation time has elapsed. The bin sampling system must also pressurize the sample canister(s) to a final pressure of approximately 15 psig. Sample holding times and storage conditions must conform to the requirements specified in Table 4-1.

For field duplicate sample collection, a total volume of 24 liters is withdrawn from the bin into two 6 liter SUMMA® canisters by pressurizing the canisters to 15 psig. During recirculation and duplicate sample collection, redundant pressure/vacuum relief controls must be provided to ensure that the internal bin pressure does not exceed 0.9 psig or fall below minus 0.8 psig.

4.5.3 Sampling Apparatus

The bin headspace sampling equipment, shown in Figure 4-6, must consist of a timer, ambient pressure and temperature sensors (not shown), a pump, flow controlling device(s) (if needed), a flow indicating device, valves, pneumatic recirculation loop, sample canister(s), and pressure regulating equipment. These components must meet the specifications described as follows:

- Flow indicating device(s) connected on the inlet side of the sample canister(s) must be capable of monitoring the flow rate of gases through the recirculation loop.
- Pneumatic recirculation loop and valves must be constructed of relatively inert materials such as stainless steel, Viton® or Teflon®. A passivated interior surface on the stainless steel components is recommended.
- An oil free pump must be capable of circulating bin headspace at a minimum rate of approximately 500 ml/min and capable of pressurizing the sample canister(s) to a pressure of 15 psig.

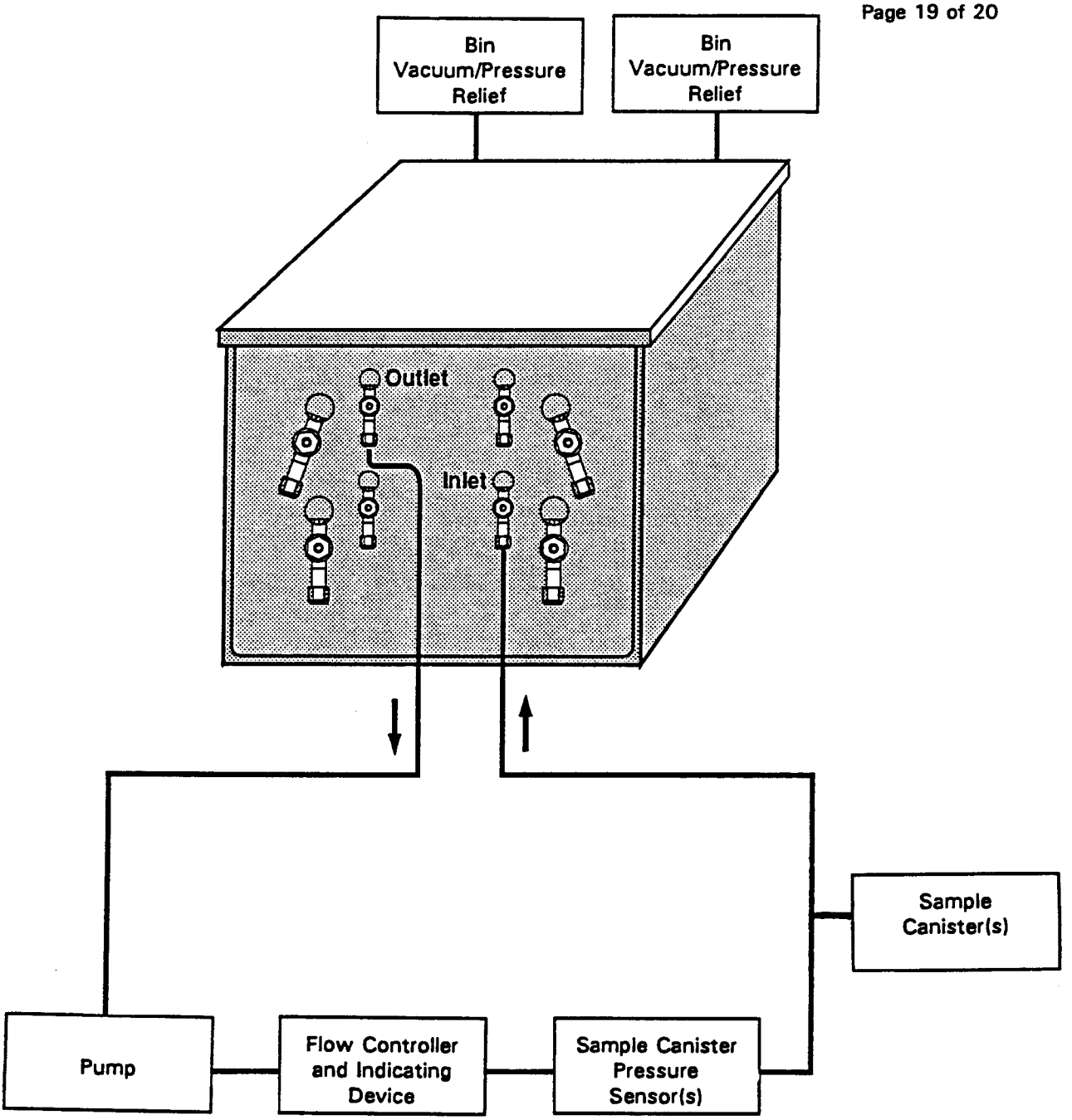


Figure 4-6. Bin Headspace Sampling Equipment Schematic

- Flow controlling devices must be capable of regulating pump flow rate if flow regulating is needed.
- Bin pressure regulating equipment must be redundant and must be capable of maintaining the internal bin pressure between a minimum of minus 0.8 psig and a maximum of 0.9 psig.
- SUMMA® passivated sample canister(s).
- Vacuum/pressure gauge(s) capable of measuring sample canister pressure between 0.05 mm Hg and 1000 mm Hg.
- Ambient pressure and temperature sensors (see Section 4.1.3).
- Timer capable of controlling, manually or automatically, the recirculation duration.

4.5.4 Quality Control Measures

Quality control measures must include cleaning and leak checking the bin sampling system. The system must be cleaned by purging with humidified zero air. After purging, a sample of zero air must be collected through the system and analyzed for the Program required analytes. System cleanliness may also be checked by purging with zero air and monitoring the zero air with a photoionization detector.

The system must also be checked for leaks after each cleaning and prior to use. This must be performed by pressurizing the system with zero air and checking for leaks by monitoring the internal pressure or a soap bubble-type check.

Reference standards and field duplicates must be collected in accordance with the frequencies specified in Table 9-5 and 9-6. A reference standard (pressurized cylinder gas containing known analytes at known concentrations) must be collected using the bin headspace sampling apparatus. The results of the analysis of this sample indicate the efficiency of sampling and analytical recovery. Provided the pressure differential across the bin does not exceed a minimum of minus 0.8 psig and a maximum of 0.9 psig, field duplicates can be collected.

5.0 SAMPLE CUSTODY

In accordance with ASME NQA-1, Elements 8 and 13, this section provides identification and control of samples, waste containers, 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 and defensibility, chain of custody (COC) must be maintained and documented for waste containers, 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 container, 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 containers and bins will be considered under effective custody control if they are sealed (i.e., unopened waste containers 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 waste containers, 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. Copies of the original COC forms shall be included in the bin case

report provided to WPO. The original COC forms shall be maintained in the site project files, unless DOE/WPO request them.

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

5.1 Waste Container COC

COC on individual waste containers shall be initiated at the time the waste containers are removed from stored inventories. Waste container custody must be maintained until the waste container's contents are placed in a bin or until the waste container is removed from the test Program as a result of failing to meet program requirements. Custody records must document which waste containers were emplaced in each bin. An example waste container COC form is provided in Figure 5-2. Site QAPjPs must include copies of forms used to document waste container 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 waste container numbers for each waste container under custody;
- Signatures of custodians relinquishing and receiving custody, along with the date and time of the transfer;
- Description of final waste container disposition, along with signature of individual removing waste container 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;

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			

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

- Signatures of custodians relinquishing and receiving custody, along with date and time of the transfer; and
- Comment section.

5.3 Bin COC

COC for bins shall be initiated upon introduction of wastes to the bin. Bin custody must be maintained until the bin is loaded in a TRUPACT-II 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 waste containers, 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.

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

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.

5.4.1 Gas 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 canister prior to shipment to the field. 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 completed tags must include the following:

- Applicable waste container or bin number
- Sample identification number (13 digits, as described later in this section)
- Sampler signature
- Ambient temperature and pressure (°C and mm Hg, respectively), when required
- Comment section
- Requested analyses
- 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.

- When a sampling manifold is used, the sampling manifold pressure must be recorded when required.

- In the analytical laboratory, canister pressure must be recorded within 24 hours of validated time of sample receipt (VTSR) and immediately prior to sample preparation or 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.

FRONT:

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

Z Z — 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	
VOCs	
Other	

* Report detected but unquantifiable analytes

Figure 5-5. Gas Sample Canister Tag
EXAMPLE ONLY

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.

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:

ZZ	MMDDYY	AAXXX
sampling site ID	date	canister ID

where ZZ 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.

5.4.3 Innermost Layer of Confinement Identification Number

Each innermost layer of confinement, as it is removed from within a waste container, must be marked with an identification number according to the following scheme. The first innermost layer of confinement removed from within each waste container must be labeled "1," and the last removed labeled "n," where n corresponds to the total number of innermost layers of confinement in the waste container. The innermost layer of confinement identification number must be recorded on the QC Sample Record Form, Figure 5-6. This record will allow field sample identification numbers to be correlated with both the innermost layer of confinement identification numbers and the waste container number.

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. Reference standards collected during bin sampling (Section 8.0) do not have to be 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:

FIELD QC SAMPLE RECORD FORM

Date: _____
Bin Number: _____
Sampler: _____

	DRUM/BIN NO.		DRUM/BIN NO.		DRUM/BIN NO.	
SAMPLE TYPE*	Field Sample Number	Time	Field Sample Number	Time	Field Sample Number	Time

Comments (note any discrepancies): _____

*Sample Type Designator

Sx	- samples	ISn	- innermost layer of confinement samples
Dx	- duplicate samples	IDn	- innermost layer of confinement field duplicate
EBx	- equipment	IEn	- innermost layer of confinement sampling equipment blanks
FBx	- field blanks	IFBn	- innermost layer of confinement field blanks
FSx	- field standards	IFSn	- innermost layer of confinement field standards

Where x = O for organic; I for inorganic; and N for oxides of nitrogen
n = innermost layer of confinement number, 1,2,3,...

Figure 5-6. QC Sample Record Form
EXAMPLE ONLY

- Sample identification numbers of each sample collected from each waste container or bin, where waste containers or bins are identified by waste container or bin 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;
- Comment section; and
- Sample type designator.

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.

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 national certifying agencies (e.g., NIST) or manufacturers. All reference standards must have valid relationships to nationally recognized standards. If national standards do not 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, respectively, to ensure and provide documentation that items which fail 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 using procedures provided by manufacturers in equipment manuals.

Calibrated equipment must be uniquely identified. This can be accomplished by the use of the manufacturer's serial number, a calibration system identification number, or some 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 isolated 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 controls established and implemented in site QAPjPs and SOPs, respectively. These procedures must address performance criteria. When RTR equipment is in use, operational checks must be conducted at the beginning of each work shift. These checks shall also include observation of a test pattern to ensure that the RTR system has adequate video quality.

6.2 Radioassay (RA)

RA equipment must be calibrated and maintained in accordance with controls established and implemented in the site QAPjPs and SOPs, respectively. SOPs must cover the system calibration, routine performance checks, and operation of the system. For any types of RA systems which are covered by ANSI, ASTM or other consensus standards, the site SOPs should be consistent with all relevant provisions of these standards.

Complete verification of calibration of RA for at least one counting geometry/sample matrix combination must be repeated at least annually. Calibration standards shall be prepared from primary standards obtained from suppliers maintaining measurement systems traceable to NIST whenever such standards are available. When standards are not available from such suppliers, the actual standards used shall be calibrated against primary standards obtained from suppliers maintaining measurement

systems traceable to NIST. The documentation of this cross-calibration shall be retained as a quality assurance record. Calibration standards shall be prepared using isotopes, geometries, and matrices having characteristics as close as possible to those expected for actual samples without compromising the quantitative integrity or homogeneity of the standard. The commonly accepted techniques of transmission and live-time corrections to compensate for matrix variations present within a drum are acceptable for the SGS gamma-ray assay technique. Computer programs used to calculate activities of radioisotopes may use correction algorithms to compensate for some waste characteristics such as waste density, gamma absorption, neutron moderator and absorption indices for the PAN systems. These programs shall be documented, verified and validated as required in NQA-1, Element 11 and Supplement 11S-2, Supplementary Requirements for Computer Program Testing.

All RA equipment shall receive routine performance checks for such parameters as system counting efficiency and system background. Spectrometry based systems shall also receive routine performance checks for energy calibration and resolution. Routine performance checks shall be performed with check sources which are stable and constant or which change only by well-established and predictable quantities (e.g., radioisotope decay). Site SOPs for performance checks shall state the standards used, frequencies for each test, record keeping, control limits, and corrective actions to be taken when a control limit is exceeded. Control charts (e.g., based on acceptable ranges or variances) should be used to track trends in the parameters measured in performance checks. Performance checks shall be performed and documented at least twice each shift. These checks shall be performed prior to any actual waste measurements on each work shift and after completion of all waste measurements for that shift. When shift operations are contiguous or overlapping, the performance checks for the end of the shift completing work can be the same performance checks as those done at the beginning of the shift starting work.

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.

6.3.1 Certification Check of the Sampling Area Pressure Sensors

The sampling area pressure sensors(s) must be certified prior to initial use, then annually, using NIST standards. If necessary, the pressure indicated by the pressure sensor(s) must be temperature compensated.

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 traceable 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 audio/video equipment. Checks of this equipment must include observation of test patterns, prior to each day's use, to ensure adequate video quality.

6.5 Determination of Headspace Gases

There are several methods for the analysis of headspace gas samples collected in SUMMA® passivated canisters for the constituents listed in Table 3-6. 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 listed in Table 3-6, 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 3-6. 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 3-6.

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 3-6 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 fail to meet the acceptance criteria of ±10% or closures fail to 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

Calibration procedures involve generating a multipoint initial calibration. This calibration curve shall be run initially, when indicated as a QC action, and when a change in the instrument may in any way affect the analytical results. The initial calibration is followed by daily verification with a calibration check standard.

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 3-6 or for the analytes to be determined by GC. These standards must be certified by the manufacturer.

6.5.2.2 Calibration Procedures

Procedures for establishing initial and continuing calibration for gas chromatography are detailed in this section.

Initial Calibration. A multipoint calibration curve, consisting of a minimum of three standards, shall be generated for all analytes using gas standards. The low standard shall be at five times the MDL or at the PRDL (Table 3-6), 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. Continuing calibration check standards must be analyzed at the beginning of each 12-hour shift. The laboratory control sample (LCS) may be used for the continuing calibration check. If a standard(s) other than the LCS is used for continuing calibration, then the LCS must be run as a sample during the analytical batch. The calibration check standard must be from a separate source than that used for the initial instrument calibration. Recalibration is required when a calibration verification fails or when a change in the instrument may affect the analytical results. The response factor (or concentration) of the continuing calibration check standard shall be compared to the corresponding average response factor (or concentration) from the most recent valid initial calibration. If the RPD between the average response factor and that of the continuing calibration check is less than 10%, the GC system will be considered calibrated.

6.5.3 Spectrophotometry

A multipoint calibration curve, consisting of a blank and a minimum of 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 0.67 moles of $NaNO_2$ produces an equivalent absorbence 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 five times the MDL or PRDL (Table 3-6), 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 Determination of Headspace 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 3-7 which will be collected in SUMMA® passivated canisters. The differences between these methods are the manner in which the gas sample is introduced into 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* (USEPA, 1988a) for gas sample introduction into the GCMS. Calibration procedures used for the analysis of VOC canister samples by these methods are derived from EPA Compendium Method TO-14 (USEPA, 1988b) and the third edition of EPA SW-846, Method 8240/8260 (USEPA, 1986b). 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.

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	%D for all compounds within 30% of initial calibration
GC	3-pt initial calibration	Initially and as needed	%RSD all compounds <30%
GC	Continuing calibration	Every 12 hours	%D for all compounds within 30% of initial calibration, RRTs within 3 standard deviations of initial calibration

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 3-7. 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 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 3-7. 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 SOPs 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 analysis of 50 ng of bromofluorobenzene (BFB). 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 3-7. One of the standards must be at an amount (nanograms) equivalent to the PRQLs specified in Table 3-7.

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

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 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 initial calibration. If the percent difference (%D) between the average response factor and the continuing midpoint response factor is less than 30%, 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 30% 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. This curve shall be verified using a 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 3-7. 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 initial calibration curve.

Initial Calibration. A multipoint external standard calibration shall be performed. The multipoint calibration must consist of a minimum of three secondary analytical standards that define the linear range of the instrument for the alcohols and ketones listed in Table 3-7. One of the standards must be at an amount (nanograms) equivalent to the PRQLs specified in Table 3-7.

Response factors shall be generated for each specified target analyte. For the initial multipoint 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.

Retention time windows shall be established for all analytes. Retention time windows are determined by injecting a minimum of three standards over a period of 72 hours. Retention time windows shall be calculated as the mean plus or minus three times the standard deviation of the individual retention times for each calibration standard analyzed in the 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight. Retention time windows must be determined for all analytes on each GC column prior to the analysis of any samples and whenever a new GC column is installed.

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 %D 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. 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.

6.7 Flammability Testing

Calibration requirements and frequencies are summarized in Table 6-3. Guidance Manual Procedure 810.1 describes the Program-required procedure for evaluating the flammability of bin headspace samples that contain a summed concentration of flammable VOCs exceeding 500 ppmv. DOE generator/storage sites' analytical laboratories or their contract laboratories shall maintain the most current revision of the Guidance Manual as the source for Procedure 810.1. Site QAPjPs and SOPs shall adopt all calibration and maintenance procedures described in Guidance Manual Procedure 810.1.

In addition to calibration procedures, operational checks shall be performed at the beginning of each working day prior to the testing of standards or samples. Operational checks shall include:

- Examination of the structural integrity of the flame test vessel (no cracks or deformations)
- Examination of the cleanliness of the flame test vessel (no residual material or soot from previous testing)
- Inspection of the ignition source power output (clearly visible spark produced between the electrodes)
- Verification of the pressure stability of the test vessel and all connecting lines (apparatus capable of maintaining a vacuum of 30 ±1.5 mm Hg for five minutes).

6.7.1 Standards Preparation

Standards for flammability testing shall use pure compounds and shall be prepared according to the procedures given in Guidance Manual Procedure 810.1.

6.7.2 Calibration Procedures

Initial calibration of the flame test apparatus shall be performed by testing the flammability of 8.0% (v/v) methanol, 15.0% (v/v) methanol, and 1.0% (v/v) diethyl ether. Calibration of the flame test apparatus shall be maintained on a daily basis with 8.0% (v/v) methanol. Calibration is achieved when, under the test conditions specified in Guidance Manual Procedure 810.1, the 8.0% (v/v) methanol and 15% (v/v) methanol standards are evaluated as flammable, and the 1.0% (v/v) diethyl ether standard is evaluated as nonflammable. The calibration frequency and performance criteria are summarized in Table 6-3.

Table 6-3. Flammability Testing Calibration Requirements

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
Flame Test	3-pt initial calibration	Initially and after every 20 samples	8.0% Methanol - Flammable 15% Methanol - Flammable 1.0% Diethyl ether - Not Flammable
Flame Test	Continuing calibration	Beginning of each working day	8.0% Methanol - Flammable

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 and site-specific Item Description Code (IDC), as applicable
- Waste items, residual materials, packaging materials, and/or waste material categories inventory, as applicable
- Quantity of free liquids.

RTR is a nondestructive 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 such as wave motion of free liquids. An RTR system normally consists 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 and data acquisition 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.

The waste container is scanned while the operator views the television screen. An audio/videotape is made of the waste container scan and is maintained as a permanent record. An RTR data form is also used to document identifiable waste items, residual and packaging materials and their quantities.

7.2 Radioassay (RA)

Three types of RA 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. Software 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, ASTM C853 (ASTM, 1982), ANSI 15.20 (ANSI, 1975), and Nuclear Regulatory Commission (NRC) standard practices and guidelines (Regulatory Guide 5.11, USNRC, 1984) 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 assays shall be performed until the remedial action is satisfactorily completed.

The assay procedure for PAN units specifies the use of a computerized data acquisition system. An operator must insert a waste container into the PAN unit and enter all waste container 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.

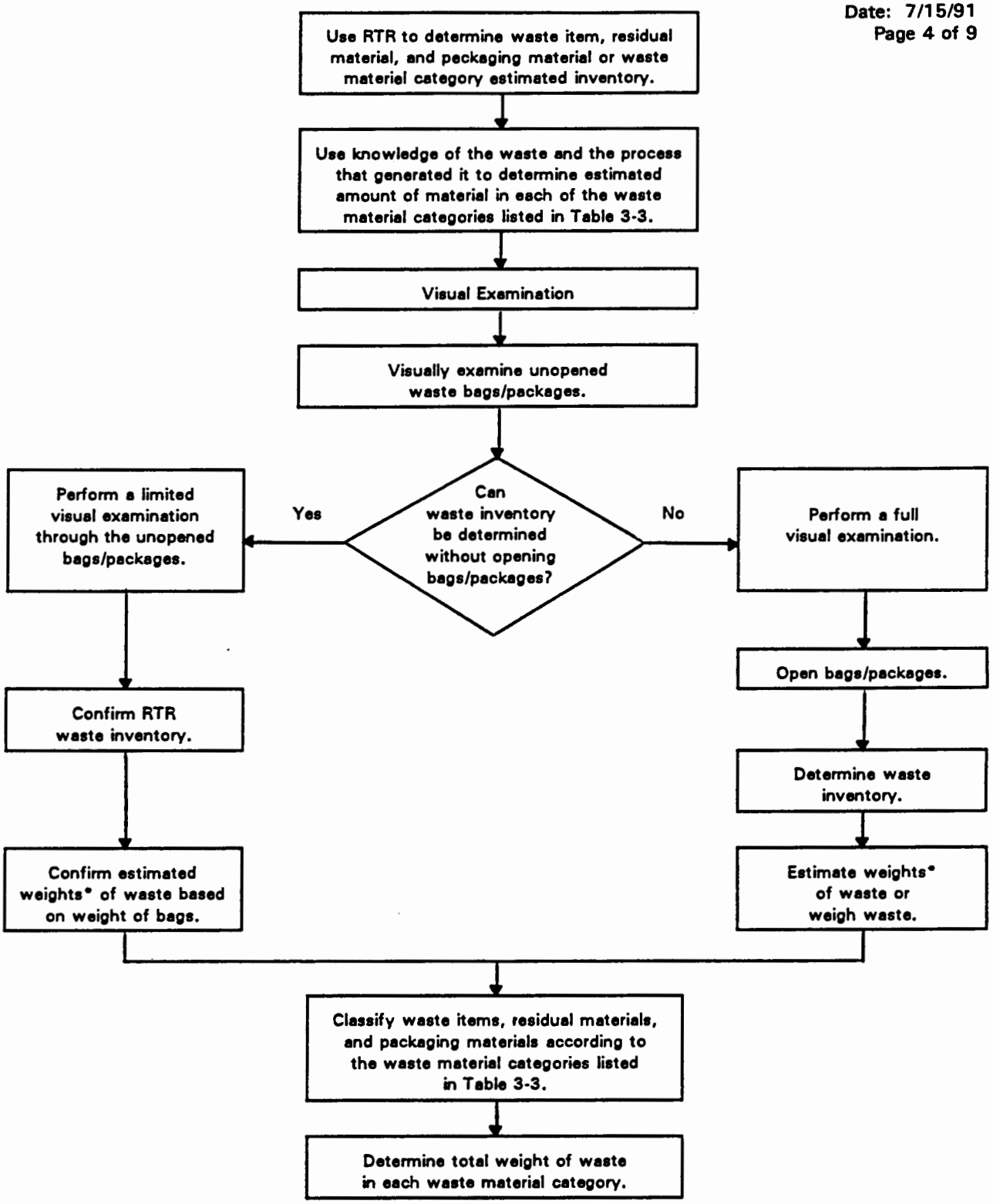
7.2.3 Passive Neutron Coincidence Counting (PNCC) Procedure

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, residual materials, and packaging materials in the waste container 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, residual materials, and packaging materials so that they can be classified according to the waste material categories listed in Table 3-3. Individual bags (or packages) and, if necessary, the contents of each bag within the waste container must be weighed, and the weights must be recorded. In cases where bags are not opened, 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.). Estimated or measured weights must also include process knowledge estimates of sorbed liquids, lead in leaded rubber, etc. for composite materials. 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.



*Include process knowledge estimates of sorbed liquids, lead in leaded rubber, etc. for composite materials.

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

Figure 7-1 illustrates a two-track approach to the visual examination of waste. Subsequent to RTR and waste container opening, 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 the weighing of bags is appropriate. If the waste is heterogeneous, the expert may decide a full visual examination involving determination of a waste item, residual material, and packaging material inventory by opening bags, segregating waste, and 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 Headspace Gases

Analytical methods for the analysis of 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 3-6 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 (optional) 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. For a positive pressure check, a canister passes if the pressure does not change by more than ± 2 psig in 24 hours. 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 3-5. 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 Gas 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 3-6. The method or methods selected for the analysis of headspace gases shall be the decision of each participating laboratory, provided the requirements of Table 3-6 and the WIPP Performance Demonstration Program (Section 10.2) are satisfied. These methods shall be specified and described in each site's QAPjP and SOPs.

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, nitrogen, oxygen, 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 and retention time windows 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 Determination of Organic Headspace Gases

Analytical methods for the analysis of VOCs collected using SUMMA® passivated canisters (see Section 7.4.1 for canister cleaning requirements) 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, 1988a). Detailed guidance procedures for the analysis of volatile organic compounds listed in Table 3-7 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 3-7.

7.5.1 VOC Analytical Methods

Analytical procedures must use GCMS for the analysis of VOCs listed in Table 3-7 (m-Xylene and p-Xylene cannot be separated by GCMS procedures). Alcohols and ketones may be analyzed by GC-FID if the GCMS methods cannot meet the criteria specified in Tables 3-5 and 3-7. The method or methods selected for the analysis of headspace VOCs shall be the decision of each participating laboratory provided the requirements of Tables 3-5 and 3-7, 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 (±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 3-7 and identification of compounds not listed in Table 3-7. 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 3-7, 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 3-5 and 3-7. Sample introduction shall be by thermostated gas injection valves with sample loops. The method must use two dissimilar 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.

7.6 Flammability Testing

The evaluation of the flammability of bin headspace samples is performed using a modified version of ASTM Method E 681-85. The modified method, which is given in the Guidance Manual as Procedure 810.1, shall be used by DOE generator/storage sites' analytical laboratories or their contract laboratories to perform flammability evaluations. The details of implementing Guidance Manual Procedure 810.1 shall be specified in site QAPjPs and laboratory SOPs.

Samples for flammability testing shall be sent to the laboratory in SUMMA® canisters. The sample introduction portion of the flame test apparatus shall provide a leak-tight seal of the SUMMA® canister to the flame test apparatus. The flame test vessel and all connecting lines shall be capable of being evacuated to less than 30 Torr. Samples shall be transferred to the 5 liter flame test vessel from SUMMA® canisters at ambient temperature.

As samples are expanded into the flame test vessel, the temperature and pressure within the flame test vessel shall be monitored and the final temperature and pressure recorded. Prior to ignition, the final temperature within the test vessel shall be at ambient and the final pressure not less than 400 Torr. At the minimum operating pressure of 400 Torr, a 2.63 L sample must be delivered to the flame test vessel.

Temperature measurements of the ambient air immediately surrounding the flame test vessel shall be accurate to ±4°C. Temperature measurements within the flame test vessel shall be accurate to ±1°C. Pressure measurements within the flame test vessel shall be accurate to ±0.2%. The calibration requirements for temperature and pressure sensors shall be specified in site SOPs.

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 here. To prevent the introduction of errors or the loss or misinterpretation of the data, adequate precautions must be taken. The general requirements discussed here should be considered minimum and will be expanded on throughout the subsections dealing with individual analytical or test techniques. Site QAPjPs must delineate the implementation of the requirements contained in this section. 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, legibly, 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, is defined as a suite of samples of similar matrix processed as a unit. This unit must not exceed 20 samples.

Data Reduction - Data Generation Level

Data reduction must be performed at the site of data generation such that all the requirements outlined in relevant 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 relevant subsections.
- The reporting units must be consistent with the appropriate method used and the requirements described in relevant subsections.
- All analytical data must be reported to the significant figures specified in relevant subsections and appropriate to the technique used.
- All data must receive independent technical review to ensure that the reduction is appropriate.

Data Reduction - Project Level

Prior to any data reduction at the site-project level, it should be verified that all data have been validated at the data generation level. The validated data must then be reduced further, when appropriate, to meet the requirements outlined in Section 8.7. Minimum requirements for data reduction shall include the following:

- The technique of data reduction shall be consistent with the equations/procedures outlined in Section 12.6.
- Appropriate unit conversions shall be made prior to comparison of the data to regulatory limits.
- All data must receive independent review to ensure that the reduction is appropriate and correct.

Data Validation

The data shall be validated at three levels. The first level of review is conducted on each analytical batch in the laboratory or on each waste container 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 waste container 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-waste container data) is complete and includes raw data, data forms, calculation records, QC summaries, narrative, COCs, and sample tags as appropriate (see text on page 8-4 for requirement regarding sample tag removal).

   - 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. QC criteria that were not met must be documented in a non-conformance report.

- 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. This verification should occur prior to project level data reduction.

   - 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.

   - Project level data reduction has occurred, when appropriate, and is consistent with the requirements outlined in Sections 8.7 and 12.0.

   - Project level reduced data are evaluated for compliance with regulatory requirements (Section 8.7).

   - Regulatory compliance data have been entered on the appropriate reporting forms.

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 minimum 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), as described in Section 12.6.

   - 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 report. If the Project Manager requests that sample canisters be retained for future use (e.g., experimental holding time study), new sample identification and chain-of-custody procedures should be initiated with a cross reference to the original sample.

Data Validation - DOE/WPO Level

The third and final level of data review shall occur at DOE/WPO and must, at a minimum, 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, compliance with regulatory requirements, and signature releases.
3. Application of procedures for the determination of data outliers detailed in Section 12.6.

DOE/WPO must validate a bin case report and notify the site of the acceptance status of the data within two weeks of report receipt. The sites will then submit an addendum report (with the bin case report cross-referenced) containing the compliance data generated from bin sampling (see Data Reporting - Project Level). This addendum report must be submitted and approved by DOE/WPO in the 20 working day time frame between bin sampling and TRUPACT-II loading.

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. In addition to the data forms, the analytical laboratory report must include:

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. The DOE-WIPP cover page form, Cover Page (Figure 8-5), is an example.

2. Case Narrative

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

RTR, RA, and Visual data must appear on forms which meet the requirements identified in subsequent subsections. The original data forms must be forwarded to the site Project Manager as soon as possible following data generation. A copy of the 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, waste container, and regulatory compliance 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

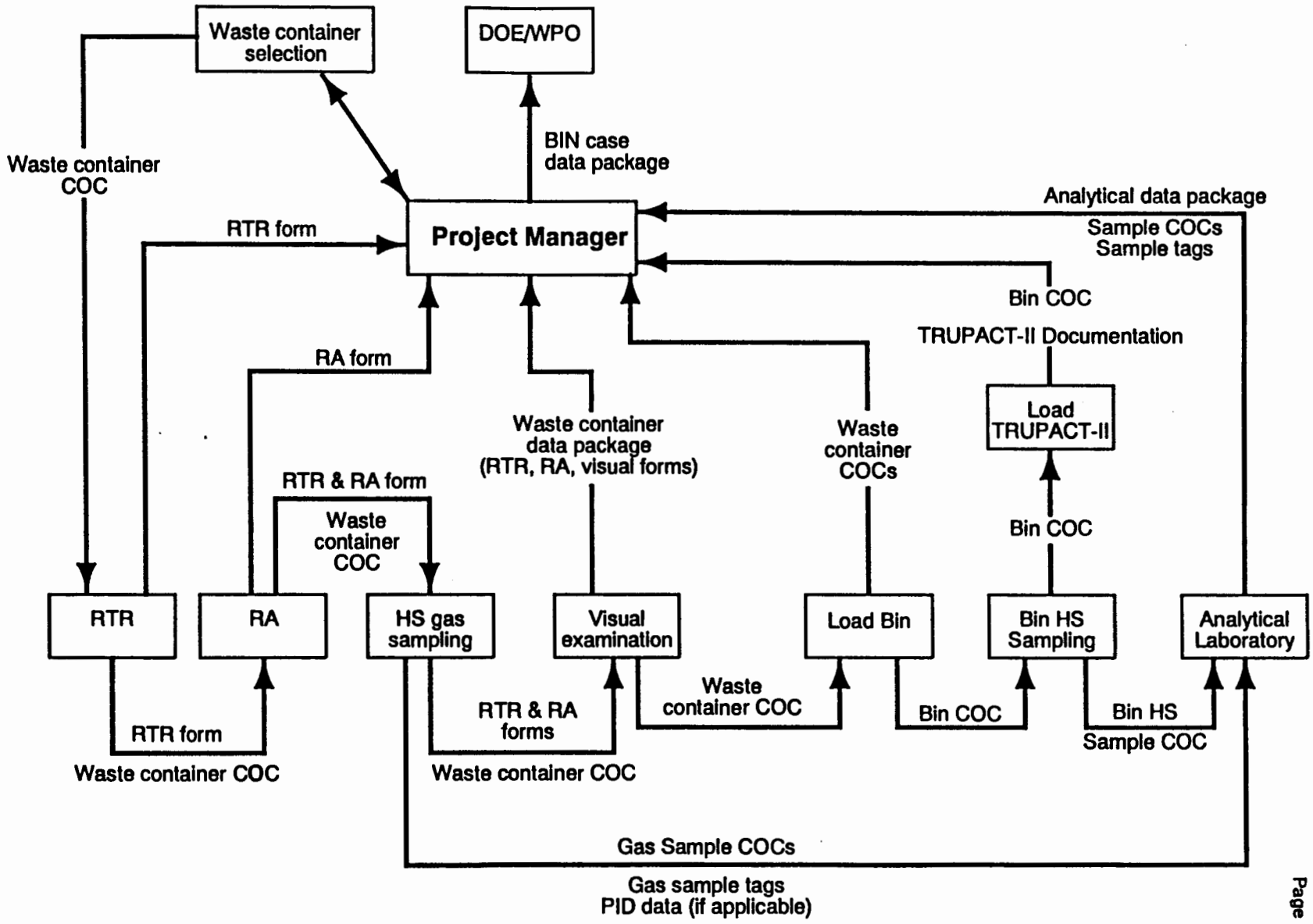


Figure 8-1. Overall Data Reporting Flow Chart

- 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 and occurrences during the sampling process that may impact data quality. Regulatory compliance shall also be discussed.
- Regulatory compliance reporting forms
- A result summary for each characterization technique
- All raw data.

Each bin case report shall have an addendum report associated with it. This addendum shall contain all the pertinent information regarding the bin sample collected (Section 4.5.1) to verify compliance with no-migration (Section 8.7.1.2) and flammability (Section 8.7.1.3). Comparability compliance data shall be submitted as part of the bin case report, not the addendum.

Addendum reports shall contain the same type of information that is required for the bin case report (e.g. narrative, raw data, chain of custody records and appropriate reporting forms) pertinent to the compliance bin sample and its analysis and reporting. DOE/WPO must notify the site of the acceptance status of the bin case report within two weeks of report receipt. The site shall then initiate bin sampling to verify compliance with the no-migration demonstration and flammability requirements. An addendum report shall be prepared by the site and submitted to DOE/WPO with the bin case report cross-referenced.

Section 4.5.1 requires that bin sampling must occur within 20 working days of TRUPACT-II loading. Considering emplacement time/date concerns, DOE/WPO must contact the sites regarding the acceptability of bin compliance (i.e. bin addendum report acceptance) within this same 20 working day time frame. DOE/WPO must notify the site (within 20 days of bin sampling) that all the applicable regulatory compliance requirements have been satisfied before a bin may be loaded into the TRUPACT II and shipped to the WIPP facility. It is recommended that the sites expedite the addendum report to allow DOE/WPO five working days for the review and site notification cycle.

The site Project Manager must maintain a record of transmittal regarding the submission of the bin case report and its addendum 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, residual materials, and packaging materials documented on an RTR data form must be used in conjunction with site-specific data on standard weights to classify the waste items, residual materials, and packaging materials into the waste material categories listed in Table 3-3 and to estimate the total weight of waste material in each of 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. Involved DOE sites should coordinate these efforts to assure maximum consistency.

8.1.2 RTR Data Reporting

If applicable, the data reporting requirements for RTR are:

- Examining site
- Waste container identification number
- Content Code or IDC, as applicable
- Any changes made to IDC
- Date of RTR examination
- Operator signature/date
- Reviewer signature/date
- Presence/absence of waste container liner (yes/no)
- Estimated inventory of waste container contents, e.g., number/count of a particular waste item, or waste material categories, as applicable
- 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 completed 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.

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 8-2a. 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. Estimated inventory of Waste Container Contents

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

Figure 8-2b. 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 8-2c. 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 MATERIAL CATEGORIES	Estimated Weight (kg)	Error (kg)
CELLULOSICS	_____	_____
Other organics	_____	_____
Plastics	_____	_____
Rubber	_____	_____
Corroding metal - steel	_____	_____
Corroding metal - aluminum	_____	_____
Noncorroding metal	_____	_____
Solid inorganic wastes	_____	_____
Inorganic sludges	_____	_____
Cements	_____	_____
Totals	_____	_____

Comments (note any descrepancies):

_____

_____

_____

Figure 8-2d. RTR Data Form

(Continued)

EXAMPLE ONLY

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
- 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

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

- 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)
- 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 3-3 (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 completed and signed. Figure 8-1 indicates how the visual examination data sheet should travel through the waste characterization process.

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 and location 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 8-4a. 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 8-4b. 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 8-4c. 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 MATERIAL CATEGORIES	Estimated Weight (kg)	Error (kg)
CELLULOSICS	_____	_____
Other organics	_____	_____
Plastics	_____	_____
Rubber	_____	_____
Corroding metal - steel	_____	_____
Corroding metal - aluminum	_____	_____
Noncorroding metal	_____	_____
Solid inorganic wastes	_____	_____
Inorganic sludges	_____	_____
Cements/Estimated water content (wt%)	_____	_____
Totals	_____	_____

Comments (note any discrepancies):

_____

_____

_____

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

8.4 Gas Analysis

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

8.4.1 Data Reduction

All analyte gas 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.

Quantitative values shall be reported in units of volume percent (vol. %) down to the MDL. Values for analytes analyzed for but not detected shall be reported as the MDL with the required "U" flag (i.e., if the MDL is 0.001 vol. %, report 0.001 U). Analytical results, including MDL values used in reporting, must be corrected for dilution.

The numerical value assigned to the MDL for a given analyte shall include two significant figures when the MDL is greater than or equal to 10% of the corresponding PRDL. If the MDL is less than 10% of the PRDL, its numerical value shall be reported with one significant figure. (Note: Under no circumstances is a value reported as all zeros acceptable.) Analytical results above the MDL shall be reported to a sufficient number of decimal places to include the least significant figure of the MDL, but to no more than three significant figures.

To illustrate implementation of these reporting requirements, consider the example of an analyte having a PRDL of 0.1 vol. %. If the MDL for this analyte is 0.024 vol. %, then a result of 0.09265 would be reported as 0.093. If the MDL is 0.0008 vol. %, then 0.09265 would be reported as 0.0926.

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.

8.4.2 Data Validation

All gas analysis data must be reviewed and approved prior to being reported. This validation process must include the three levels of review which are 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 3-6 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 DOE/WPO 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. 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 3-6. 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. Gas analysis data qualifying flags shall be used. These are as follows:

      Flag qualifier:
      B - Analyte detected in the blank.
      E - Analyte exceeds the calibration curve.
      J - Analyte less than the PRDL and greater than or equal to the MDL.
      U - Analyte was undetected.

      The method qualifiers (M) are:
      MS - Mass Spectrometry
      GC - Gas Chromatography
      CO - 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 3-6. 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, and date and time the blank 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

DOE-WIPP
COVER PAGE

Lab Name: _____

Lab Code: _____ Analytical Batch No.: _____

Field 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

EXAMPLE ONLY

DOE-WIPP
FORM 1
GAS ANALYSIS DATA SHEET

FIELD SAMPLE NO.

Lab Name: _____

Lab Code: _____

Analytical Batch No.: _____

Lab Sample ID: _____

Date Received: _____

Analysis Date: _____

Concentration Units (Vol. %)

Analyte	Concentration	Flag	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

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.

2. 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, to include sample calculations and forms.

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.

DOE-WIPP
FORM 4
DUPLICATES

FIELD 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			
Nitrogen 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

8.5.2 Data Validation

Data review at the generation and site project levels shall verify that criteria specified in Tables 3-5, 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. Reporting form flags:
   B - If the analyte was detected in the associated laboratory blank.
   E - If the analyte concentration exceeded the initial calibration curve.
   J - If the analyte was detected at levels less than the PRQL but greater than the MDL.
   U - If the analyte was not detected.

2. GCMS reporting forms:

   EPA Contract Laboratory Program (CLP) reporting forms, modified to accommodate the Table 3-7 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 listed in Table 3-7 analyzed for by GCMS (p-xylene and m-xylene may be reported as p/m-xylene). For analytes not detected, the analyte MDL corrected for sample volume must be reported. 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 3-7 and analyzed by GCMS 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 3-7 and analyzed by GCMS. 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 3-7 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.

i. Copies of data system quantitation reports (including all mass spectra and chromatograms) for all calibration standards, samples and blanks.

j. Copies of mass spectra for all reported TICs.

3. 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 3-7. For analytes not detected, the analyte MDL corrected for sample volume must be reported. 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.

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			
p/m-Xylene			
o-Xylene			

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

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 listing the response factors for the alcohols and ketones listed in Table 3-7 at all calibration points, the average response factor or for each analyte, the average retention time, the retention time window, and the percent relative standard deviation for each analyte across the 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 average response factor, and absolute retention time for each alcohol and ketone listed in Table 3-7. 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.

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 3-7 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.

8.6 Flammability Testing

Flammability testing results shall be documented and available to data users on standardized forms approved by DOE/WPO.

8.6.1 Data Reduction

The qualitative results from flammability testing require no data reduction. Flammability testing results shall be reported with the information required in Section 8.6.3.

8.6.2 Data Validation

The general data validation and review requirements of Section 8.0 are sufficient to ensure that the criteria in Table 6-3 have been satisfied for all flammability testing results.

8.6.3 Data Reporting

The results of flammability testing shall be reported on a standardized form approved by DOE/WPO. An example form is detailed in Figure 8.8. At a minimum, the form shall contain the following information:

- Laboratory name
- Bin identification number
- Bin field sample identification number
- Flammability test date and time
- Initial calibration data
- Continuing calibration data
- Field sample test data.

Flammability test results shall be included in bin case data packages/reports when the summed concentration of flammable VOCs in a bin headspace sample exceeds 500 ppmv at the 90% confidence level.

8.7 Regulatory Compliance Reduction and Reporting Requirements

Prior to shipment of experimental waste to the WIPP for bin-scale and alcove testing, it must be demonstrated, by waste type, that all applicable regulatory requirements addressed under this Program have been met. Report documentation and all supporting data must be submitted to and approved by DOE/WPO before experimental waste may be shipped to the WIPP facility. Report documentation shall include the bin case report and the associated addendum report.

Experimental waste characterization data must be validated at the site project level and then subjected to another data reduction step. Section 12.6 details the reduction calculations that must be applied to regulatory compliance data prior to reporting. These calculations adjust the measured data to the upper confidence limit (X_{UL}) to enable comparison with the regulatory limits. In addition, data must be expressed in appropriate units before it is compared to regulatory limits in order to demonstrate compliance.

Procedures for regulatory compliance data reporting are identified in this section. Specific procedures for the implementation of reporting requirements must be detailed in site-specific QAPjPs and SOPs, as appropriate.

Bin ID Number:_____ Flammable: _____ Yes _____ No

Laboratory Name: _____

Laboratory Code:_____

Initial Calibration Information:[a]

Standard	Date	Time	Flammability Evaluation[b]
8.0% Methanol			
15.0% Methanol			
1.0% Diethyl ether			

Continuing Calibration Data:[c]

Standard	Date	Time	Flammability Evaluation
8.0% Methanol			

Sample Data:

Field Sample ID No.	Date	Time	Flammability Evaluation

[a] Valid initial calibration data
[b] Yes/No
[c] If applicable

Figure 8-8. Flammability Testing Reporting Form
EXAMPLE ONLY

8.7.1 Reporting Requirements for the WIPP No-Migration Determination (NMD)

Procedures for the evaluation of NMD compliance data and minimum reporting requirements are identified in this subsection. Regulatory compliance data must be reported on DOE/WPO approved forms. Example forms are provided throughout subsequent subsections.

8.7.1.1 Comparability

In order to comply with the maximum concentration comparability requirement of the NMD, as outlined in Section 1.3, the upper 90% confidence limit values (X_{UL}) of the measured headspace concentrations from the 55-gallon poly bag and innermost layers of confinement of each waste container must be compared to the maximum allowable VOC concentrations in EPA's NMD. If the regulatory requirement is satisfied, the waste container may be included in the bin-scale test.

Table 1-3 lists the maximum allowable concentrations (herein referred to as the regulatory limits) of the five major VOC hazardous constituents of concern.

Data obtained (X_{UL}) for headspace samples must be compared to the regulatory limits by the method described below:

- For each waste container, identify the highest X_{UL} data point for each constituent. (Consider the 55-gallon poly bag and the innermost layer headspace data.)
- Compare this highest X_{UL} constituent concentration to the regulatory limits listed in Table 1-3.
- If the highest X_{UL} constituent concentration is below the regulatory limit, then the waste container passes for that constituent.
- Repeat the comparison for all constituents.
- If all the highest X_{UL} constituent concentrations are below the regulatory limits, then the waste container has met the regulatory requirement and may be included in the bin-scale test.

Comparability compliance data shall be reported for each waste container to be included in the bin-scale test and must include the following minimum information:

- Bin identification number
- Waste container identification number
- Waste type (as identified in USDOE, 1989)

- All the inner layers of confinement (55-gallon poly bag and innermost layers of confinement) headspace data for the five hazardous constituents, X_{UL}
- The appropriate regulatory limits for each constituent (i.e., as identified by waste type in Table 1-3)
- Identification of the highest X_{UL} data point for each constituent (for all the headspace samples pertaining to the waste container)
- Decision indicator regarding the comparison of headspace data to the regulatory limits.

Comparability data shall be submitted as part of the bin case report. Figure 8-9 is provided as an example of a comparability reporting form.

8.7.1.2 No-Migration Demonstration

In order to comply with the demonstration of no-migration during the bin-scale tests, the bin headspace concentrations of three VOC hazardous constituents must not exceed the NMD mean values (regulatory limits). These hazardous constituents and the associated regulatory limits are listed in Table 1-4.

The upper 90% confidence limit values (X_{UL}) of the measured bin headspace concentrations of these constituents must not exceed the regulatory limits for a bin to be shipped to the WIPP. Compliance with this regulatory requirement must be verified and documented on a reporting form for inclusion in the bin case report. DOE/WPO must verify that the no-migration demonstration requirements have been met before the bin can be shipped. The no-migration demonstration reporting form must consist of the following minimum information:

- Bin identification number
- Bin headspace sample identification number
- Waste type
- X_{UL} sample data for the three hazardous constituents
- Constituent regulatory limits as identified in Table 1-4, as appropriate to the waste type
- Decision indicator regarding whether the bin headspace data (X_{UL}) are less than the regulatory limits.

Figure 8-10 depicts an example No-Migration Demonstration Compliance Reporting Form.

Bin ID Number:_____ Comparability Criteria Met: ____ Yes ____ No

Waste Type[a]: _____

Waste Container ID Number: _____

Constituent	Carbon Tetrachloride	Methylene Chloride	1,1,1-Trichloroethane	Trichloro-ethylene	1,1,2-Trichloro-1,2,2-trifluoroethane
Field Sample ID#[b]	Sample Data[c] (X_{UL})				
Regulatory Limit[d]					
Highest Constituent Concentration (X_{UL})					
Decision[e]					

[a]Waste types are identified in USDOE, 1989.

[b]To include 55-gallon poly bag (first entry on form) and all innermost layer of confinement samples.

[c]Sample constituent concentrations (volume %) at the upper confidence limit (X_{UL}).

[d]Maximum allowable VOC headspace concentrations (volume %) as appropriate for the waste type (Table 1-3).

[e]Pass if constituent concentration (X_{UL}) is less than the regulatory limit.

Fail if constituent concentration (X_{UL}) is greater than the regulatory limit.

Figure 8-9. Comparability Compliance Reporting Form

EXAMPLE ONLY

Bin ID Number: _____ NMD Criteria Met: ____ Yes ____ No

Bin Sample ID Number: _____

Waste Type[a]: _____

Constituent	Regulatory Limit[b]	Bin Sample Data[c] (X_{UL})	Decision[d]
Carbon Tetrachloride			
Methylene Chloride			
Trichloroethylene			

[a]Waste types are identified in USDOE, 1989.

[b]Allowable mean volatile organic headspace concentrations (volume %) as appropriate for the waste type (Table 1-4).

[c]Bin sample constituent concentrations (X_{UL}) in volume %.

[d]Pass if constituent concentration (X_{UL}) is less than the regulatory limit.
Fail if constituent concentration (X_{UL}) is greater than the regulatory limit.

Figure 8-10. No-Migration Demonstration Compliance Reporting Form

EXAMPLE ONLY

No-migration demonstration compliance data shall be sumbitted to DOE/WPO as part of an addendum report with the appropriate bin case report cross-referenced. (See Data Reporting-Project level, Section 8.0.)

8.7.1.3 Flammability

Bin flammability compliance must be documented on a DOE/WPO approved reporting form. DOE/WPO must verify that flammability requirements have been met before the bin can be shipped to the WIPP facility. An example Flammability Compliance Reporting Form is provided in Figure 8-11. The data reporting requirements for flammability are:

- Bin identification number
- Waste type
- Bin loading date
- Upper confidence limit (X_{UL}) concentrations of flammable VOCs
- Flame test results, if applicable
- Upper confidence limit (X_{UL}) concentrations of hydrogen and methane
- Result of Le Chatelier calculation for hydrogen and methane; fraction of the LEL
- Indication of whether the upper confidence limit of the summed flammable VOC concentrations is greater than 500 ppmv
- Indication of whether flame test was positive or negative, if applicable
- Indication of whether hydrogen/methane are present at concentrations less than 50% of the mixture's LEL.
- Number of days remaining until 50% of the LEL is exceeded, specifying method of determination, i.e., sampled or not sampled.

The method for determining emplacement time is:

(a) For sampled bins,

- Obtain the bin headspace hydrogen and methane concentrations.
- Identify the TRUPACT-II shipping category.
- Based on these values, the number of days remaining until 50% of the LEL is exceeded is determined from Tables 12-3 through 12-7. The times reported in Tables 12-3 through 12-7 include an allowance for a three-day shipping period.

Bin ID Number: _____

Bin Sample ID Number: _____ Flammability Criteria Met: _____ Yes _____ No

Waste Type:[a] _____

Date Bin Loaded: _____

Flammable VOCs	Volume %[b]
Acetone	
Benzene	
n-Butanol	
2-Butanone	
Chlorobenzene	
Cyclohexane	
1,1-Dichloroethane	
1,2-Dichloroethane	
1,1-Dichloroethene	
cis-1,2-Dichloroethene	
Ethyl benzene	
Diethyl ether	
Methanol	
4-Methyl-2-pentanone	
Toluene	
1,2,4-Trimethylbenzene	
1,3,5-Trimethylbenzene	
o-Xylene	
m-Xylene	
p-Xylene	
Total	
Total (X_{UL})	

Figure 8-11a. Flammability Compliance Reporting Form
EXAMPLE ONLY

Gases	X_{UL} (Vol. %)
H_2	
CH_4	

Le Chatelier Calculation[c]	Result
H_2/CH_4	

Regulatory Criteria	Decision[d]
≥500 ppmv Flammable VOCs	
Flame test	

50% of the LEL Evaluation for H_2/CH_4	Emplacement Time (Days)[e]	Emplacement Date[f]
Sampled[g]		
Not sampled[h]		

[a] Waste types are identified in USDOE, 1989.

[b] A value of one-half the MDL must be used when a flammable VOC is not detected.

[c] If over 0.5 of the LEL, this bin may be shipped but will require purging at the WIPP prior to emplacement.

[d] Yes/No

[e] If the bin is not emplaced within this designated time frame, it must be purged prior to emplacement.

[f] Date by which the bin must be emplaced at the WIPP without sampling and/or purging.

[g] Value from Tables 12-3 through 12-7, Section 12.6.

[h] Value from Table 12-2, Section 12.6.

Figure 8-11b. Flammability Compliance Reporting Form
(Continued)
EXAMPLE ONLY

(b) For bins not sampled,

- Identify the TRUPACT-II shipping category.
- Determine the number of days using Table 12-2 and the time since the bin was loaded.

Flammability compliance data shall be submitted to DOE/WPO as part of the addendum report, with the appropriate bin case report cross-referenced. (See Data Reporting - Project Level, Section 8.0.)

In addition, a DOE/WPO approved Flammability Quality Assurance Objectives Reporting Form (Figure 8-12) must be submitted as part of the addendum report. This form will verify that the ±10% precision and accuracy requirements for flammable VOCs have been met. Flammability QAO determination procedures are described in Section 12.6.2.5. QAOs must be determined initially and then updated according to the frequency specified in Section 12.6.2.5. The data reported on the form must reflect the updates when appropriate. All data supporting these flammable VOC QAO determinations must be documented and available for auditing purposes.

VOC Flammability Quality Assurance Objectives [a]

Total Measured VOCs	Value (ppmv)
T_1 T_2 T_3 T_4 T_5 . . . . . T_{30}	
Average of Total VOCs ($\bar{T}$)[b]	
Total of the known VOCs (KT)[b]	
Standard Deviation (S_T)	
Bias[c] ($\bar{T}$/KT)	
Precision[d] ($S_T/\bar{T}$)	

[a] As described in QAPP Section 12.6.2.5 and updated semiannually.
[b] Expressed in ppmv.
[c] $\leq 0.90 \leq$ Bias ≤ 1.10.
[d] Precision ≤ 0.10.

Figure 8-12. VOC Flammability Quality Assurance Objectives Reporting Form
EXAMPLE ONLY

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 internal QC tests including routine measurements of performance standards, replicates, and blanks. These internal 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 allowed 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 performed 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. The system shall be operated in statistical control as determined by the control limits established by site SOPs.

As discussed in Section 6, routine performance checks shall be performed on all RA systems according to approved SOPs. Performance checks are required 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 in accordance with the site QAPjP and SOPs. 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 allowed to operate an RA system.

9.1.3 55-Gallon Poly Bag 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 55-gallon poly bag prior to sampling. Required 55-gallon poly bag 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 55-Gallon Poly Bag 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 prior to work	1/day prior to work	1/day prior to work
Field reference standards[b]	1/bin	1/bin	1/bin
Field duplicate samples	1/bin	1/bin	1/bin

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

[b] One field reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Section 3.3.2.1 prior to first use, then at the specified frequency thereafter.

* For Gas Mass Spectrometry analysis, a separate sample for NO_x determination is not required to be collected in addition to the gas sample.

Table 9-2. Summary of the Number and Type of 55-Gallon Poly Bag Field QC Headspace Samples

Routine	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[b]	1/bin	1/bin	1/bin
Field duplicate samples	1/bin	1/bin	1/bin

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

[b] One field reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Section 3.3.2.1 prior to first use, then at the specified frequency thereafter.

* For Gas Mass Spectrometry analysis, a separate sample for NO_x determination is not required to be collected in addition to the gas sample.

Field Blanks (FBs). FBs shall be used to evaluate background levels. FBs must initially be collected prior to sample collection 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 prior to first use of the manifold for sample collection. After the initial accuracy check, FRSs must be collected at a frequency of one per bin. FRS results shall be acceptable if the criteria specified in Tables 3-5 and 3-6 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 that 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. Field duplicate results shall be acceptable if the criteria in Tables 3-5 and 3-6 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 Innermost Layer of Confinement Headspace Sampling

Field QC checks shall be accomplished by the preparation and submission of control samples from the field. Control samples shall be used to check equipment and sampling operations in the field and to monitor ambient air quality in the vicinity of the innermost layer of confinement sampling operation. Required innermost layer of confinement field QC sample frequencies are summarized in Tables 9-3 and 9-4 for initial and routine operations.

Table 9-3. Summary of the Number and Type of Innermost Layer of Confinement QC Headspace Samples Required for the First Two Bins Prepared at Each Site

First Two Bins	All Program Required Analytes, except NO_x
Field Blanks	1/day
Equipment Blanks[a]	1/day, prior to work
Field Reference Standards[b]	1/bin
Field Duplicates	1/bin or 1/20 samples collected, whichever is more frequent

[a] One innermost layer of confinement sampling equipment blank should be collected, analyzed, and demonstrated clean prior to first use (all analytes except NO_x), then at the specified frequency thereafter.

[b] One field reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Tables 3-5 and 3-6 prior to first use, then at the specified frequency thereafter.

Table 9-4. Summary of the Number and Type of Innermost Layer of Confinement QC Headspace Samples

Routine	All Program Required Analytes, except NO_x
Field Blanks	1/day
Equipment Blanks[a]	1/day prior to work
Field Reference Standards[b]	1/quarter annum
Field Duplicates	1/bin or 1/20 samples collected, whichever is more frequent

[a] One innermost layer of confinement sampling equipment blank should be collected, analyzed, and demonstrated clean prior to first use (all analytes except NO_x), then at the specified frequency thereafter.

[b] One field reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Tables 3-5 and 3-6 prior to first use, then at the specified frequency thereafter.

Field Blanks (FBs). FBs shall be used to evaluate background levels of the Program required analytes. FBs must be collected at a frequency of one per day. The site Project Manager shall use the FB data to assess its impact on the sample results.

Equipment Blanks (EBs). EBs shall be used to assess the cleanliness of the sampling equipment prior to first use of the direct canister sampler or the low internal volume manifold for sample collection. After the initial cleanliness check, EBs must be collected at a frequency of one per day, prior to sample collection, for all the Program required analyses. The site Project Manager shall use the EB data to assess its impact on the sample results and have the responsibility to ensure that corrective action measures are taken when EB data indicates the sampling equipment is contaminated.

Field Reference Standards (FRSs). FRSs shall be used to assess the accuracy with which the sampling equipment can collect the Program required analytes into SUMMA® canisters prior to first use of the sampling equipment. After the initial accuracy check, FRSs must be collected at a frequency of one per bin for the first two bins and quarterly thereafter. FRS results shall be acceptable if the QAOs specified in Tables 3-5 and 3-6 are met. 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 that corrective action measures are taken when the QAOs are not achieved.

Field Sample Duplicates (FSDs). Duplicate innermost layer of confinement samples must 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 one per twenty innermost layers of confinement whichever is more frequent. Field duplicate results shall be acceptable if the QAOs specified in Tables 3-5 and 3-6 are met. 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 that corrective action measures are taken when the QAOs are not achieved.

9.1.5 Bin Headspace Sampling

Quality control checks shall be accomplished in the field or in the laboratory (prior to shipment of the bin headspace sampling equipment to the field). Required bin QC sample frequencies are summarized in Tables 9-5 and 9-6 for initial and routine operations.

Equipment Blanks (EBs). EBs shall be used to assess the cleanliness of the bin sampling equipment prior to first use of the equipment for sample collection. After the initial cleanliness check, EBs must be collected at a frequency of one per bin, prior to sample collection. The site Project Manager shall

Table 9-5. Summary of the Number and Type of Bin QC Headspace Samples Required for the First Two Bins Prepared at Each Site

First Two Bins	All Program Required Analytes except NO_x
Equipment Blanks[a]	1/bin, prior to work
Reference Standards[b]	1/bin
Field Duplicates	1/bin

[a] One bin sampling equipment blank must be collected, analyzed, and demonstrated clean prior to first use (flame test and all analytes except NO_x), then at the specified frequency thereafter.

[b] One reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Tables 3-5 and 3-6 prior to first use (flame test and all analytes except NO_x), then at the specified frequency thereafter.

Table 9-6. Summary of the Number and Type of Bin QC Headspace Samples

Routine	All Program Required Analytes, except NO_x
Equipment Blanks[a]	1/bin
Reference Standards[b]	1/5 bins
Field Duplicates	1/5 bins

[a] One bin sampling equipment blank must be collected, analyzed, and demonstrated clean prior to first use (flame test and all analytes except NO_x), then at the specified frequency thereafter.

[b] One reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Tables 3-5 and 3-6 prior to first use (flame test and all analytes except NO_x), then at the specified frequency thereafter.

use the EBs data to assess its impact on the sample results and have the responsibility to ensure that corrective action measures are taken when EB data indicates the bin sampling equipment is contaminated.

Reference Standards (RSs). RSs shall be used to assess the accuracy with which the bin sampling equipment can collect headspace samples into SUMMA® canisters prior to first use of the bin sampling equipment for sample collection. After the initial accuracy check, RSs must be collected at a frequency of one per bin for the first two bins, then one per five bins thereafter. RS results shall be acceptable if the QAOs specified in Tables 3-5 and 3-6 are met. The site Project QA Officer shall have the responsibility to monitor and document RS results. The site Project Manager shall also have the responsibility to ensure that corrective action measures are taken when the QAOs are not achieved.

Field Sample Duplicates. Duplicate bin samples must 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 for the first two bins, then one per five bins thereafter. Field duplicate results shall be acceptable if the QAOs specified in Tables 3-5 and 3-6 are met. 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 that corrective action measure are taken when the QAOs are not achieved.

9.1.6 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 item, material (residual or packaging), or waste material category 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 in order 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 to be taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, procedure 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 to be taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, procedure proficiency samples, and blind audit samples. In addition, acceptable method performance must be demonstrated prior to the analysis of samples.

9.2.1.1 Initial and Continuing Procedure Demonstration

All laboratories using these procedures must demonstrate acceptable performance prior to the analysis of any samples. Demonstration of acceptable performance shall be achieved by analyzing commercially available gas standards and demonstrating acceptable performance in the Performance Demonstration Program. These standards must contain all of the analytes listed in Table 3-6 at concentrations appropriate to determine parameters specified in Table 3-6. Initially, 30 replicate standards shall be analyzed in order to demonstrate that the criteria specified for precision, accuracy and method detection limits, listed in Table 3-6, can be met. Demonstration of acceptable procedure performance shall be repeated semiannually by the analysis of seven replicates. Procedures for the evaluation of procedure performance data are given in Section 12. All procedure performance standards (replicates) must be at the same concentration.

It shall be the responsibility of the laboratory QA Officer to monitor and document procedure performance. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when acceptable procedure performance is not met.

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 3-6. The concentration of analytes must be in the linear range of the analytical instrument.

LCS results shall be acceptable if the criteria in Table 3-6 are satisfied. LCSs must be analyzed at a frequency of one per analytical batch. 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 the 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 3-6 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.

Equipment Blanks (EBs). EBs shall be collected by attaching a clean canister to the sampling equipment 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 or NO_x) listed in Table 3-6 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

The laboratory shall analyze field samples in duplicate at a minimum frequency of one per analytical batch. 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. 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 to be taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, procedure proficiency samples, and blind audit samples.

9.2.2.1 Initial and Continuing Procedure Demonstration

All laboratories analyzing samples for this program must demonstrate acceptable procedure 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 or laboratory-prepared standards and demonstrate acceptable performance in the Performance Demonstration Program. These standards must contain all of the analytes listed in Table 3-7 at concentrations appropriate to determine the parameters specified in Table 3-5. Initially, 30 replicate standards shall be analyzed in order to demonstrate that the criteria for precision, accuracy and method detection limits, listed in Table 3-5, can be met. Demonstration of acceptable procedure performance shall be repeated semiannually by the analysis of seven replicates. Procedures for the evaluation of procedure performance data are given in Section 12. All procedure performance standards (replicates) must be at the same concentration.

It shall be the responsibility of the laboratory QA Officer to monitor and document procedure performance. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when acceptable procedure 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 3-7. 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 3-5 are satisfied. LCSs must be analyzed at a frequency of one per analytical batch. 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.

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 or nitrogen. The site Project Manager must be notified if VOCs are detected at levels exceeding 50% of the PRQLs listed in Table 3-7. It shall be the responsibility of the site Project Manager to ensure that corrective action measures are taken.

Equipment Blanks (EBs). EBs shall be collected by attaching a clean SUMMA® canister to the sampling equipment and filling it with ultra-high purity air or nitrogen. The site Project Manager must be notified if VOCs are detected at levels exceeding 50% of the PRQLs listed in Table 3-7. It shall be the responsibility of the site Project Manager to ensure that corrective action measures are taken.

Laboratory Blanks (LBs). SUMMA® canisters must be filled with zero air or nitrogen 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 3-7 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.

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 5% 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 for the continuing calibration standards are within the range of 50% to 200% of the average internal standard area counts from the most recent five-point calibration, and for samples if the area counts are within 50% to 200% of the latest continuing calibration standards. 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.

9.2.3 Flammability Testing

Flammability testing provides a qualitative evaluation of whether or not bin headspace samples are flammable. Operator training and experience are critical for assuring the quality of flammability test results, and only trained personnel shall operate the flame test apparatus. Training requirements shall be specified in site QAPjPs and SOPs.

The samples of an analytical batch shall be analyzed by the same flame test operator who performed the initial calibration and continuing calibrations associated with that analytical batch. Calibration requirements and frequencies are summarized in Table 6-3. All field sample duplicates shall be analyzed. Laboratory duplicates shall be analyzed at a frequency of one per analytical batch. For flammability testing, an analytical batch shall consist of not more than 20 samples.

The 1.0 v/v% diethyl ether initial calibration standard is a functional blank, and no additional blanks are 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 WIPP 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 PDP (Section 1.11). DOE/WPO shall be responsible for administering the PDP.

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 determine the cause of the adverse finding and 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 the 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.

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 semi-annually. 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 analyzing 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 (USDOE, 1991a). 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 an item or activity shall be planned, executed, and documented. Preventive maintenance must have two aspects: (1) a schedule of preventive maintenance activities to ensure the accuracy of measurement systems 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, and MDL for the program are as follows.

12.1 Precision

Precision is 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:

$$RPD = \frac{C_1 - C_2}{(C_1 + C_2) / 2} * 100 \qquad (12\text{-}1)$$

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:

$$RSD = s / \bar{y} * 100 \qquad (12\text{-}2)$$

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

The standard deviation, s, is defined as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n - 1}} \qquad (12\text{-}3)$$

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:

$$\%R = \frac{C_m}{C_{srm}} * 100 \qquad (12\text{-}4)$$

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:

$$\%R = \frac{S - U}{C_{sa}} * 100 \qquad (12\text{-}5)$$

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:

$$\%C = \frac{V}{n} * 100 \qquad (12\text{-}6)$$

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:

$$Comparability = \frac{CC}{TD} * 100 \qquad (12\text{-}7)$$

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:

$$MDL = t_{(\eta-1,1-\alpha=.99)} * s \qquad (12\text{-}8)$$

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 (Glaser, 1981).

12.6 Procedures for Assessing Compliance With Data Quality Objectives and Regulatory Compliance Limits

Statistical procedures are needed to assess compliance with NMD requirements for potential headspace gas flammability, waste comparability, and no-migration finding. These procedures must be applied to laboratory analytical data prior to comparing these data with the regulatory limits specified in Section 1.3.2.

12.6.1 Statistical Procedure for Assessing Compliance With NMD Requirements

The statistical procedure that must be used to assess compliance with regulatory limits established in the NMD is based on the following:

- Laboratory precision and accuracy data resulting from procedure performance testing
- Normal distribution of laboratory procedure performance data (population standard deviation (σ) is approximately equivalent to the sample standard deviation (s) for 30 laboratory procedure performance samples)
- Homogeneous nature of the sample matrix
- Majority of the quantifiable sampling and measurement error due to laboratory error.

Upper confidence limit values (concentration or LEL) are calculated using the following equations:

$$X_{UL} = \Sigma\, a_i\, X_{ci} + \Sigma\, a_i\, S_i\, Z_{\alpha=0.1} \qquad (12\text{-}9)$$

$$X_{ci} = \frac{100\, X_{mci}}{\%R_i} \qquad (12\text{-}10)$$

$$S_i = \frac{(\%RSD_i)\, X_{mci}}{\%R_i} \qquad (12\text{-}11)$$

where

X_{UL}	=	90% upper confidence limit value of the measured concentrations
a_i	=	Independent variable (for flammability calculations, this is the reciprocal of the LEL; for comparability and no-migration finding, the variable is one)
X_{ci}	=	Measured concentration of compound i, corrected for recovery
S_i	=	Standard deviation of compound i
$Z_{\alpha=0.10}$	=	Standard normal deviate (Z-value) at 90% confidence level (one-tail) is 1.29
$\%RSD_i$	=	Percent relative standard deviation of compound i (obtained from laboratory procedure performance data)
$\%R_i$	=	Percent recovery of compound i (obtained from laboratory procedure data)
X_{mci}	=	Experimentally measured concentration of compound i.

Equation 12-9 must be used to determine compliance with the NMD waste characterization requirements. Upper confidence limit values shall be calculated using Equation 12-9 and compared to

regulatory limits as described in Sections 1.3 and 8.7. A value of one-half the MDL must be used when flammable VOCs are not detected.

12.6.2 Procedure for Obtaining Precision and Accuracy Data Used in Determining X_{UL}

The overall approach to evaluating procedure performance data is shown in Figure 12-1. Initially, precision and accuracy shall be assessed using procedure performance data obtained from the analysis of 30 laboratory reference standards (initial laboratory procedure demonstration). The precision and accuracy obtained from this initial data set shall be used to determine X_{UL}. Precision and accuracy data for all procedures shall be continuously monitored by the analysis of seven replicates every six months.

The precision and accuracy obtained from the first continuing data set must be compared with the initial procedure performance data set. Comparison of the initial and continuing data sets must be made at the 95% confidence level using established procedures (Taylor, 1987; Dowdy, 1983), as described below. If no statistical differences exist between the initial and continuing data sets, the two sets of precision and accuracy data shall be pooled. The precision and accuracy from the pooled data shall then be used to determine X_{UL}. Subsequent continuing data sets must be statistically compared to the most recent pooled data set and the two sets combined to form a new pooled data set if no statistical difference exists.

If a significant statistical difference does exist at the 95% confidence level between the initial or pooled and the continuing data sets, another set of seven replicates must be analyzed. If no statistical difference exists at the 95% confidence level between the two sets of seven replicates, the precision and accuracy of these two seven-replicate sets shall be pooled. The precision and accuracy from this pooled data shall be used to determine X_{UL}. Corrective actions must be taken if two consecutive data sets of seven replicates are not statistically equivalent.

The process of analyzing seven replicates and comparing the resulting precision and accuracy data to the most recent pooled data must be repeated semiannually. If the resulting pooled data set has less than 30 samples, the impact on calculating X_{UL} values must be evaluated. Procedures should be implemented to ensure that the pooled data set contains only data generated within a two-year period.

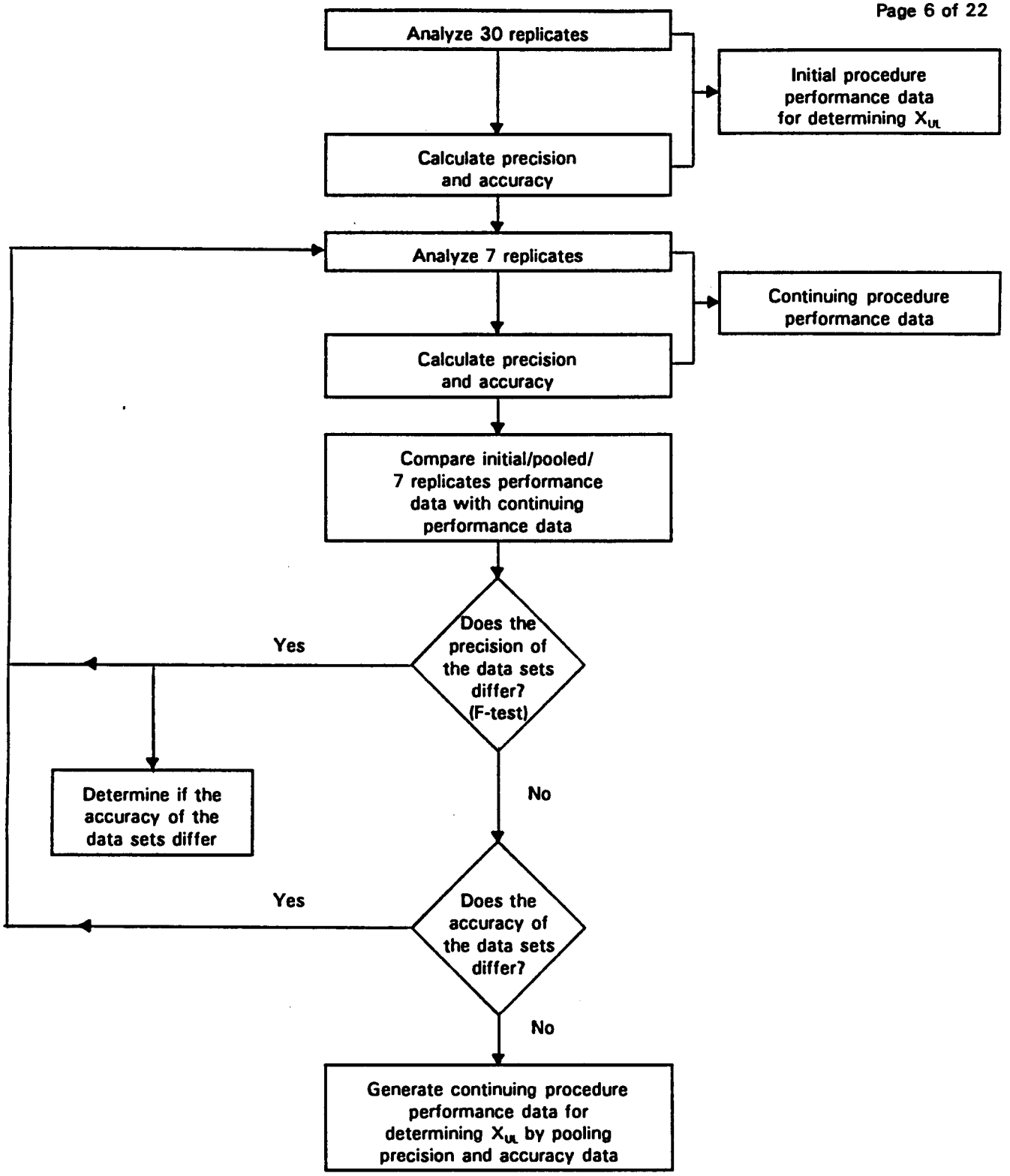


Figure 12-1. Statistical Analysis of Procedure Performance Data

12.6.2.1 Analysis of Precision

The F-test shall be used to determine if the precisions obtained from different data sets are statistically the same. Comparison of data sets shall be done at the 95% confidence level (two-tailed). A calculated F-value is determined by taking the ratio of the two data set variances:

$$F_{v1,\,v2} = s_1^2 \,/\, s_2^2 \qquad (12\text{-}12)$$

where

v_1 = number of degrees of freedom for data set 1

v_2 = number of degrees of freedom for data set 2

s_1 = standard deviation of data set 1

s_2 = standard deviation of data set 2.

When calculating F values, s_1 and s_2 must be chosen so that s_1 is greater than s_2, and therefore s_1^2/s_2^2 is always greater than one. This calculated F value is then compared to the critical F_c value found in tables of critical F values (Abramowitz, 1972). The F_c value must be obtained at the 95% confidence level with the degrees of freedom (v_1,v_2) such that v_1 is degrees of freedom for the numerator and v_2 is the degrees of freedom of the denominator. The precision obtained from two data sets is statistically equivalent if the calculated F value is less than the critical F_c value.

12.6.2.2 Analysis of Accuracy

The accuracy obtained from two data sets shall be evaluated at the 95% confidence level (two-tailed) by comparing the data set average using the t-statistic. If the standard deviations of two data sets are statistically equivalent by the F-test, a pooled estimate of the standard deviation can be calculated as follows:

$$s^2 = [(n_1 - 1)\, s_1^2 + (n_2 - 1)\, s_2^2\,]/(n_1 + n_2 - 2) \qquad (12\text{-}13)$$

where

n_1 = number of samples in data set 1

n_2 = number of samples in data set 2.

The t value is given by:

$$t_v = (\bar{X}_1 - \bar{X}_2) / [s\,(1/n_1 + 1/n_2)^{1/2}] \qquad (12\text{-}14)$$

where

v = number of degrees of freedom ($n_1 + n_2 - 2$)

$\bar{X}_1$ = mean concentration of data set 1

$\bar{X}_2$ = mean concentration of data set 2.

If the standard deviations of the two data sets are significantly different, the t value is given by:

$$t_v = (\bar{X}_1 - \bar{X}_2) / \left[\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right]^{1/2} \qquad (12\text{-}15)$$

With the degrees of freedom, v, given as:

$$v = \left[\frac{\left[\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right]^2}{\frac{(s_1^2/n_1)^2}{n_1 + 1} + \frac{(s_2^2/n_2)^2}{n_2 + 1}}\right] - 2 \qquad (12\text{-}16)$$

For either case, the mean concentrations of the two data sets are not significantly different if the t value obtained is less than the critical value of t_c (Abramowitz, 1972; Taylor, 1987; Dowdy, 1983). If the mean concentrations do not differ significantly, then the accuracies for the two data sets do not differ.

12.6.2.3 Procedures to Obtain Pooled Data Sets

Estimates of the standard deviation shall be pooled using the following equation:

$$s_{pooled} = \left[\frac{v_1 s_1^2 + v_2 s_2^2}{v_1 + v_2}\right]^{1/2} \qquad (12\text{-}17)$$

Mean values shall be pooled using the following equation:

$$\bar{X}_{pooled} = \frac{n_1\bar{X}_1 + n_2\bar{X}_2}{n_1 + n_2} \qquad (12\text{-}18)$$

12.6.2.4 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 of all values exclusive of the suspect result.

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

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 12-1.

12.6.2.5 Procedures for Evaluating NMD Flammability Quality Assurance Objectives

This section describes the procedure that must be used to demonstrate compliance with the NMD requirement that all flammability testing meets the quality assurance objectives (QAOs) of ±10% precision and accuracy. This requirement must be met to demonstrate compliance with QAOs for flammable VOCs as a class.

The following procedure must be followed:

1. Obtain 30 sets of analytical data for flammable VOCs, as described in Section 12.6.2.
2. Sum the measured concentrations of each of the flammable VOCs to yield 30 values for the sum of flammable VOCs.
3. Average the results of the 30 summations from step 2.
4. Sum the concentrations of the flammable VOC standards that are obtained for the same set of flammable VOC samples.
5. To determine the accuracy, divide the average sum of measured flammable VOCs by the sum of the standard or known concentrations of flammable VOCs.
6. To determine the precision, calculate the relative standard deviation of the 30 values for the sum of flammable VOCs using Equation 12-22.

Table 12-1. 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

7. This procedure must be applied to the seven sets of analytical data collected semiannually, as described in Section 12.6.2.

Figure 12-2 provides an example of how the data should be reported. Equations 12-19 through 12-22 are used to calculate the required information related to the performance data using the 30 replicates for each flammable VOC.

$$T_i = \sum_{j=1}^{m} X_{i,j} \tag{12-19}$$

$$\bar{T} = \frac{\sum_{i}^{n} T_i}{n} \tag{12-20}$$

$$KT = \sum_{j=1}^{m} K_j \tag{12-21}$$

$$S_T = \sqrt{\frac{\sum_{i=1}^{n} (T_i - \bar{T})^2}{n - 1}} \tag{12-22}$$

where:

$X_{i,j}$ = Measured concentration (ppmv) of the ith determination of flammable VOC "j"

T_i = Total measured flammable VOC concentration (ppmv) of the ith determination

$\bar{T}$ = Average of the total measured flammable VOC concentration (ppmv) for all determinations

K_j = Known concentration of flammable VOC "j"

KT = Total of the known flammable VOC concentrations (ppmv)

S_T = Standard deviations of the total measured flammable VOC concentrations for all "i" determinations.

Assume there are "m" different, flammable VOCs and there a total of "n" analyses performed of each standard.

To meet accuracy QAO, the following must be demonstrated:

Flammable Analytes (j)	Determination Number (i)				Avg	Std Dev (S_j)	Known Value (K_j)
	1	2	3	4... \ \ n			
1	$X_{1,1}$	$X_{2,1}$	$X_{3,1}$	$X_{4,1}$...\ \ $X_{n,1}$	$\overline{X}_1$	S_1	K_1
2	$X_{1,2}$	$X_{2,2}$	$X_{3,2}$	$X_{4,2}$...\ \ $X_{n,2}$	$\overline{X}_2$	S_2	K_2
3	$X_{1,3}$	$X_{2,3}$	$X_{3,3}$	$X_{4,3}$...\ \ $X_{n,3}$	$\overline{X}_3$	S_3	K_3
4	$X_{1,4}$	$X_{2,4}$	$X_{3,4}$	$X_{4,4}$...\ \ $X_{n,4}$	$\overline{X}_4$	S_4	K_4
\	\	\	\	\	\	\	\
\	\	\	\	\	\	\	\
m	$X_{1,m}$	$X_{2,m}$	$X_{3,m}$	$X_{4,m}$...\ \ $X_{n,m}$	$\overline{X}_m$	S_m	K_m
SUMS:	T_1	T_2	T_3	T_4 ...\ \ T_n	$\overline{T}$	S_T	KT

Figure 12-2. Calculation Method for Evaluating Performance Data In Relation to QAOs for Flammability Testing

$$0.90 \leq \frac{\bar{T}}{KT} \leq 1.10 \qquad (12\text{-}23)$$

To meet precision QAO, the following must be demonstrated:

$$\frac{S_T}{\bar{T}} \leq 0.10 \qquad (12\text{-}24)$$

12.6.3 Procedures for Determining Emplacement Time During Which H_2/CH_4 Remain Below 0.5 LEL in Experimental Bins

The emplacement time during which H_2/CH_4 concentrations remain below 0.5 LEL levels inside a bin is calculated iteratively using the numerical solutions to the differential equations which describe the mass balances on H_2/CH_4 within each confinement volume (bin, SWB or TRUPACT-II) prior to emplacement in the WIPP. The analysis to determine the maximum emplacement times is similar to the analysis presented in the TRUPACT-II Safety Analysis Report (SAR) for determining aspiration times (NuPac, 1989). The emplacement times derived in this section refer to compliance with the NMD criteria, and do not indicate that the H_2/CH_4 concentrations exceed flammable levels beyond these times. In fact, the conservative analysis used in deriving the decay heat limits in the TRUPACT-II SAR ensure that waste containers are safe (i.e., less than H_2/CH_4 LEL) at all times with respect to H_2/CH_4 concentrations. Two sets of differential equations have been solved numerically for the following configurations:

1. An experimental bin inside a standard waste box (SWB) venting to the atmosphere at the bin preparation site. This represents the time the bin is in interim storage at the DOE generator/storage site.

2. Two experimental bins each inside a standard waste box which are present inside the Inner Containment Vessel (ICV) of a TRUPACT-II. This represents the time the bin is being transported.

The following equations describe mass balance of H_2/CH_4 for bins in interim storage:

$$dX_{hdspc}/dt = C_1 - C_2 (X_{hdspc} - X_{bin}) \qquad (12\text{-}25)$$

$$dX_{bin}/dt = C_3(X_{hdspc} - X_{bin}) - C_4(X_{bin} - X_{SWB}) \qquad (12\text{-}26)$$

$$dX_{SWB}/dt = C_5(X_{bin} - X_{SWB}) - C_6X_{SWB} \qquad (12\text{-}27)$$

The following equations describe mass balance of H_2/CH_4 during transport in TRUPACT-II:

$$dX_{hdspc}/dt = C_1 - C_2(X_{hdspc} - X_{bin}) \quad (12\text{-}28)$$

$$dX_{bin}/dt = C_3(X_{hdspc} - X_{bin}) - C_4(X_{bin} - X_{SWB}) \quad (12\text{-}29)$$

$$dX_{SWB}/dt = C_5(X_{bin} - X_{SWB}) - C_6(X_{SWB} - X_{TRU}) \quad (12\text{-}30)$$

$$dX_{TRU}/dt = C_7(X_{SWB} - X_{TRU}) \quad (12\text{-}31)$$

where

C_1 = $CG \times R \times T/(P \times V_1)$

X_{hdspc} = mole fraction H_2/CH_4 in the void volume within the two bin liners (i.e., the bin headspace)

X_{bin} = mole fraction H_2/CH_4 in the annular volume between the bin liners and the bin

X_{SWB} = mole fraction H_2/CH_4 in the SWB void volume outside the experimental bin

X_{TRU} = mole fraction H_2/CH_4 in the TRUPACT-II ICV outside the SWBs

C_2 = The effective release rate of H_2/CH_4 across the two bin liners in series divided by the void volume within the bin liners (day^{-1})

C_3 = The effective release rate of H_2/CH_4 across the two bin liners in series divided by the void volume outside of the bin liners (i.e., the volume between the bin and the liners) (day^{-1})

C_4 = The effective release rate of H_2/CH_4 across two bin filters in parallel divided by the void volume outside of the bin liners (day^{-1})

C_5 = The effective release rate of H_2/CH_4 across two bin filters in parallel divided by the SWB void volume outside the experimental bin (i.e., the volume between the bin and the SWB) (day^{-1})

C_6 = The effective release rate of H_2/CH_4 across two SWB filters in parallel divided by the SWB void volume outside the experimental bin (day^{-1})

C_7 = The effective release rate of H_2/CH_4 across four (two from each SWB) SWB filters in parallel divided by the TRUPACT-II ICV void volume outside the SWBs (day^{-1})

t = Time (day)

CG = The allowable flammable gas generation rate per experimental bin (mole/day)

R = The gas law constant (0.08206 atm liter/mole °K)

T = Absolute temperature (294 °K) (NuPac, 1989)

P = Absolute pressure (1 atm) (NuPac, 1989)

V_1 = The void volume within the bin liners (liter).

These systems of differential equations were solved numerically using the Runge-Kutta Fourth Order numerical integration method (Perry et al., 1984; Reklaitis, et al., 1983). Numerical solution implies

obtaining the mole fractions of H_2/CH_4 in each confinement volume as a function of time and by waste type.

12.6.3.1 Input Parameters

Several parameters determine the maximum allowable emplacement times for each of the waste types. The values of the parameters are identical to those used in the TRUPACT-II SAR (NuPac, 1989). These parameters are pressure, temperature, allowable H_2/CH_4 generation rates, H_2/CH_4 release rates across layers of confinement, void volumes, and transportation time inside the TRUPACT-II.

Shipping Period Duration Inside TRUPACT-II

The TRUPACT-II SAR shipping Package (NuPac, 1989) provides an analysis of transit times which indicates that "The normal transit time ranges from 0.6 day for shipments from Rocky Flats Plant to 1.7 days for shipments from Hanford." For the purpose of conservatism, three days is assumed for a maximum normal transit time. In determining the emplacement times, a transit period of three days has been used in the calculations.

Flammable Gas Release Rates

The release rates of H_2/CH_4 across each confinement layer are based on the lowest measured release rates (NuPac, 1989) and provide a margin of safety by predicting the smallest allowable emplacement times.

Void Volumes for Waste Types

The void volumes used in the calculations were based on the data from NuPac (1989), and Molecke (1990).

H_2/CH_4 Generation Rates

The allowable H_2/CH_4 generation rates used in the calculations are the bounding values for each waste type, and are dependent on the packaging configurations within the waste containers (NuPac, 1989). These gas generation rates are the bounding values used to determine the decay heat limits in the TRUPACT-II SAR, and ensure that the gas concentrations in the bins remain below flammable levels at all times (NuPac, 1989). The emplacement times derived in this section refer to compliance with the NMD criteria, and are not meant to imply that gas concentrations exceed flammable levels beyond these times. In fact, the conservative analysis used in deriving the decay heat limits in the TRUPACT-II SAR ensure that waste containers are safe at all times with respect to H_2/CH_4 concentrations.

12.6.3.2 Tables of Allowable Emplacement Times

Two options are available to determine the emplacement time during which $H2/CH_4$ will remain below 50% of the mixture LEL. The first option is based on the length of time which has elapsed since the bin was prepared. Table 12-2 summarizes the emplacement times at the time of bin preparation for the various shipping categories. The second option is based on sampling the bin headspace for H_2/CH_4. The emplacement times for use with this option are summarized in Tables 12-3 through 12-7. It should be noted that the transportation time has been included in these emplacement times.

Table 12-2. Allowable Emplacement Times Based on the Date of Bin Preparation[a]

Waste Type	Shipping Category	Emplacement Time (Days)
I	I.1D2, I.2D2, I.3D2	123
II	II.1D2	152
III	III.1D2	140
III	III.1D5	160
IV	IV.1D2	125

[a] This option and the corresponding times should be used if no sampling of headspace gases is to be performed after bin preparation.

Table 12-3. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type I, Shipping Categories I.1D2, I.2D2, I.3D2

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	123
0.1	119
0.2	114
0.3	110
0.4	105
0.5	100
0.6	95
0.7	90
0.8	84
0.9	78
1.0	73
1.1	66
1.2	60
1.3	54
1.4	47
1.5	39
1.6	32
1.7	24
1.8	16
1.9	8

Table 12-4. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type II, Shipping Category II.1D2

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	152
0.1	147
0.2	142
0.3	136
0.4	130
0.5	124
0.6	118
0.7	112
0.8	105
0.9	99
1.0	91
1.1	84
1.2	76
1.3	68
1.4	60
1.5	51
1.6	41
1.7	31
1.8	21
1.9	11

Table 12-5. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type III, Shipping Category III.1D2

Mole Percent H_2/CH_4 Gas	Emplacement Time (Days)
0.0	140
0.1	135
0.2	130
0.3	125
0.4	120
0.5	115
0.6	109
0.7	103
0.8	97
0.9	91
1.0	85
1.1	78
1.2	71
1.3	63
1.4	55
1.5	47
1.6	38
1.7	29
1.8	20
1.9	10

Table 12-6. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type III, Shipping Category III.1D5

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	160
0.1	154
0.2	147
0.3	141
0.4	134
0.5	127
0.6	120
0.7	113
0.8	106
0.9	98
1.0	91
1.1	83
1.2	75
1.3	66
1.4	57
1.5	48
1.6	39
1.7	30
1.8	20
1.9	9

Table 12-7. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type IV, Shipping Category IV.1D2

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	125
0.1	121
0.2	116
0.3	111
0.4	107
0.5	101
0.6	96
0.7	91
0.8	85
0.9	80
1.0	74
1.1	67
1.2	61
1.3	54
1.4	47
1.5	40
1.6	32
1.7	24
1.8	16
1.9	7

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 the 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 memoranda, 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. Completion of the corrective action for 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 PDP 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 - Pressure measured relative to absolute 0 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 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.

BINARY MIXTURE - A mixture of hydrogen and methane in air.

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

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

COMPLETENESS - The percentage of measurements made 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 (e.g., concentration and 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 DOE goals.

EMPLACEMENT - When the bin instrumentation is connected to the Radiological Control Barrier as required for conducting the bin-scale tests.

EMPLACEMENT ACTIVITIES - Those activities associated with waste handling within the Salado Formation (i.e., within the unit boundary as defined in the NMD).

EMPLACEMENT TIME - The time from when the waste is loaded in a bin and the bin is closed and placed in a standard waste box through emplacement in the WIPP underground without exceeding 50% of the LEL for the binary mixture of H_2/CH_4.

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.

FLAME TEST - The procedure specified in Guidance Manual Procedure 810.1 for determining the flammability of headspace samples containing concentrations of flammable VOCs greater than 500 ppmv.

FLAMMABLE MIXTURE - (1) A binary mixture with concentrations of hydrogen and methane exceeding 50% of the theoretical LEL, or (2) a mixture of flammable VOCs in air that propagates a flame during a flame test.

FLAMMABLE VOC - A VOC identified as noncombustible or considered, by EPA, not to be a significant fire hazard under WIPP repository conditions. Flammable VOCs that are evaluated for the Program are listed in Table 1-5.

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.

GASES - The analytes listed in Table 3-6 or the Program-required subset of the Table 3-6 analytes.

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.

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

HOLDING TIME - The maximum permissible time allowed between time of sample collection and time of analysis.

INDEPENDENT STANDARD - Laboratory prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

INNERMOST LAYER OF CONFINEMENT - Within a waste container, a plastic bag that is closest to waste that may be a source of VOCs and/or hydrogen and methane and has a minimum of one liter of headspace.

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

INSTRUMENT DETECTION LIMIT (IDL) - The minimum signal that the instrument can detect with 99% confidence that the analyte concentration is greater than zero. Determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INTERFERENTS - Substances which affect the analysis for the element or compound of interest.

ITEM DESCRIPTION CODE - A site-specific, internal numerical code applied to individual waste forms to provide identification which is used for physical segregation and computerized recordkeeping and tracking.

LABORATORY BLANK - An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sampling processing. The laboratory blank is used to document contamination resulting from the laboratory sample preparation and analytical process.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition. Gas laboratory control samples are analyzed using the same analytical methods employed for the DOE-WIPP samples received.

LABORATORY 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.

LABORATORY RECEIPT DATE - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

LE CHATELIER FORMULA - A simple formula of additive character that connects the lower explosive limits of two or more fuels (flammable gases and/or VOCs) with the lower explosive limit of any mixture of them.

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the analytical curve remains linear.

LOWER EXPLOSIVE LIMIT - The lowest concentration of a fuel (flammable gas and/or VOC) in air that will propagate a flame from an ignition source. The lower explosive limit (LEL) is synonymous with the

lower limit of flammability (LLF). LEL values are typically expressed as the volume of fuel per volume of air (v/v%).

MANAGEMENT APPRAISAL - A determination of managerial effectiveness in establishing and implementing quality assurance program plans which conform to Departmental policy requirements. It is based on an analysis of functional appraisals, internal audits, and other information, and on the application of appropriate criteria. It is a review and evaluation of management performance covering all quality assurance and management responsibilities to assure proper quality assurance program balance.

METHOD BLANK - An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank must be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

METHOD DETECTION LIMIT (MDL) - The minimum concentration of a substance that can be measured and reported for a given method with 99% confidence that the analyte concentration is greater than zero. MDL is determined from analysis of a sample in a given matrix type containing the analyte of interest.

NARRATIVE (Case Narrative) - Portion of the data package which includes laboratory, contract, Case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete Case Narrative specifications are included in Exhibit B.

OUT OF CONTROL - One or more of several conditions relating to the plotting of control data and indicating unacceptable results.

PACKAGING MATERIAL - Flexible containment materials, e.g., plastic bags.

PERCENT DIFFERENCE (%D) - The difference between the average initial calibration response factor and the continuing calibration response factors divided by the average initial calibration response.

PERFORMANCE ASSESSMENT - A determination of the long-term performance of the WIPP disposal system in accordance with the requirements of the EPA Standard, 40 CFR 191, Subpart B.

PERFORMANCE EVALUATION (PE) SAMPLE - A sample of known composition provided as a single-blind sample to the analytical laboratory. Used by DOE to evaluate analytical laboratory performance.

PRECISION - A measure of mutual agreement among individual measurements of the same property, made under prescribed similar conditions; often expressed in terms of a standard deviation.

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

PROGRAM REQUIRED DETECTION LIMIT (PRDL) - Minimum level of detection acceptable under the WIPP-QAPP.

PROGRAM REQUIRED QUANTITATION LIMIT (PRQL) - Minimum level of analyte quantitation acceptable under the WIPP-QAPP. An analyte PRQL should be a minimum of three times the MDL.

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 the use of 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, defensible, 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) - The 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 - 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. Usually expressed as a percent.

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 or packaging material.

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.

TARGET COMPOUNDS - Those gases and VOCs identified by the Program as analytes. Gas target compounds are listed in Table 3-6; VOC target compounds in Table 3-7.

VOLATILE ORGANIC COMPOUNDS - The target compounds listed in Table 3-7 and any additional compounds tentatively identified by the VOC analytical procedures used to satisfy Program requirements, or Program-required subset of the Table 3-7 analytes.

WASTE CONTAINER - A disposable containment vessel for waste materials including integral liner or shielding materials intended for emplacement at the WIPP (i.e., 55-gallon waste drums or standard waste boxes).

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

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

WASTE TYPE - The classification system describing the physical types of waste [i.e., solidified inorganics (Waste Type I), solid inorganics (Waste Type II), solidified organics (Waste Type III), and solid organics (Waste Type IV) as established by the TRUPACT-II Safety Analysis Report.

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 Albuquerque Operations Office
DOE/WPO	DOE WIPP Project Office
DOT	Department of Transportation
DQO	Data Quality Objective
EB	Equipment Blank
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	Associate 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 Blank
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 Blank
LCS	Laboratory Control Sample
LEL	Lower Explosive Limit
MDL	Method Detection Limit
MMDDYY	Month-Day-Year Format

MS	Mass Spectrometry
NDA	Nondestructive Assay
NEIC	National Enforcement Investigation Center
NFPA	National Fire Protection Association
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
PDP	Performance Demonstration Program
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
TRU	Transuranic
TRUCON	TRUPACT-II Content Codes
TRUPACT-II	Transuranic Package Transporter
VOC	Volatile Organic Compound
VTSR	Validated Time of Sample Receipt
WAC	Waste Acceptance Criteria
WACCC	Waste Acceptance Criteria Certification Committee
WIPP	Waste Isolation Pilot Plant

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-- FINAL DRAFT --

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

Revision 2

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

October 12, 1992

Approved by _____ Date _____
Deputy Assistant Secretary, Office of Waste Management (EM-30)

Approved by _____ Date _____
Director, Office of Waste Management Projects (EM-34)

Approved by _____ Date _____
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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) and satisfies all applicable requirements of DOE Order 5700.6C, Quality Assurance (USDOE, 1991). 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 experimental-waste characterization 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

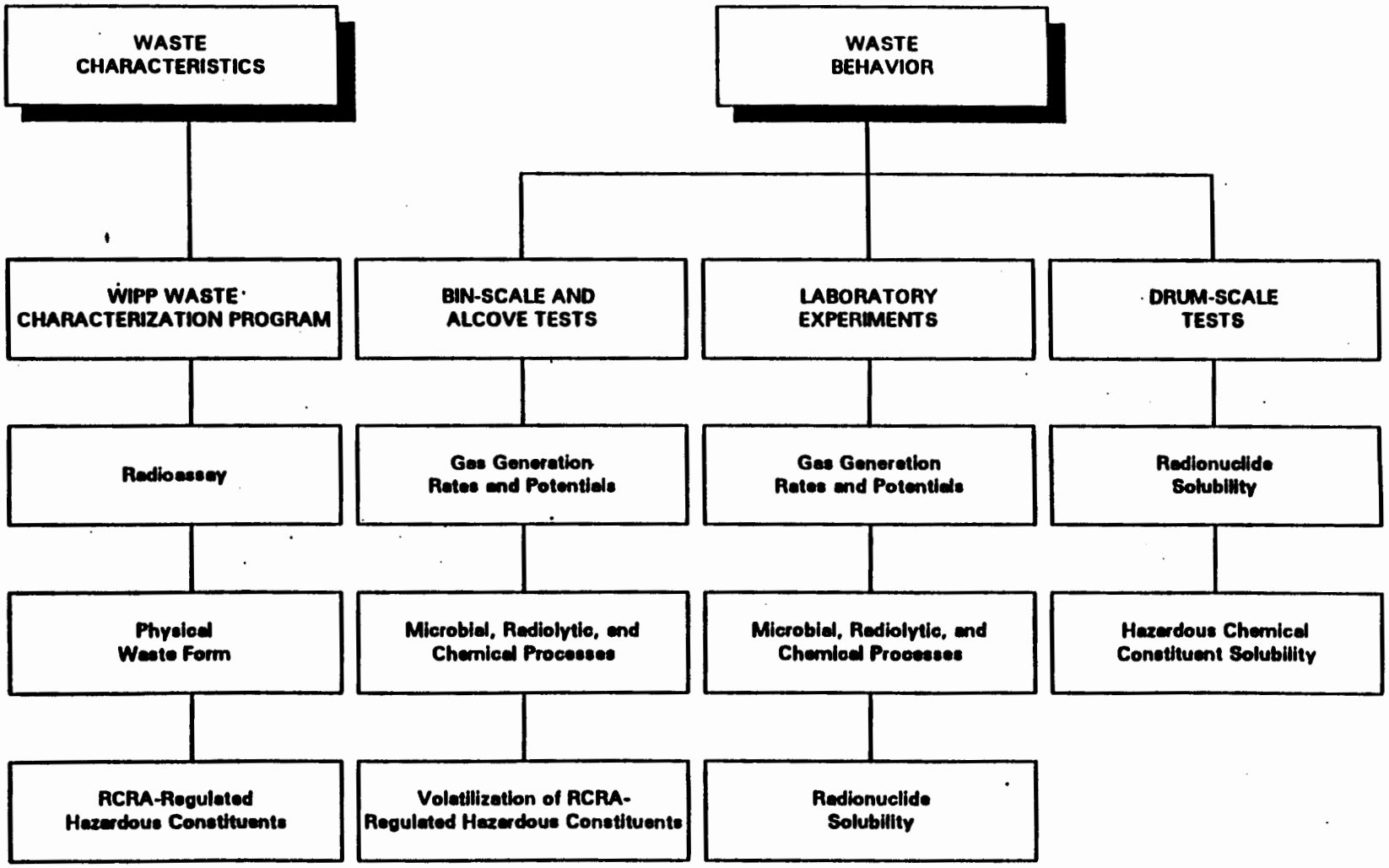


Figure 1-1. Aspects of WIPP Activities Related to Waste Characterization and Test Phase Programs

- 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 5700.6C (USDOE, 1991) establishes quality assurance requirements to ensure that risks and environmental impacts are minimized and that safety, reliability, and performance are maximized through the application of effective management systems commensurate with the risks posed by the facility and its work. Programs developed and properly implemented using ASME NQA-1 meet the majority of the requirements of this Order. DOE Order 5820.2A (USDOE, 1988a) requires that DOE facilities managing nuclear materials comply with all applicable quality elements in ASME NQA-1. For the purposes of this document, "Element" refers to the specified Basic Requirement and its Supplement(s). 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 DOE Order 5700.6C criteria and 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. Since this QAPP was developed using ASME NQA-1, it meets the majority of the requirements of DOE Order 5700.6C. Only those aspects of the ten criteria of DOE Order 5700.6C not addressed are ASME NQA-1 elements that will be identified in the appropriate sections of this QAPP to assist in the review for compliance with DOE Order 5700.6C.

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. Director of the WIPP Project Integration Office (DOE/WPIO) shall immediately be notified of any conflicts between the document and any existing requirements.

Program 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 procedures 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

TABLE 1-1. CROSS REFERENCE FOR DOE ORDER 5700.6C CRITERIA TO ASME NQA-1 ELEMENTS

DOE Order 5700.6C Criteria	ASME NQA-1 Elements	Section of the QAPP
MANAGEMENT		
1 Program	1 Organization 2 Quality Assurance Program	2 Organization and Responsibility 1 Program Description
2 Personnel Training and Qualification	2 Quality Assurance Program	1.8 Indoctrination and Training
3 Quality Improvement	15 Control of Nonconforming Items 16 Corrective Action	1.13 Quality Improvement 13 Corrective Actions 14 Quality Assurance Reports to Management
4 Documents and Records	6 Document Control 17 Quality Assurance Records	1.5 Document Review, Approval, and Control 1.6 Quality Assurance Records 1.9 Site Quality Assurance Project Plans 8 Data Reduction, Validation, and Reporting
PERFORMANCE		
5 Work Processes	5 Instructions, Procedures, and Drawing 8 Identification and Control of Items 9 Control of Processes 12 Control of Measuring and Test Equipment 13 Handling, Storage, and Shipping	1.7 Procurement Document Control/Control of Subcontractors 1.12 Control of Processes 4 Sampling Procedures 5 Sample Custody 6 Calibration Procedures and Frequencies 7 Analytical Procedures 9 Internal QC Checks and Frequency 11 Preventative Maintenance
6 Design	3 Design Control	3 Quality Assurance Objectives
7 Procurement	4 Procurement Document Control 7 Control of Purchase Items and Services	1.7 Procurement Document Control/Control of Subcontractors
8 Inspection and Acceptance Testing	10 Inspection 11 Test Control 14 Inspection, Test, and Operating Status	1.11 Performance Demonstration Program 6 Calibration Procedures and Frequencies 7.2 Radioassay (software) 9 Internal QC Checks and Frequency 11 Preventative Maintenance 12 Specific and Routine Procedures to Asses Data Quality
ASSESSMENT		
9 Management Assessment	2 Quality Assurance Program	1.14 Management Assessment 14 Quality Assurance Reports to Management
10 Independent Assessment	18 Audits	10 Performance and System Audits and Frequency

requirements for a given task. If a program participant chooses to use other procedures, the burden of proof of the efficacy of the procedures 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. The final results from the WIPP test program are expected in 3 to 5 years after 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 EPA's conditional no-migration determination (NMD) (55 FR 47700), and includes quantification of hazardous constituents identified through the Program to support both compliance with the determination as well as future efforts by DOE to petition EPA for a long-term no-migration determination after completion of the Test Phase (Figure 1-2).

- *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.

From a technical perspective, then, this QAPP addresses the activities associated with TRU waste characterization efforts in general, and applies to all such activities conducted under the Program. All current 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.

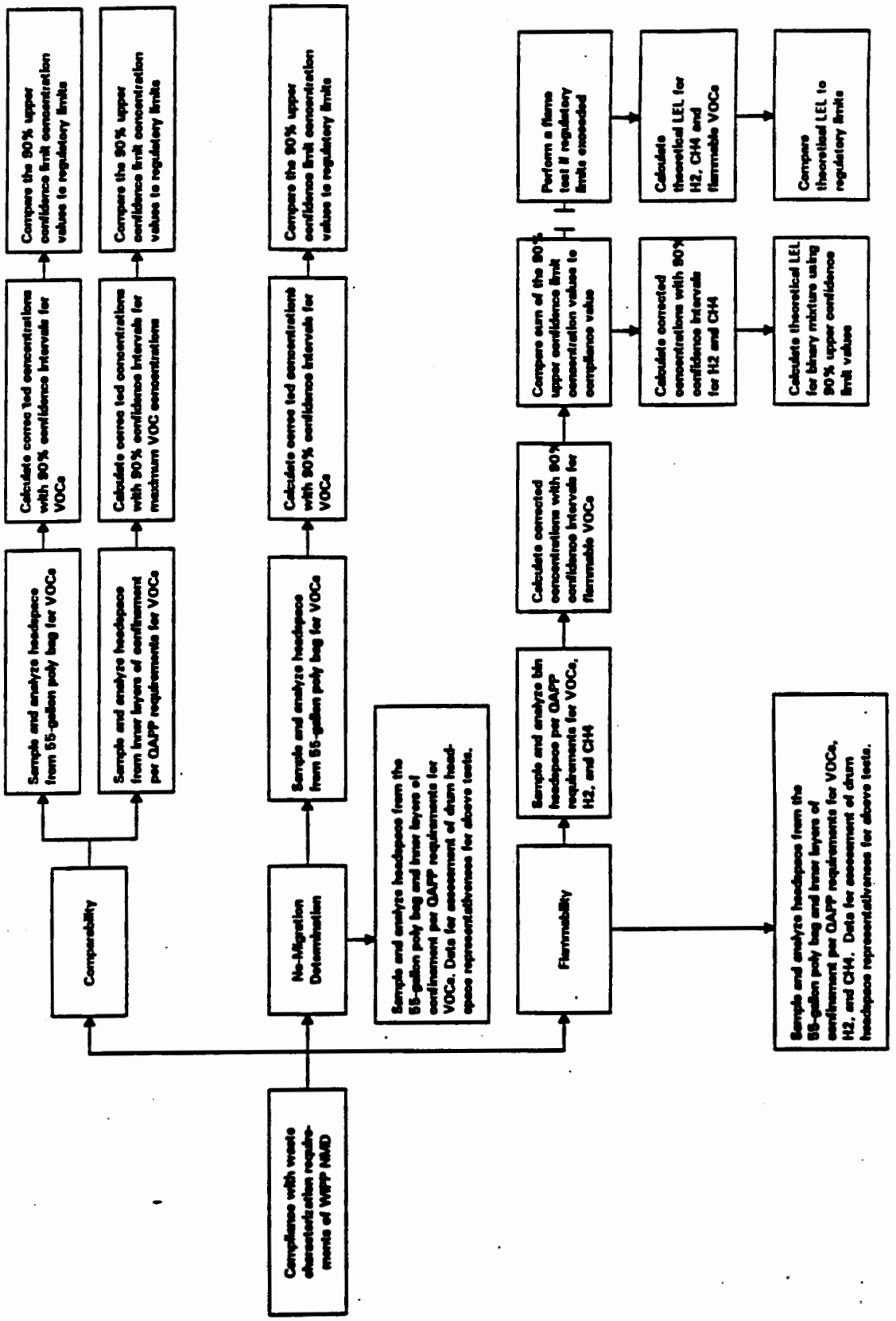


Figure 1-2. Overall Approach to Compliance With WIPP No-Migration Determination

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, E.N. Lorusso, and R.L. Hunter, 1991, "Rationale for Revised WIPP Bin-Scale Gas-Generation Tests with CH TRU Wastes at the Waste Isolation Pilot Plant," SAND90-2481, Sandia National Laboratories, Albuquerque, New Mexico.
- Department of Energy, 1991, "Waste Characterization Program Plan for WIPP Experimental Waste," DOE/WIPP 89-025, Current Revision, 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. A summary of the waste characterization requirements and the Program design is provided in Table 1-2.

1.3.1 Bin-Scale and Alcove Test Programs

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 a description of 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 containers of waste in accordance with SNL requirements from their inventory of TRU waste that can be 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. Figure 1-3 illustrates the sequence of events for the characterization of TRU waste to be included in the WIPP test program.

Prior to supercompaction, waste to be included in the WIPP test program must be characterized by RTR, RA, and visual examination. Headspace sampling and analysis must be performed on all inner layers of confinement of 55-gallon drums containing pucks of supercompacted waste and on bins prior to shipment.

Table 1-2a. Summary of Waste Characterization Requirements and Program Design for Performance Assessment[a]

PARAMETER	TECHNIQUES	DATA QUALITY OBJECTIVES[b]
Total Alpha	**Radioassay[c]** • Passive-Active Neutron • Segmented Gamma Scan • Passive Neutron Coincidence Counting	• Gas Generation Rate and Potential
Waste Descriptors Content Code/Item Description Code Packaging Configuration Waste Material Category Cellulosics, Plastics, Rubber, Corroding Metals/Steel, Corroding Metals/Aluminum, Non-Corroding Metals, Solid Inorganics, Inorganic Sludges, Cements/Additives, Other Organics (resins, oils, organic sludges, solvents)	**Waste Examination** Non-Destructive[c] • Real-time Radiography Destructive[d] • Visual Examination	• Verification of Process Knowledge • Physical Waste Forms • Gas Generation Rate and Potential
Headspace Gas Analysis Gases Argon, Carbon monoxide, Carbon dioxide, Hydrogen, Nitrogen, Oxygen, Methane, Ethane, Propane, Nitrogen Oxides	**Headspace Gas Analysis[d]** • Gas Mass Spectroscopy (MS) • Gas Chromatography (GC) • Spectrophotometric Methods or Equivalent[e]	• Initial Processes: - radiolysis - microbial - chemical

[a] Performance Assessments are detailed in 40 CFR 191, Subpart B.

[b] See Section 1.4 of the QAPP for a complete description of the DQOs.

[c] Conducted for all wastes.

[d] Conducted for all wastes included in bin-scale tests.

[e] For NO_2 and NO only.

Table 1-2b. Summary of Waste Characterization Requirements and Program Design for No-Migration Determination[a]

PARAMETERS	TECHNIQUES	DATA QUALITY OBJECTIVES
Carbon tetrachloride Methylene chloride 1,1,1-Trichloroethane Trichloroethylene 1,1,2-Trichloro-1,2,2-trifluoroethane	**Headspace Gas Analysis** • Gas Chromatography (GC) • Gas Chromatography/ Mass Spectrometry (GCMS)	• Process Knowledge Verification • Comparability
Carbon tetrachloride Methylene chloride Trichloroethylene	**Headspace Gas Analysis** • Gas Chromatography (GC) • Gas Chromatography/ Mass Spectrometry (GCMS)	• Process Knowledge Verification • No-Migration Demonstration
Acetone Benzene n-Butanol 2-Butanone Chlorobenzene Cyclohexane 1,1-Dichloroethane 1,2-Dichloroethane 1,1-Dichloroethene cis-1,2-Dichloroethene Ethyl benzene Ethyl ether Hydrogen Methane Methanol 4-Methyl-2-pentanone Toluene 1,3,5-Trimethylbenzene 1,2,4-Trimethylbenzene m-Xylene o-Xylene p-Xylene	**Headspace Gas Analysis** • Gas Chromatography (GC) • Gas Chromatography/ Mass Spectrometry (GCMS) • Gas Mass Spectroscopy (MS) **Flammability Analysis[b]** • Flame Test	• Process Knowledge Verification • Flammability
Benzene Bromoform Carbon tetrachloride Chlorobenzene Chloroform 1,1-Dichloroethane 1,2-Dichloroethane 1,1-Dichloroethene cis-1,2-Dichloroethene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethane Toluene 1,1,1-Trichloroethane Trichloroethylene 1,1,2-Trichloro-1,2,2-trifluoroethane	**Headspace Gas Analysis** • Gas Chromatography (GC) • Gas Chromatography/ Mass Spectrometry (GCMS)	• Process Knowledge Verification • Quantify Headspace Concentration of Hazardous Constituents

[a] No-Migration Variance Petitions are detailed in 40 CFR 268.6 and, EPA's Final No-Migration Determination (NMD) for the WIPP, i 55 FR 47700.

[b] For headspace samples exceeding the regulatory threshold of 500 ppmv.

Table 1-2c. Summary of Waste Characterization Requirements and Program Design for General Waste Analysis[a,b]

PARAMETERS	TECHNIQUES	DATA QUALITY OBJECTIVES
Headspace Volatile Organic Compounds	Headspace Gas Analysis • Gas Chromatography (GC) • Gas Chromatography/ Mass Spectrometry (GCMS)	• Verification of Process Knowledge • Quantify Headspace Concentrations of Hazardous Constituents
Waste Category	• Real-time Radiography	• Verification of Process Knowledge • Determine Waste Categories
Ignitables, Reactives, and Corrosives[c]	• Real-time Radiography • Waste Acceptance Criteria Certification[d]	• Verification of Process Knowledge • Determine if waste exhibits a hazardous characteristic
Total Metals	• Process Knowledge (Group I Waste Categories) • SW-846 Method or Equivalent (Group II and Group III Waste Categories)	• Hazardous Waste Determination (Group I, Group II and Group III Waste Categories) • Quantify Hazardous Constituents (Group II and Group III Waste Categories)
Total Volatile Organic Compounds	• Process Knowledge (Group I Waste Categories) • SW-846 Method or Equivalent (Group II and Group III Waste Categories)	• Hazardous Waste Determination (Group I, Group II and Group III Waste Categories) • Quantify Hazardous Constituents (Group II and Group III Waste Categories)
Total Semi-Volatile Organic Compounds	• Process Knowledge (Group I Waste Categories) • SW-846 Method or Equivalent (Group II and Group III Waste Categories)	• Hazardous Waste Determination (Group I, Group II and Group III Waste Categories) • Quantify Hazardous Constituents (Group II and Group III Waste Categories)
Polychlorinated Biphenyls	• SW-846 Method or Equivalent (Group II and Group III Waste Categories)	• Verification of Process Knowledge • Identify waste containing greater than 50 ppm PCBs

[a] General Waste Analyses are detailed in 40 CFR 264.13 and 40 CFR 265.13.

[b] Current waste characterization parameters apply only to the Group I Waste Categories described in the WIPP Waste Analysis Plan (USDOE, 1992). The application of the remaining parameters to Group II and Group III Waste Categories will be addressed in future revisions of this QAPP.

[c] Ignitables, reactives, and corrosives are defined in 40 CFR 261, Subpart C.

[d] Per WIPP Waste Profile Form (DOE, 1992).

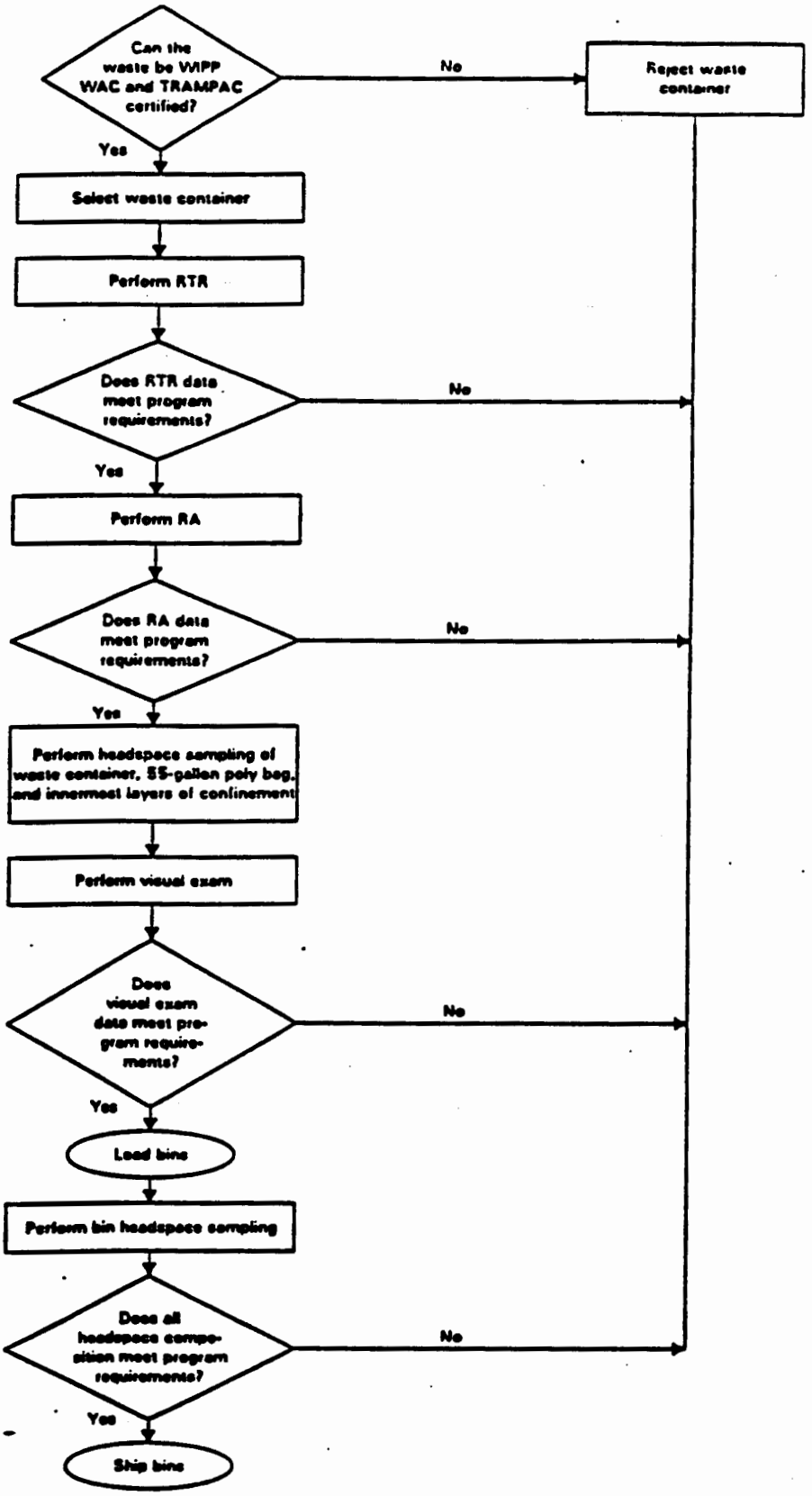


Figure 1-3. Sequence of Events for Waste Characterization

The wastes will be characterized as specified in Table 1-2a prior to shipment to WIPP by the facility originating the shipment. This data provided by RFP and INEL will be required for the interpretation of bin-scale and alcove test results with regard to gas generation.

1.3.2 No-Migration Determination Waste Characterization Requirements

In response to the DOE's No-Migration Variance Petition (Petition) for the WIPP, EPA published its notice of the final conditional NMD for the WIPP on November 14, 1990 (USEPA, 1990). As a result of this NMD, DOE may place a limited amount of untreated TRU waste subject to the land disposal restrictions under RCRA in the WIPP for the purposes of testing and experimentation. The NMD imposes several conditions on such placement and is for a maximum of ten years. This QAPP includes the requirements for compliance with those conditions associated with the waste characterization requirements imposed by EPA (Table 1-2b). Compliance with the other conditions of the NMD (e.g., the air monitoring requirements at the WIPP) are addressed in the WIPP Part B permit application (USDOE, 1992a) and supporting WIPP documents. Figure 1-2 provides a schematic of the overall waste characterization approach for compliance with the NMD.

To comply with the requirements of the NMD associated with waste sampling and analysis, the following conditions must be met:

- *Headspace Sampling* - Obtain a representative headspace sample of gases and VOCs from the waste containers to be included in the WIPP test program. Within the constraints of the matrix to be sampled, samples must be collected from all layers of confinement that may contain sources of gases or VOCs.

- *Comparability* - Demonstrate, by waste type, that the composition of hazardous constituents in the headspace of containers is similar to those concentrations reported in the Petition. The headspace concentrations of the five major hazardous constituents reported by DOE must be less than or equal to the maximum allowable concentrations in the NMD (Table 1-3).

- *No-Migration Demonstration* - Demonstrate, by waste type, that the no-migration finding by EPA remains valid by demonstrating that for each container of waste to be sent to the WIPP (drum or bin), the mean headspace concentrations of three of the hazardous constituents reported in the Petition are less than or equal to the mean headspace concentrations included in the NMD (Table 1-4).

- *Flammability* - Demonstrate that each waste container emplaced underground at the WIPP has no layer of confinement containing flammable mixtures of gases (H_2 and CH_4) and VOCs (Table 1-5) or mixtures of gases and VOCs that could become flammable when mixed with air.

Table 1-3. Maximum Allowable Volatile Organic Compound Headspace Concentrations (Volume Percent) by Waste Type[a,b]

Constituent	Type I	Type II	Type III	Type IV
Carbon tetrachloride	0.08	0.18	0.58	8.18
Methylene chloride	0.44	0.84	0.50	1.42
1,1,1-Trichloroethane	1.88	5.68	2.12	14.96
Trichloroethylene	0.08	0.34	0.28	0.28
1,1,2-Trichloro-1,2,2-trifluoroethane	0.05	1.62	5.74	20.80

[a] Waste types are identified in USDOE, 1989.
[b] Concentration values from the No-Migration Determination (USEPA, 1990). These concentrations are obtained by multiplying the maximum VOC headspace concentrations reported in the Petition by two.

Table 1-4. Allowable Mean Volatile Organic Compound Headspace Concentrations (Volume Percent) by Waste Type[a,b]

Constituent	Type I	Type II	Type III	Type IV
Carbon tetrachloride	0.24	0.26	0.30	6.90
Methylene chloride	0.39	0.42	0.33	0.93
Trichloroethylene	0.25	0.28	0.29	0.38

[a] Waste types are identified in USDOE, 1989.
[b] Concentration values from the No-Migration Determination (USEPA, 1990). These concentration values are obtained by multiplying the mean VOC headspace concentrations reported in the Petition by ten.

Table 1-5. Flammable Volatile Organic Compounds[a] (VOCs) and their Lower Explosive Limit (LEL) Values[b,c]

VOCs	LEL (v/v%)
Acetone	2.5
Benzene	1.3
n-Butanol	1.4
2-Butanone	1.4
Chlorobenzene	1.3
Cyclohexane	1.3
1,1-Dichloroethane	5.6
1,2-Dichloroethane	6.2
1,1-Dichloroethene	6.5
cis-1,2-Dichloroethene	5.6
Ethyl benzene	1.0
Diethyl ether	1.9
Methanol	6.0
4-Methyl-2-pentanone	1.2
Toluene	1.2
1,2,4-Trimethylbenzene	0.9
1,3,5-Trimethylbenzene	1.4
o-Xylene	1.0
m-Xylene	1.1
p-Xylene	1.1

[a] VOCs identified as flammable by EPA for the NMD. EPA determined flammability by assessing potential fire hazards under WIPP conditions on a compound by compound basis.

[b] The lower explosive limit is also referred to as the lower flammable limit (ASTM, 1989 and NFPA, 1986).

[c] Only the most conservative lower explosive limit values are reprinted (NFPA, 1986 and De Renzo, 1986).

1.3.2.1 Headspace Sampling

Pretest waste characterization for the bin-scale tests will include sampling gases and VOCs from three areas within drums of TRU waste. These areas include (1) the drum headspace (i.e., the headspace directly under the drum lid), (2) the 55-gallon poly bag headspace, and (3) the innermost layers of confinement headspace. The drum, 55-gallon poly bag, and the innermost layers of confinement headspace sampling procedures are described in Section 4.0. In addition, bin headspace samples shall be obtained in accordance with procedures also described in Section 4.0.

In order to demonstrate compliance in the future when drums could be directly emplaced in the WIPP, DOE must demonstrate to EPA's satisfaction that a drum headspace gas sample is representative of the gases and VOCs within the entire drum. Although compliance with specific conditions of the NMD during the bin-scale tests may be demonstrated based on the results of sampling and analyzing the headspace gases and VOCs in an experimental bin, samples must be obtained from all layers within the drums included in that bin. The data obtained from all the sampling locations will be evaluated by DOE to assess the need to obtain additional information on the concentrations of hazardous constituents within drums that may be emplaced in the WIPP during the alcove tests.

1.3.2.2 Comparability

To comply with the maximum concentration comparability condition of the NMD, headspace samples from within the drum, 55-gallon poly bag, and from the innermost layers of confinement shall be analyzed using Program-approved analytical procedures to determine the concentrations of carbon tetrachloride (CCl_4), methylene chloride (CH_2Cl_2), 1,1,1-trichloroethane (TCA), trichloroethylene (TCE), and 1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113) (Figure 1-4). The upper 90% confidence limit value of the highest measured concentration of each of these five major constituents found in the innermost layers of confinement shall be compared to the compliance concentrations listed in Table 1-3.

The upper 90% confidence limit value of the measured concentration of the five major hazardous constituents within the drum and within the 55-gallon poly bag headspace shall also be compared to the allowable concentrations in Table 1-3. If these comparisons and those described above indicate that the highest concentrations of each of the five major hazardous constituents are less than the maximum allowable concentrations in Table 1-3, then DOE may place the contents of that drum into

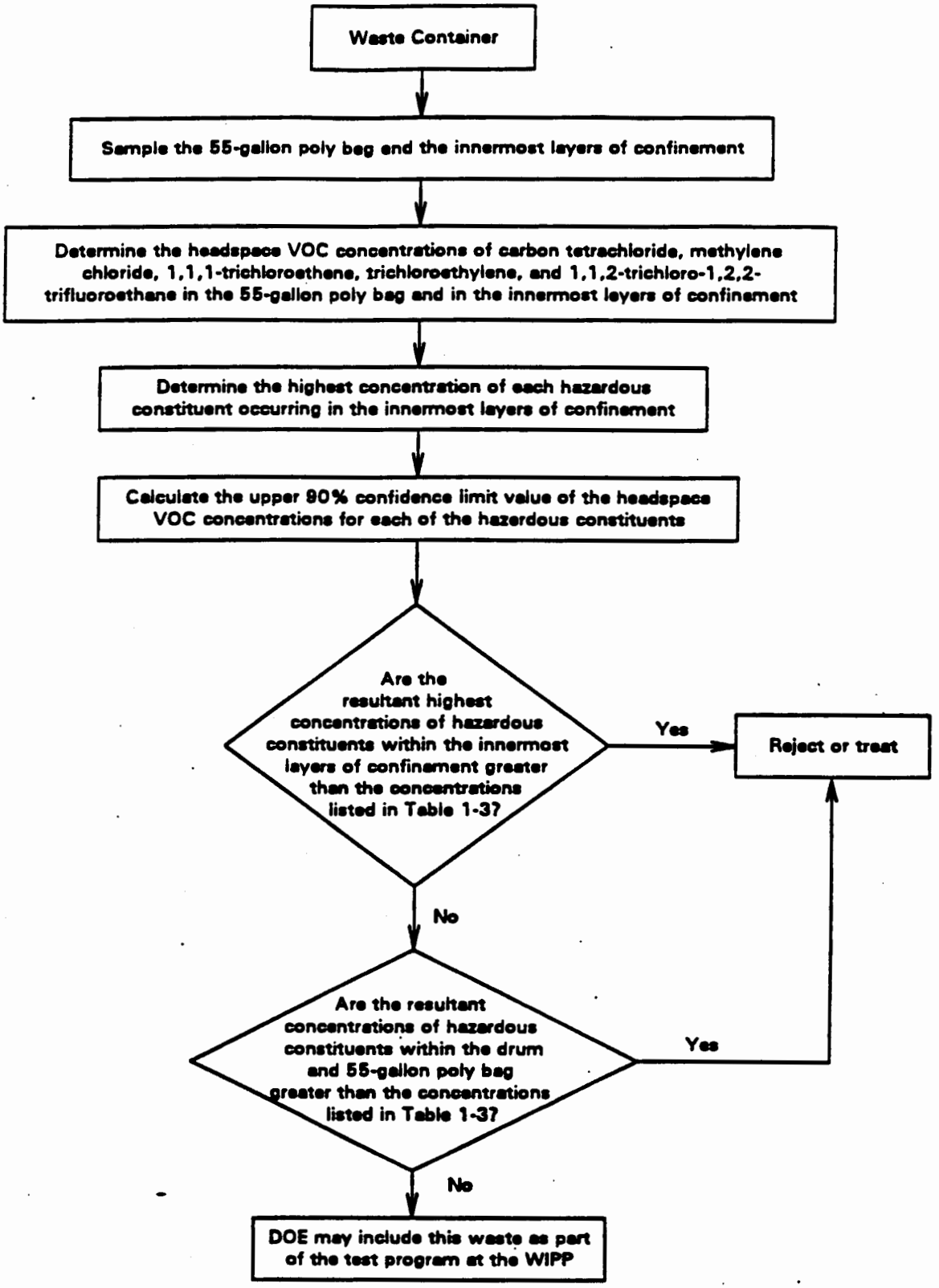


Figure 1-4. Comparability Flowchart

a bin for inclusion in the WIPP test program. If drums of waste are loaded into a bin prior to receipt of results of headspace sampling and analyses, the entire bin would be rejected if the data indicate that any drum is not in compliance with regard to the comparability criteria. Results of drum headspace sampling will be compared to 55-gallon poly bag and innermost layers of confinement to assess demonstration of comparability.

1.3.2.3 No-Migration Demonstration

To comply with this condition of the NMD during the bin-scale tests, a headspace sample from within each experimental bin shall be collected (Section 4.5). Headspace samples from the experimental bins shall be analyzed using Program-approved analytical procedures to determine the concentrations of CCl_4, CH_2Cl_2, and TCE (Figure 1-5). Once determined, the upper 90% confidence limit value of the measured headspace concentrations of each of these hazardous constituents shall be compared (Section 12.6) to the allowable concentrations specified in Table 1-4. If this comparison indicates that the headspace concentrations of each of the three hazardous constituents are less than those specified, the bin may be emplaced in the WIPP as part of the WIPP test program.

1.3.2.4 Flammability

Figure 1-6 shows the overall approach to compliance with the flammability determination requirements. A representative sample shall be obtained from the bin headspace (Section 4.3) and analyzed for flammable VOCs (Table 1-5) using Program-approved procedures. Other headspace data obtained during waste characterization in support of the bin-scale tests shall be used to assess compliance for alcove tests.

Flammable VOCs

Bin headspace analytical results shall be evaluated to determine if significant levels of flammable VOCs are present in the bins included in the WIPP test program. Significant levels of flammable VOCs (Table 1-5) are defined as measured concentrations of 500 ppmv or greater. To perform this evaluation, the upper 90% confidence limit value of the measured concentrations (Section 12.6) of all analytes (Table 1-5) shall be summed. If the summed value exceeds 500 ppmv, a theoretical lower explosive limit (LEL) shall be calculated using the concentrations of the flammable VOCs plus hydrogen and methane. The flammability of the headspace sample shall be determined by using a modified version of ASTM Method E 681-85 (Section 7.6). For purposes of complying with the NMD, a headspace VOC mixture shall be considered flammable if, under the specified conditions of the test, the mixture propagates a flame from the ignition source to the test vessel walls.

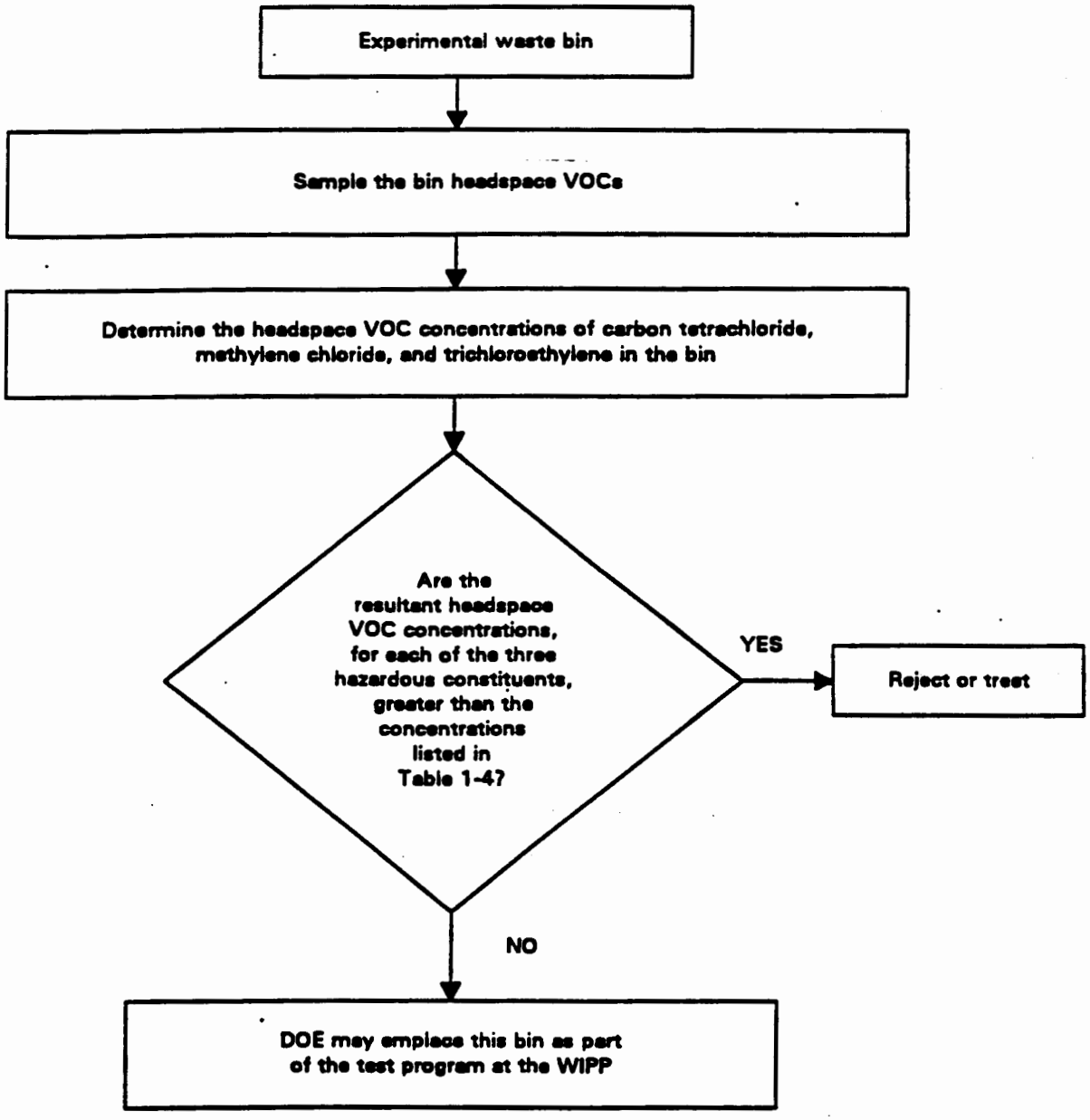


Figure 1-5. No-Migration Demonstration Flowchart

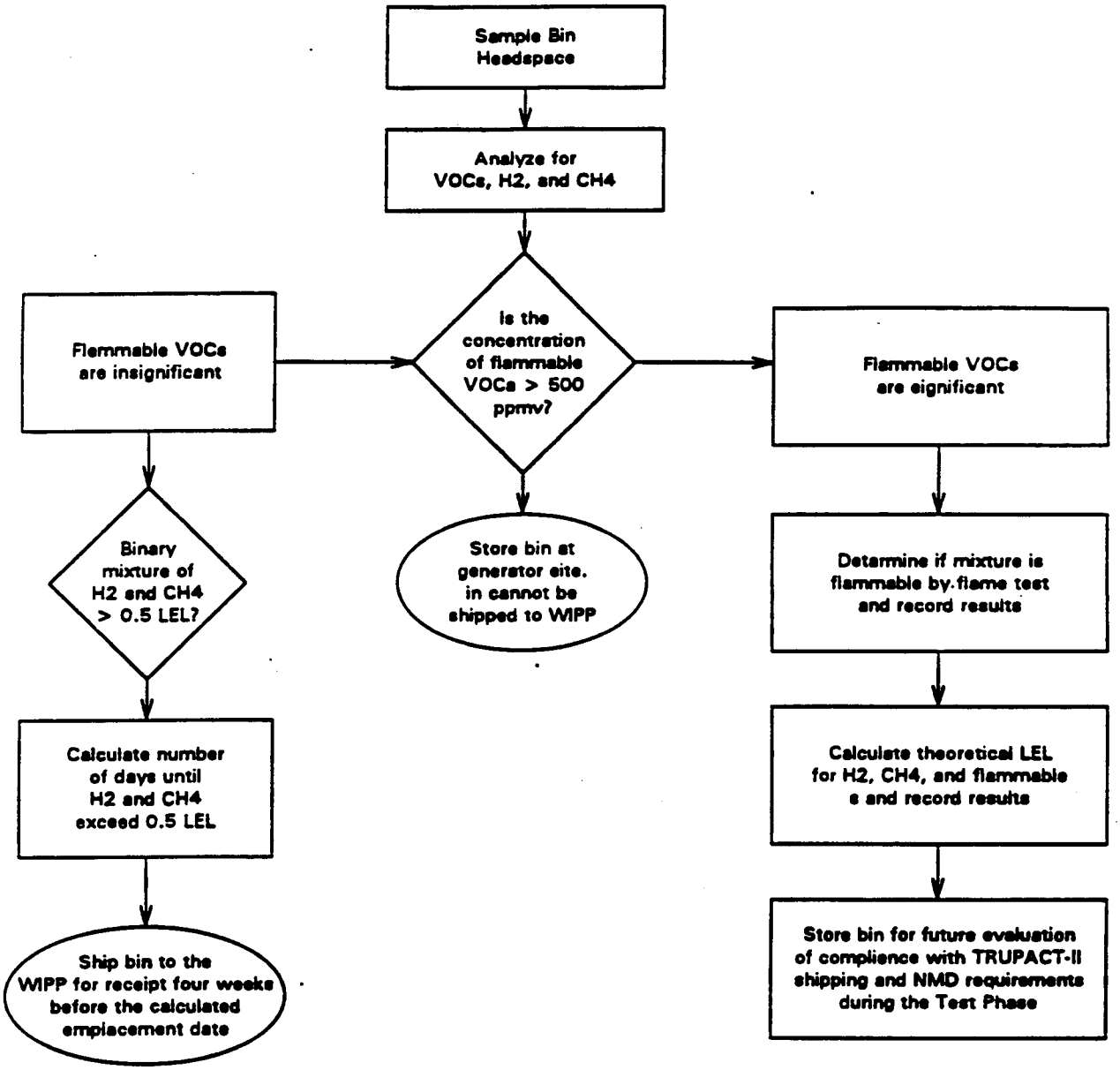


Figure 1-6. Compliance With Flammability Testing Requirements

In accordance with the Nuclear Regulatory Commission's (NRC) Certificate of Compliance, if the headspace concentrations of flammable VOCs exceeds 500 ppmv, this bin currently may not be transported in the TRUPACT-II.

Le Chatelier Formula

For bins of waste that contain headspace concentrations of flammable VOCs less than 500 ppmv, the Le Chatelier formula (Coward and Jones, 1952) shall be used to determine if a headspace gas mixture exceeds 50% of the lower explosive limit (LEL) for binary mixtures of hydrogen and methane. The Le Chatelier formula is generated when the reciprocal of the LEL is substituted for the independent variable "a" in Equation 12-9 (Section 12.6). To perform the Le Chatelier determination:

1. Correct the measured analyte concentrations for recovery using Equation 12-10, Section 12.6.

2. Calculate analyte standard deviations using the measured analyte concentrations and analyte relative standard deviation values using Equation 12-11, Section 12.6.

3. Calculate the percentage of the theoretical LEL using the Le Chatelier formula expressed as Equation 12-9, Section 12.6, using the upper 90% confidence limit value of the H_2 and CH_4 concentrations. The mixture exceeds 50% of the LEL if the result of this computation exceeds 50%.

Emplacement Time Determination

The length of time during waste handling activities in which the mixture of hydrogen and methane will remain below 50% of the theoretical LEL shall be determined, and the bin shall be emplaced prior to exceeding that time limit. Generator/storage sites must perform an initial sampling and analysis of the bin headspace for VOCs (Table 3-7), hydrogen and methane. Generator/storage sites will submit a calculated emplacement time window (time in which hydrogen and methane concentrations remain below 50% of the theoretical LEL) in the bin case addendum report (Section 8.0). Generator/storage site must ensure that the remaining emplacement time (Sections 8.7.1.3 and 12.6) is always greater than four weeks. If the bin is not shipped within the calculated emplacement time window, i.e., emplacement date minus four weeks, generator/storage sites must resample the bin and determine a final emplacement time window prior to shipment. The final emplacement time window calculation requires updated hydrogen and methane concentration values, but not updated VOC concentrations. Generator/storage sites must obtain updated hydrogen and methane concentration values by re-sampling the bin headspace.

The procedure for calculating emplacement time windows is as follows:

- An initial value for bin headspace hydrogen and methane concentrations is obtained.

- Based on the initial hydrogen and methane concentrations, the number of days remaining until 50% of the LEL is exceeded is determined from Tables 12-3 to 12-7 (Section 12.6). The times reported in Tables 12-3 to 12-7 include an allowance for a three-day shipping period.

- If the bin is not sampled, Table 12-2 (Section 12.6) shall be used to determine the remaining emplacement time, given knowledge of the time since the bin was loaded.

Bin sampling and analysis allows for maximization of the amount of time prior to emplacement.

1.3.3 General RCRA Waste Analysis Requirements

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 adequately characterize each waste (Table 1-2c). The criteria used to determine the requirements for the types and frequency of analyses are specifically addressed in the Waste Analysis Plan (USDOE, 1992). Data pursuant to waste characterization will be sent to, and approved by, DOE/WPSO prior to the shipment of the waste to the WIPP facility.

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 WIPP test 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. This QAPP shall be revised, as necessary, to ensure that it establishes the DQOs for future waste characterization data acquisitions. The DQOs are:

Radioassay (RA)

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.

Real-time Radiography (RTR) and Visual Examination

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.

Real-time Radiography (RTR)

Identify the RCRA Waste Category as defined in the WIPP Waste Analysis Plan.

Gas Sampling and Analyses

Determine the concentrations of gases (Table 3-6) 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.

Data on the concentrations of hydrogen and methane also will be used to demonstrate that binary mixtures of these gases do not exceed 50% of the Le Chatelier theoretical lower explosive limit (LEL) in waste containers and bins to be emplaced in the WIPP. In addition, based on the data collected or the time since bin loading (i.e., the waste has been loaded in the bin and the bin closed and placed in the standard waste box) at the generator/storage site, the number of days remaining until 50% of the LEL is exceeded will be determined (Section 12.6) and documented.

Volatile Organic Compound (VOC) Sampling and Analyses

Determine the concentrations of VOCs in the headspace of waste containers and bins at the DOE generator/storage facilities. These data shall be used to demonstrate compliance with the conditions of the no-migration determination requirements associated with waste characterization. In addition, as suggested by EPA in the NMD, these data shall be used in a future operational-phase no-migration variance petition.

The specific DQOs for demonstrating compliance with the waste characterization requirements in the NMD are as follows:

Comparability - To demonstrate that the upper 90% confidence limit values of the measured headspace concentrations of volatile organic compounds listed in Table 1-3 do not exceed two times the maximum concentrations reported in DOE's Petition (USDOE, 1990b).

No-Migration Demonstration - To demonstrate that the upper 90% confidence limit values of the measured headspace concentrations of VOCs listed in Table 1-4 do not exceed ten times the mean concentrations reported in DOE's Petition.

Flammability - To demonstrate that (1) the upper 90% confidence limit values of the measured headspace concentrations of flammable VOCs listed in Table 1-5 do not exceed 500 ppmv, (2) if a mixture of flammable VOCs does not exceed 500 ppmv then to demonstrate that the upper 90% confidence limit valves of the headspace concentrations of hydrogen and methane do not exceed 0.5 of the LEL, and (3) if a mixture of flammable VOCs exceeds 500 ppmv, then to demonstrate, using an explicit "flame test," that the mixture is nonflammable.

Because wastes that contain headspace concentrations of flammable VOCs that exceed 500 ppmv currently may not be shipped in the TRUPACT-II, they will not be included as part of the WIPP test program unless DOE obtains an amendment to the TRUPACT-II Safety Analysis Report from the Nuclear Regulatory Commission to transport these wastes. Headspace concentrations of flammable VOCs less than 500 ppmv are considered by EPA to not significantly contribute to the potential flammability of the waste, therefore, as described under headspace sampling and analysis DQOs, the calculated theoretical LEL will be determined based on a binary mixture of hydrogen and methane.

RCRA-Regulated Hazardous Waste Characterization

Verify existing waste characterization information that is based on knowledge of the materials and processes that generate the waste using the information obtained from the Program. The QAPP will be revised to include the data quality objectives and the sampling and analytical protocols when the method evaluations are finalized.

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 characterizing TRU waste to be sent to the WIPP 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 program 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 1-6 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 all quality documents for the Program shall be accomplished 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 by those organizations indicated in Table 1-6, and thereafter, at least annually reviewed, by DOE/WPIO to ensure the QAPP addresses the current needs of the Program. Based on the annual review, if changes to the QAPP are required, the Director of DOE/WPIO shall be responsible for scheduling and coordinating the review and approval of the document. It is the responsibility of DOE/WPIO to schedule and coordinate the annual review.

Table 1-6. Requirements for Review, Approval, Implementation, and Control of the QAPP and QAPjPs

Responsible Party	Deputy Assistant Secretary DOE/ EM-30	Associate Director DOE/ EM-20	Director DOE/ EM-34	Director DOE/ EM-342	Director DOE/ WPIO	Chief EP&WB DOE/WPIO	Manager QA and Compliance Branch DOE/WPIO	Site Manager DOE/ WPSO	Chairperson WIPP/ WACCC	Sandia National Laboratories	DOE Field Office	Site Project Manager	Site Project QA Officer
QAPP													
Review		X		X					X	X	X		
Review/Approval	X		X		X	X	X	X					
Implementation[a]									X				
Change Approval	X		X		X	X	X	X					
Change Control						X							
QAPjPs													
Review			X	X	X	X	X	X		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.

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 Field Office, the WIPP Waste Acceptance Criteria Certification Committee (WACCC) Chairperson, 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 Field 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/WPIO to the applicable DOE Field Offices. Distribution lists for this QAPP shall be used to control the issuance of revisions and shall be maintained by DOE/WPIO.

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 Field 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. 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. In accordance with DOE Order 5700.6C, revised submittals shall identify the changes, the pages affected, the reason for the changes, and the basis for concluding that the revised program continues to satisfy the requirements of the Order.

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-34, DOE/WPIO, and DOE/WPSO prior to initiation of the change at the applicable DOE facilities. Changes shall be reported by DOE/WPIO to the DOE Field 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 left-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 Field Office. No changes that can affect the performance criteria or data quality shall be made without prior approval of the DOE Field Office, the WACCC, and DOE/WPIO and DOE/EM-34.

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/WPSO. In accordance with DOE Order 5700.6C, each site's system shall meet the requirements of DOE Order 1324.2A, "Records Disposition" (USDOE, 1988b).

Each DOE generator/storage site shall maintain in its project files the original or a copy, as appropriate, of all QA/QC waste characterization data for waste that is sent to the WIPP. In addition, all QA/QC records associated with TRU wastes that are characterized but not sent to the WIPP because of noncompliance with the current programmatic or regulatory waste characterization requirements shall be maintained, for future evaluation, at the DOE generator/storage sites in accordance with the recording keeping requirements specified in this section.

The data/records management systems shall provide adequate control and retention for all the Program-related information. All waste characterization data and related QA/QC records that are to be transferred to DOE/WPSO 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," (USDOE, 1991b), 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/WPSO.

1.6.1 Central Files

Each DOE site shall develop a records management system for hard copies of all information transmitted to DOE/WPSO 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/WPSO who shall maintain all the 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/WPSO must be traceable to the original, raw data records. This information shall be maintained by DOE/WPSO 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 an original hard copy 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/WPSO.

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/WPSO or its contractor's office central program files. Records sent to DOE/WPSO shall be reviewed, approved, and transmitted in accordance with the requirements specified by DOE/WPSO. Sites shall keep a record of all documents or records transmitted to DOE/WPSO.

At 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 1-7 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

Table 1-7. 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.

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 Investigation Center (NEIC) guidelines (USEPA, 1986a).

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. The site Project Manager is responsible for verifying supplier compliance with the applicable QA/QC requirements.

1.7.2 Control of Subcontractors

Performance requirements must be communicated to all subcontractors involved in the Program. Performance requirements and compliance with this QAPP shall be part of subcontractor agreements. DOE operating contractors shall perform and document the results of 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 the 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 Sections 10.0 and 13.0.

All subcontractors providing analytical services in support of the Program must participate in the Performance Demonstration Program (PDP) as described in Section 1.11. Each subcontractor for analytical services shall demonstrate their ability to meet the QA objectives for the Program by successfully completing the requirements of the PDP prior to sample analysis. DOE WIPP Project Integration Office (DOE/WPIO) shall review and approve the results of the PDP prior to the initiation of work by the subcontractor for analytical services. DOE/WPIO, as the administrator of the PDP, 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.

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. 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. 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 identification and control 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 identification and traceability of items may include item identification from initial receipt up to and including installation and use, physical identification, clear and legible marking, or a combination thereof. Items having limited calendar or operating life shall be identified and controlled to preclude use of items whose shelf life or operating life has expired.

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 and documented at periodic intervals.

Criterion 2 of DOE Order 5700.6C requires that personnel shall be provided continuing training to ensure that job proficiency is maintained. Training included both education in principles and enhancement of skills and practices. Each site shall include in its QAPP a description of the procedures for implementing this aspect of personnel training and qualification in accordance with the guidance of DOE Order 5700.6C.

Training may not be waived for visual examination of waste (Section 7.3). 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 1-8. 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.

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

Table 1-8. 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 1 year independent spectral interpretation or demonstrated expertise
Gas Mass Spectrometer Analysts * Gas Chromatograph-Mass Spectrometer Analysts *	B.S. or equivalent experience and 1 year applicable experience
UV-VIS Spectrophotometer Analysts *	3 months applicable experience
Flammability Apparatus Analysts */Interpreter	3 months applicable experience

* Analysts are those persons responsible for the overall technical operation and development of a specific laboratory technique.

- 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 and consistent with EPA QAMS-005, 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.

Consistent with DOE Order 5820.2A, TRU waste operations shall be conducted in accordance with the applicable elements of ASME NQA-1 and the analogous and supplementary DOE Order 5700.6C ten criteria. A cross reference in the QAPjP may be used to indicate the applicable sections pertaining to each requirement.

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 PDP and demonstrate conformance to the QA objectives for the Program. Each laboratory, through participation in the PDP, will demonstrate and document its performance characteristics. The PDP Plan (USDOE, 1992b) includes a detailed description of the requirements of the PDP.

A DOE/WPIO 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.

1.12 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 approved 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).

1.13 Quality Improvement

DOE/WPIO senior management shall be responsible for implementation, assessment, and improvement of this QAPP. The objective is to ensure quality through appropriate planning, control of work operations, verification, and review, and to achieve a rising standard of quality through continuous improvement. The focus of quality improvement should be to reduce the variability of every process which influences the quality of the data. Each site shall include in its QAPjP, a description of the procedures for identifying items and processes needing improvement to satisfy this aspect of DOE Order 5700.6C, Criterion 3, using the guidance provided in the Order. Management assessments shall be performed as a means of improving quality.

1.14 Management Assessment

In accordance with DOE Order 5700.6C, Criterion 9, management at all levels shall periodically assess the integrated quality assurance program and its performance. The Director of DOE/WPIO is responsible for direction and participation in the management assessment program which will identify management problems that hinder the organization from achieving its objectives. Management assessment results shall be documented. The Director of DOE/WPIO shall perform periodic evaluations of the effectiveness of management corrective actions and the status of the quality program by review and participation in self assessments, independent assessment, management assessments, and external appraisals.

2.0 ORGANIZATION AND RESPONSIBILITY

DOE Order 5700.6C (USDOE, 1991) establishes the policies and responsibilities to assure quality in DOE programs. This DOE Order requires that quality assurance programs include a description of the organizational structure, functional responsibilities, levels of authority, and interfaces for those managing, performing, and assessing the adequacy of work. In compliance with DOE Order 5700.6C and 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 the 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 Integration Office (DOE/WPIO), the DOE WIPP Project Site Office (DOE/WPSO), 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. The document hierarchy of program quality assurance requirements for the Program is illustrated in Figure 2-2.

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 reviewed and approved by DOE Headquarters (Table 1-6). 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 Assistant Secretary of Office of Environmental Restoration and Waste Management (EM-1)

The Assistant Secretary of the Office of Environmental Restoration and Waste Management is responsible for providing DOE policy and guidance to the WIPP program elements and coordinating all WIPP-related activities at DOE Headquarters, as well as with Congress, the Environmental Protection Agency (EPA), Nuclear Regulatory Commission (NRC), and other Governmental and civilian entities,

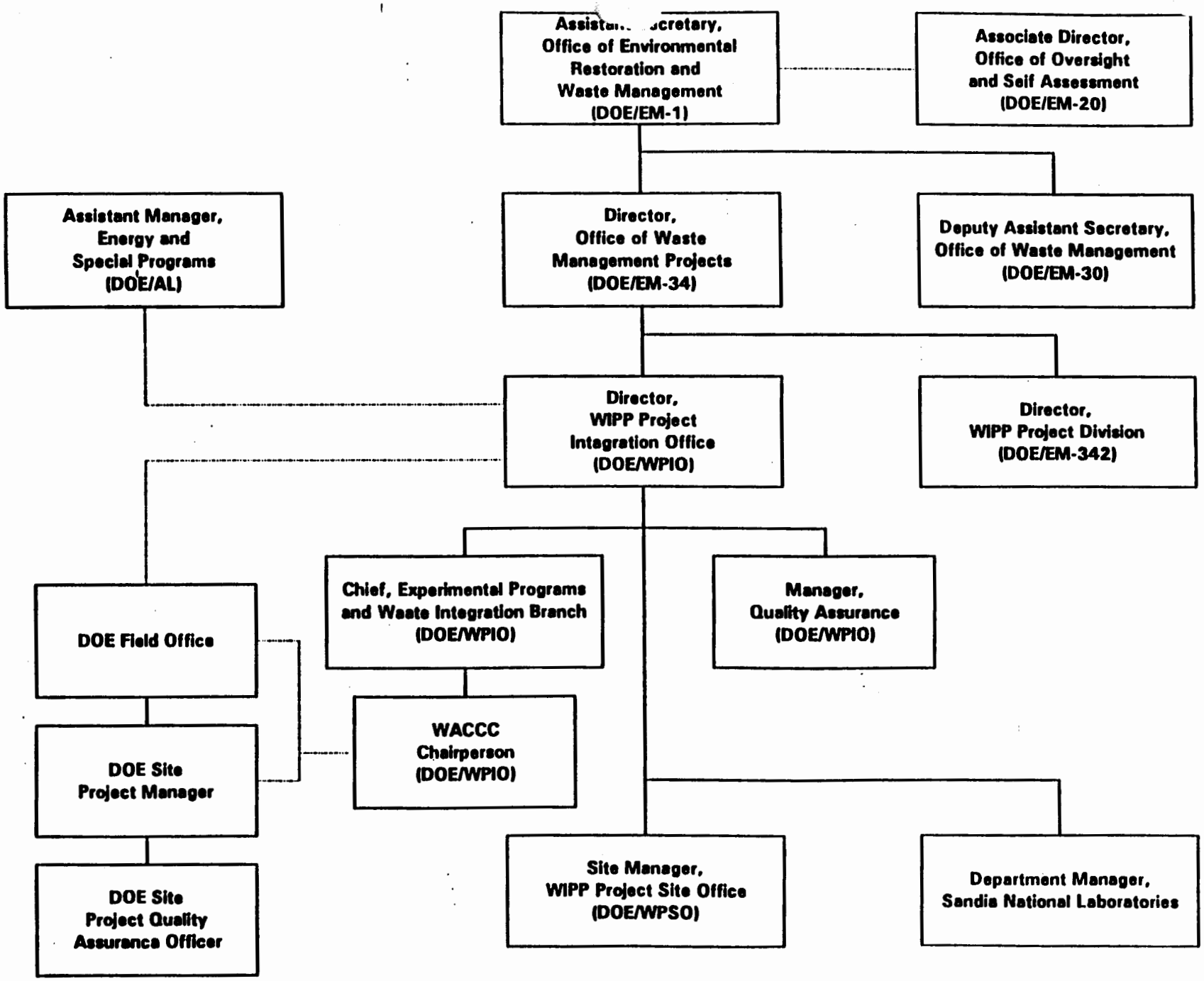


Figure 2-1. QAPP Functional Organization Chart

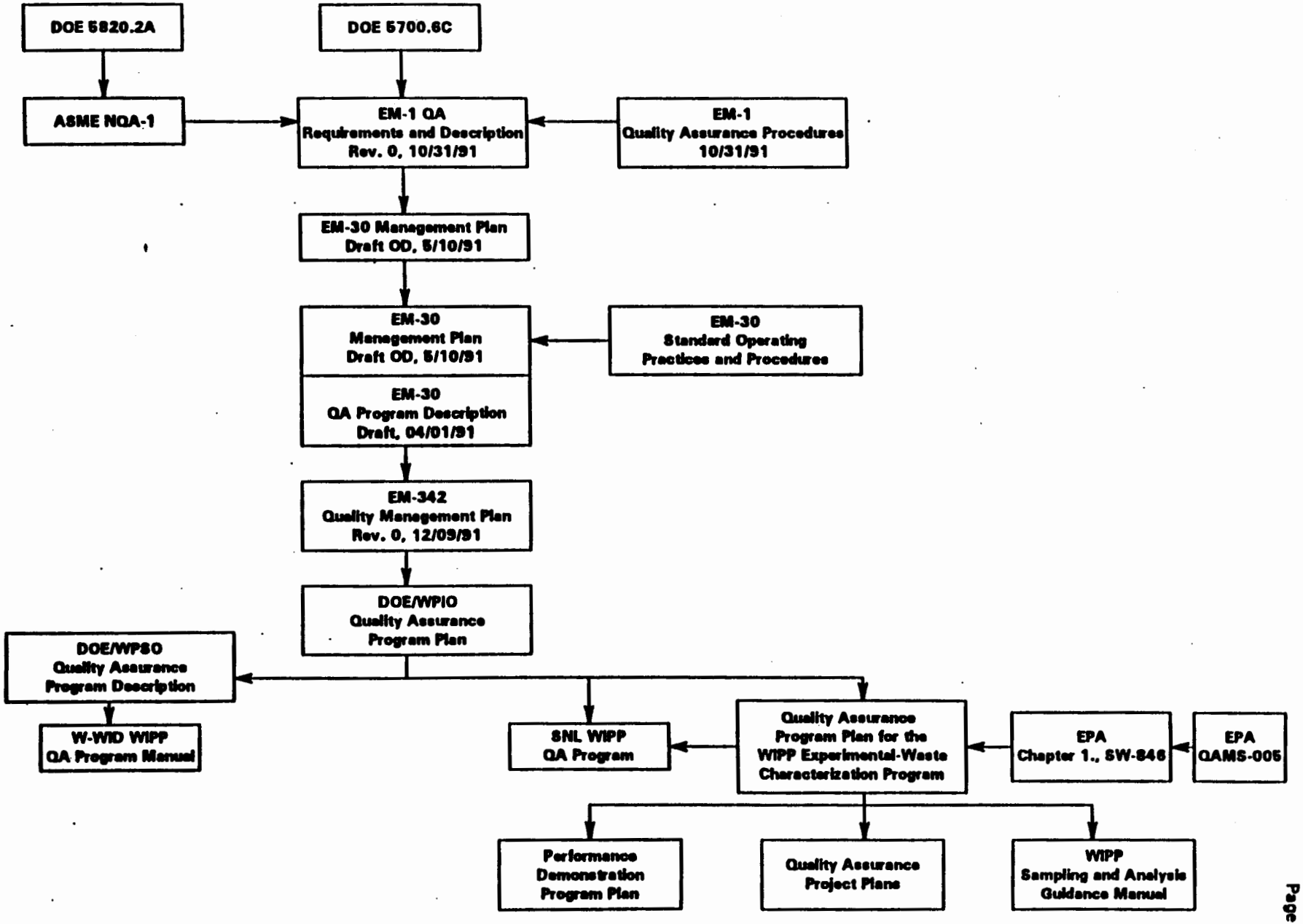


Figure 2-2. WIPP Waste Characterization Quality Assurance Requirements Flowchart

as appropriate. The Assistant Secretary has the overall responsibility and authority for ensuring that DOE quality assurance policy is implemented. The Assistant Secretary provides guidance and direction to the field organizations consistent with requirements related to quality assurance. These responsibilities are delegated to EM-34 for implementation on a day-to-day basis.

2.1.2 Associate Director of the Office of Oversight and Self Assessment (EM-20)

The Associate Director of the Office of Oversight and Self Assessment 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 Associate Director provides interpretations of requirements and interfaces and coordinates with other federal agencies for programs and activities managed by EM.

The Associate Director develops and oversees the implementation of the EM QA Program based on DOE Orders, national standards, EPA requirements, and EM program requirements. The Associate Director shall provide an independent oversight function regarding the requirements of this QAPP and shall review this QAPP for consistency with all applicable regulations and requirements. The Associate Director may participate in selected field organization audits and appraisals of contractor facilities/operations that may be scheduled at least annually by the Field Offices or EM-34.

2.1.3 Deputy Assistant Secretary, Office of Waste Management (EM-30)

The Deputy Assistant Secretary 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 Deputy Assistant Secretary manages the on-site and off-site preparation, packaging, and transportation of waste within the DOE complex. The Deputy Assistant Secretary develops, promulgates, and monitors the effectiveness of DOE policies related to the safe handling, storage, treatment, or disposal of TRU wastes throughout the system. In accordance with these responsibilities, the Deputy Assistant Secretary shall review and approve the QAPP to ensure adequate planning and resources to support the Program.

2.1.4 Director, Office of Waste Management Projects (EM-34)

The Director of Waste Management Projects is responsible for implementing the directions, policies and guidance set forth by EM-1. In this capacity, the Director will coordinate with EM-30 on budget; change control for budget, schedule, and technical criteria; strategic planning; and other matters, as appropriate. A primary responsibility of the Director is to approve project implementation plans and to assess the status and adequacy of the WIPP project implementation. As part of this responsibility,

the Director of the Office of Waste Management Projects will review and approve this QAPP. In addition the Director shall review the site-specific QAPjPs.

2.1.5 Director, WIPP Project Division (EM-342)

The Director of the WIPP Project Division is responsible for supporting the program management, technical direction, and oversight requirements of EM-34. The Director monitors the WIPP program progress and verifies compliance with the technical requirements through a combination of program, technical, and readiness reviews. These activities, as well as the overall quality assurance program for the EM-342 WIPP Division are described in the EM-342 *Quality Management Plan*, Rev. 0, dated December 9, 1991. The Director will review the QAPP and provide comments to EM-34 regarding Program implementation.

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

DOE Albuquerque Field Office has established the WIPP Project Integration Office (WPIO) (Section 2.1) and, in consultation with EM-1, selects the Project Director. In support of the Project Director, the Assistant Manager will provide matrix support for those activities involving legal, finance/budget, procurement, industrial relations, payroll, contracts, personnel, quality assurance, safety, environmental protection, and safeguards and security. The Assistant Manager will also provide appropriate management oversight for assessment of this support to assure DOE/WPIO effectiveness in carrying out EM policy, guidance, and direction.

2.3 DOE WIPP Project Integration Office (DOE/WPIO)

Designated by DOE/AL, the DOE/WPIO, located in Albuquerque, New Mexico, has the responsibility and the prerequisite authority for the day-to-day implementation of DOE Headquarters policy, program guidance, and technical direction of the WIPP project, as provided by EM-34. The QAPP shall be approved, issued, and controlled by DOE/WPIO. The DOE/WPIO is responsible for management activities related to the WIPP experimental program development, including performance assessment and analysis of experimental results and the RCRA waste characterization requirements. The DOE/WPIO also is responsible for WIPP project integration of activities supported by the Sandia National Laboratories, the DOE/WPSO, and other Program participants.

2.3.1 Director, WIPP Project Integration Office (DOE/WPIO)

The Director coordinates all WIPP related activities with DOE/EM, DOE/AL, and DOE/WPSO. In addition, the Director has direct responsibility and accountability for execution and implementation of all WIPP related activities, including integration of the prime contractors (i.e., Westinghouse/WID and Sandia

National Laboratories) and integration of TRU waste preparation, characterization, packaging and transportation activities at the DOE generator sites. Other responsibilities related to the QAPP activities include providing TRU waste integration in direct support of the WIPP test program, development of the Test Phase strategy, including waste characterization, and management of the Bin Preparation Task Force. The Director provides guidance on DOE Headquarters policy and program direction through primary contractors and participating field offices. The Director shall review and approve the QAPP for consistency with DOE TRU waste long-term management plans and DOE TRU waste generator/storage facilities' implementation requirements.

2.3.2 Chief, Experimental Programs and Waste Integration Branch (DOE/WPIO)

The Chief of the Experimental Programs and Waste Integration Branch is responsible for all TRU waste characterization activities for the WIPP program. The Chief integrates all waste characterization activities throughout the DOE TRU waste management complex and ensures these activities are consistent with program objectives and needs. In this regard, the Chief shall review and approve the QAPP. The Chief also has the overall responsibility for management of all activities related to the conducting of the Test Phase at the WIPP.

In addition, the Chief of the Experimental Programs and Waste Integration Branch shall be responsible for administration of the PDP (Section 1.11). In this role, the Chief shall be responsible for coordinating implementation of the PDP 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 Chief shall be responsible for scheduling and coordinating the PDP, and resolving any issues related to conformance with the Quality Assurance Objectives (Section 3.0) of this program. The Chief also shall be responsible for formally qualifying and approving each analytical laboratory's participation in the Program.

The Chief also is responsible for review of all new and revised sampling and analytical procedures for TRU waste that are included in the *WIPP Sampling and Analysis Guidance Manual*. The Chief is responsible for the production and distribution of the Guidance Manual. Although the Guidance Manual is not a controlled document, the Chief shall ensure that all of the Program participants and other agencies included on the distribution, receive all updates of new or revised procedures that are part of this manual.

2.3.3 Manager, Quality Assurance and Compliance Branch (DOE/WPIO)

The Manager of the Quality Assurance and Compliance Branch of the DOE/WPIO is responsible for providing independent quality assurance oversight for all WIPP activities including the waste characterization program. In this regard, the Manager will review the QAPP for compliance with all quality assurance requirements. In addition the Manager of Quality Assurance will oversee QA audits, quality verification inspections, and corrective actions related to the QAPP.

2.3.4 WACCC Chairperson (DOE/WPIO)

The WACCC Chairperson reports to the Chief of the Experimental Programs and Waste Integration Branch. The authority of the WACCC is established by DOE Order 5820.2A (USDOE, 1988). Two of the primary responsibilities of this committee related to the Program are the development of the WIPP Waste Acceptance Criteria (WAC) and the verification of the 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.

2.3.5 Sandia National Laboratories (SNL)

As a Scientific Advisor to DOE/WPIO, 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, Subpart B. SNL reports directly to the Director of the DOE/WPIO concerning experimental objectives and data requirements.

SNL is responsible for the development of the WIPP test 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 Director as well as input to the QAPP based on the test requirements.

2.4 DOE WIPP Project Site Office (DOE/WPSO)

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

2.4.1 WIPP Project Site Manager (DOE/WPSO)

The WIPP Project Site Manager reports directly to the Director of DOE/WPIO and is responsible for the management of the WIPP Management and Operating Contractor. The WIPP Project Site 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 Site Manager shall review and approve the QAPP for consistency with DOE policies and regulatory requirements pertaining to the WIPP (Table 1-3). The WIPP Project Site Manager reports to DOE/WPIO to ensure that adequate funding and personnel are available to implement and oversee the activities related to waste management at the WIPP.

The WIPP Project Site Manager also is responsible for managing all WIPP site activities related to conducting of the Test Phase. The WIPP Project Site 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 Site 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.

2.5 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 Orders and policies are implemented.

2.5.1 DOE Field Office

As a part of the Program, each DOE Field Office will review and approve the site QAPjPs for the facility under that office's responsibility. The Field Offices 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 Field Offices prior to implementation of the change. The DOE Field Offices 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.

2.5.2 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 1-3). The site QAPjPs shall include a description of the line management responsibilities for implementation of the Program.

2.5.3 DOE Site Project Quality Assurance Officer

Each DOE facility's operating contractor shall designate a site Project QA Officer for the 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 1-3). 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 designed to obtain data to support the data quality objectives, as described in Section 1.4.

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. Six waste characterization techniques must be used:

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

The relationships between these waste characterization techniques and the Program data quality objectives (Section 1.4) 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 and semi-quantitative 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.

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
Flammability testing	NR	NR	Yes[a]	YES	YES

NR = Characterization not required to meet objective.

[a] Hydrogen and methane only.

The objectives of RTR for the Program are the following:

1. If applicable, verify waste Content Code (Table 3-2); site-specific Item Description Code (IDC), as defined in Section 15.0; and RCRA Waste Category as defined in USDOE, 1992a.

2. Where applicable, provide an estimated inventory of the waste items, residual materials, and packaging materials and/or waste material categories.

3. If present, estimate the quantity of free liquids.

4. Provide the information necessary for making an estimate of waste items residual and packaging materials weight for homogeneous 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 WIPP bin-scale and alcove test phase (Molecke, 1990a, 1990b), 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, packaging materials, and residual materials to estimate weights of the waste material categories specified in Table 3-3. These data on standard weights should include weights of packaging and residual materials as well as the weights of standard waste items such as HEPA filters, Raschig rings, and Fulflo® filters. These data should also include information on the proportion of materials in composites such as leaded rubber, cemented process liquids, and sludges.

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.

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 estimated inventories of waste items and residual 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. The sites should document how accurately an operator can make these determinations. The precision and accuracy of weight data estimates shall be enhanced by utilizing standardized RTR procedures and operator qualifications.

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

*1XX is used to designate the Content Code of newly generated waste. 2XX is used to designate the Content Code of retrievably stored waste.

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

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

3.1.2 Completeness

An audio/videotape of the RTR examination and an RTR data form, validated according to the requirements in Section 8.0, must be obtained for 100% of the waste containers used in the Program.

3.1.3 Comparability

RTR data shall be used to verify previous process knowledge and compared with data from the visual examination. If RTR data are found to be comparable with visual data and process knowledge, then RTR may be used to limit the extent of the visual examination to characterize waste in particular Content Codes or Item Description Codes for the WIPP bin-scale and alcove tests.

3.2 Radioassay

The following three RA 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 indicates 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 WIPP 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 (Molecke, 1990a; Molecke and Lappin, 1990).

Each generator/storage facility must use one or more RA techniques. Each facility shall demonstrate and technically justify that any RA techniques used are appropriate for their specific installation. The basis for using a specific assay technique should be the physical form of the waste, the radionuclide content, and the 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 TRU content, and its distribution.

The accuracy, precision, and completeness quality assurance objectives to be used for the three identified RA techniques are summarized in Table 3-4. These objectives apply to wastes with homogeneous distributions, low neutron absorber and moderator content, and little self-shielding of the fissile sources. They are expected to be achievable in the presence of backgrounds generated by

Table 3-4. Quality Assurance Objectives for Radioassays

	SGS		PAN		PNCC[c]	
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		±15%		±30%		±30%
Completeness	100%		100%		100%	

[a] ±2 standard deviations, counting statistics only
[b] WG Pu = Weapons grade plutonium
[c] Values also apply to PAN passive mode

alpha and gamma emitting sources and in the presence of quantities of neutron and gamma absorbing and moderating material, as is the case for most waste encountered in this program.

3.2.1 Precision and Accuracy

The precision and accuracy of the measurement technique must be determined through replicate processing and comparison with calibration standards, 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

RA data shall be obtained for 100% of the waste containers used in the Program. RA data shall be validated according to the requirements in Section 8.0.

3.2.3 Comparability

If multiple systems are planned for use in this program, the DOE sites should perform multiple independent assays of a sample of waste containers. For example, these independent assays may include a passive gamma assay such as SGS followed by passive/active neutron assay. When multiple independent RAs are run on a waste container, the sites must document the comparability of the techniques.

3.3 Headspace Sampling

The TRU wastes to be emplaced in WIPP as part of the Test Phase can be packaged in 55-gallon drums or in bins. Pretest waste characterization for the bin-scale tests will include sampling and analysis of headspace gas from three areas within drums of TRU waste (see Figure 3-1): (1) the drum headspace (i.e., the headspace directly under the drum lid), (2) the 55-gallon poly bag headspace, and (3) the innermost layers of confinement headspace. Headspace gas sampling prior to supercompaction is not a Program requirement.

The results of analyses of headspace samples from within each containment area in a drum must be used to demonstrate compliance with the comparability requirements of the NMD (Section 1.3.2).

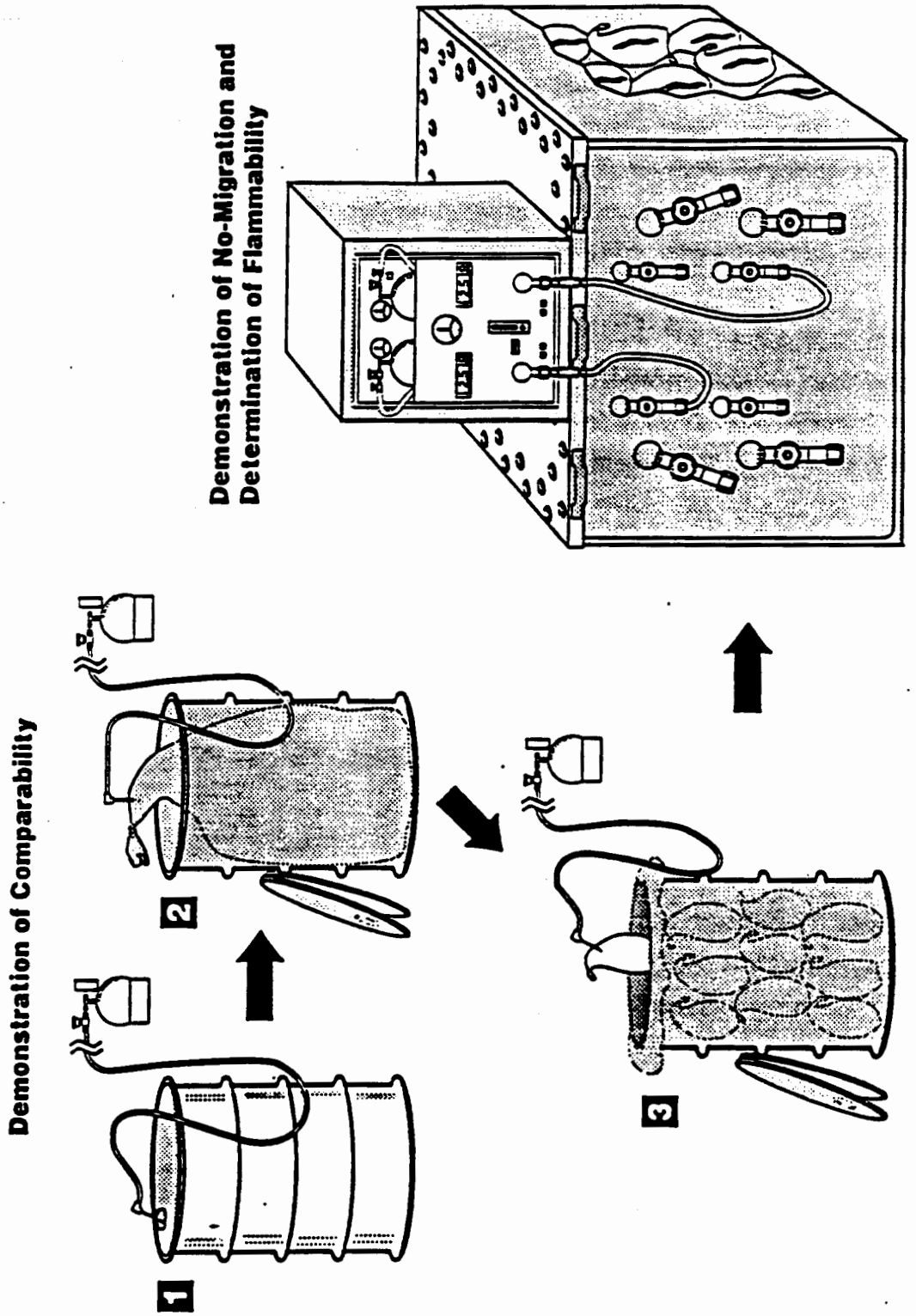


Figure 3-1. Overall Sampling Compliance Scheme

Bin headspace samples must also be obtained from within each loaded bin (see Figure 3-1). The results of analyses of headspace samples from within experimental bins must be used to demonstrate compliance with the no-migration requirements and the flammability testing requirements of the NMD. The following sections detail quality assurance objectives for drum and bin headspace sampling operations. Table 3-5 illustrates which classes of analytes are required for each type of sample collected. Section 9 specifies the frequency at which QC samples must be collected. In the following text, "drum" is used to represent all layers of confinement that must be sampled within a drum (i.e., directly under the drum lid, the 55-gallon poly-bag, and the innermost layers of confinement).

3.3.1 Drum Headspace Sampling

The precision and accuracy of the drum headspace sampling operations must be assessed by analyzing field QC headspace samples. These field QC headspace samples must include equipment 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 apparatus cleaning, must be taken.

3.3.1.1 Precision

The precision of the headspace sampling and analysis 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 ±25%; (2) for gases, the RPD exceeds ±10%; and (3) for NO_x, the RPD exceeds ±20%.

3.3.1.2 Accuracy

A field reference standard must be collected using the headspace sampling equipment to assess the relative percent accuracy (RPA) of the headspace sampling and analysis operation. Remedial action must be taken if (1) for VOCs, the RPA exceeds ±30%; (2) for gases, the RPA exceeds ±10%; and (3) for NO_x, the RPA exceeds ±50%.

3.3.1.3 Representativeness

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

- Headspace sample canister cleaning and leak check
- Headspace sampling equipment cleaning or disposal after use
- Headspace sampling equipment leak check

Table 3-5. Analyses Required For Each Type of Headspace Sample Collected

Waste Container	Sample Type	VOCs[1]	Gases[2]	NO_x	H_2,CH_4
Drum	Drum Lid	•			•
	55-Gallon Poly Bag	•	•	•	
	Innermost Layer of Confinement	•	•		
	Field Duplicate	•	•	•	
	Field Blank	•			
	Equipment Blank	•			
	Field Reference Standard	•	•		
Bin	Bin	•			•
	Field Duplicate	•			•
	Equipment Blank	•			
	Field Reference Standard	•			•

[1] See Table 3-6
[2] See Table 3-7, includes hydrogen and methane

- Use of headspace sample canisters with passivated internal surfaces
- Use of a low internal volume headspace sampling equipment
- Collection of small sample volume: low sample volume to available headspace volume ratio
- Careful pressure regulation
- Performance audits
- Collection of equipment blanks, field reference standards, field blanks, and field duplicates.

3.3.1.4 Completeness

Headspace samples must be collected from within all drums and associated inner 55-gallon poly bags. In addition, one hundred percent of the innermost layers of confinement containing a minimum of 1 liter of available headspace must be sampled. This volume is dictated by the analytical sample size requirement of 100 ml and the objective of not removing more than 10% of the available headspace. The headspace within rigid innermost layers of confinement (e.g., glass, metal, and rigid plastic containers) will not be sampled, but a headspace sample will be collected from the closest external non-rigid layer of confinement.

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 sufficient headspace samples are collected to meet the Program objectives. Site SOPs must address documenting any non-routine events or occurrences that may affect the quality of the headspace sample collected.

3.3.1.5 Comparability

Consistent use and application of uniform procedures and equipment (in accordance with USDOE, 1991c) should ensure that headspace sampling operations are comparable when sampling different layers of confinement as well as comparable at the different headspace sampling sites.

3.3.2 Bin Headspace Sampling

The field QC samples collected during bin headspace sampling operations must include equipment blanks, field reference standards, and field duplicates. Field blanks are not required during bin sampling operations since the bin sampling equipment is carefully sealed to the bin prior to sample collection, thus preventing ambient air intrusion.

3.3.2.1 Precision

The precision of the bin headspace sampling and analysis operation must be assessed by collecting field duplicates for VOC and gas determinations. Remedial action must be taken if (1) for VOCs, the RPD exceeds ±25%; (2) for gases, the RPD exceeds ±20%.

3.3.2.2 Accuracy

A field reference standard must be collected in the laboratory or field prior to bin headspace sample collection, through the bin sampling equipment to assess the RPA of the sampling and analysis operation. Remedial action must be taken if (1) for VOCs, the RPA exceeds ±30%; (2) for gases, the RPA exceeds ±10%.

3.3.2.3 Representativeness

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

- Recirculation of the headspace gases to achieve homogeneity prior to sampling
- Canister cleaning and leak check
- Equipment cleaning and leak check
- Use of sample canisters with passivated internal surfaces
- Collection of small volume samples: low sample volume to available headspace volume ratio
- Careful pressure regulation and flow control
- Performance audits
- Collection of bin headspace sampling equipment blanks, reference standards, and field duplicates.

3.3.2.4 Completeness

A bin headspace sample must be obtained from 100% of the bins to be used in the WIPP bin-scale test.

3.3.2.5 Comparability

Consistent use and application of uniform procedures and equipment (in accordance with USDOE, 1991c) should ensure that bin headspace sampling operations are comparable at the different sampling sites.

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 (Lappin et al., 1991). 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, determined during the visual examination, will be used as input to the gas generation behavior model.

3.4.1 Precision and Accuracy

The precision and accuracy of weight estimates obtained from visual examination are unknown at the present time. However, the quality of the visual examination data shall be enhanced by the use of standardized procedures and operator qualifications. 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. The precision and accuracy with which weight estimates can be made must be evaluated and reported.

3.4.2 Completeness

An audio/videotape of the visual examination and a visual examination data form, validated according to the requirements in Section 8.0, must be obtained for 100% of the waste containers used in the WIPP bin-scale test and for a representative portion (to be determined by Sandia National Laboratories) of the waste containers used during the WIPP alcove test.

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 DQOs 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. Program required limits, such as the PRDL associated with gas analysis and the Program Required

Quantitation Limit (PRQL) associated with VOC analysis, are specified by the Program to ensure that the analytical data collected satisfy the requirements of all data users. 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 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 and field duplicates and replicate analyses of laboratory control samples and PDP 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.

3.5.2 Accuracy

Accuracy is the degree of agreement between a measured analyte concentration (or the average of replicate measurements of a single analyte concentration) and the true or known 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.

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 MDL is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL will be expressed in nanograms for VOCs and volume percent for gases. The MDLs will be determined (Glaser, 1981) by

Table 3-6. VOC Quality Assurance Objectives

Compound	Precision [a] (%RSD or RPD)	Accuracy [a] (%R)	MDL (ng)	Completeness (%)
Benzene Bromoform 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, total number of nanograms delivered to the analytical system per sample)

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

Table 3-7. Gas Analysis Quality Assurance Objectives

Analyte Gases	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[a] (combined)		±20%	50-150%	0.01	90%
a. Nitric Oxide	10102-43-9				
b. Nitrogen Dioxide	10102-44-0				

a = Analyses of Nitrogen Oxides are not required for innermost layers of confinement.
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
PRDL = Program required detection limit

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 Table 3-6 and the PRDLs listed in Table 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 the collection of sufficient numbers of samples using a clean sampling manifold that does not introduce sample bias.

3.6 Flammability Testing

The quality assurance objectives for flammability testing are summarized in Table 3-9. Flammability testing uses a modified version of ASTM Method E 681-85 as a flammable/nonflammable test to evaluate the flammability of bin headspace samples.

3.6.1 Precision and Accuracy

Flammability test results are considered qualitative. Since qualitative evaluations do not lend themselves to statistical analysis, precision will be enhanced by requiring that sites use a standardized flame test apparatus and a standardized analytical method in conjunction with operator training. Duplicate samples will be collected for flame testing when sufficient bin headspace is available. The accuracy of each laboratory's flame test apparatus shall be assessed and qualified prior to the evaluation of samples and annually thereafter. A flame test apparatus is qualified when it successfully evaluates the flammability of each of the test mixtures (Table 6-3). Each flame test apparatus shall have an accuracy of 100% (Table 3-9).

3.6.2 Completeness

The measure of completeness for flammability testing shall be 100% (Table 3-9). Samples from all bins with headspace containing a summed concentration of flammable VOCs that cannot be shown to be below 500 ppmv shall be analyzed with valid results using the flame test apparatus.

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

Volatiles	CAS Number	PRQL[a] (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[b]	108-38-3	1
28. o-Xylene	95-47-6	1
29. p-Xylene[b]	106-42-3	1

[a] Values based on delivering 10 mL to the analytical system.
[b] These xylene isomers cannot be resolved by the analytical methods employed in this program.

Table 3-9. Flammability Testing Quality Assurance Objectives

Evaluation	Accuracy[a] (%)	Completeness[b] (%)
Flammability	100	100

[a] Accuracy expressed as,
(Number of test mixtures whose flammability is correctly evaluated/Total number of test mixtures evaluated by flammability testing specialist)*100

[b] Completeness expressed as,
(Number of bin headspace samples evaluated with valid data/Number of bin headspace samples requiring flammability testing)*100

3.6.3 Comparability

Comparability between sites shall be achieved by using a standardized flame test apparatus and a standardized analytical method. On a per sample basis, flame test results should be comparable to a theoretical flammability evaluation made using the Le Chatelier formula (Section 12.6). The procedure for performing a theoretical flammability evaluation is presented in Section 12.6 and detailed in ASTM E 681-85. Sites must provide this comparability data to DOE/WPSO even though the flame test is currently the only acceptable method of evaluating the flammability of bin headspace samples. DOE is collecting these data so that in the future, headspace flammability can be evaluated with a theoretical calculation.

3.6.4 Representativeness

The quality characteristic of representativeness as applied to flammability testing shall be achieved by the use of clean headspace sampling equipment that does not introduce sample bias and by proper sampling techniques.

4.0 SAMPLING PROCEDURES

The sampling requirements described in this section must be followed to collect representative samples from within TRU waste containers and bins. As required by ASME NQA-1, Element 5, detailed sampling procedures shall be issued as approved and controlled SOPs implementing the site QAPjP. 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. Sampling procedures can be found in the Guidance Manual (USDOE, 1991c).

All sampling must be accomplished within a radiation containment area. The configuration of the containment area at each 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 sampling location is also expected to differ. If applicable, descriptions of the remote handling equipment must be provided in the site QAPjP.

In the following text, "protocol" material constitutes the absolute minimum requirements for compliance with this program. "Guidance" material consists of recommended practices. "Procedures" consist 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.

Site SOPs must address documenting any non-routine events or occurrences that may affect the quality of the sample collected. Any deviations from the SOPs which implement the sampling protocol outlined in this section of the QAPP must be described in a Record of Variance, Figure 4-1. See Section 13.2 for site implementation. The site Project QA Officer shall evaluate the impact of the variance and notify the appropriate personnel.

Drums and their contents must be allowed to equilibrate to the temperature of the sampling area for a minimum of 72 hours prior to sampling.

To accomplish drum headspace sampling, two SUMMA® canister-based headspace sampling systems have been developed. The first of these uses a headspace sampling manifold, and is described in Section 4.1. The second, a direct canister headspace sampling system described in Section 4.2,

Sample Identification Number:	
Reason for deviation from 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 4-1. Record of Variance
EXAMPLE ONLY

employs a needle and filter attached directly to the SUMMA® sample canister. Generator storage sites have the option of using either the headspace sampling manifold or the direct canister methods, or a combination of these, for some or all layers of confinement and for collecting QC samples. Site QAPjPs must specify which apparatus is used. A SUMMA® canister based bin headpsace gas sampling protocol is described in Section 4.3.

4.1 Protocol for Drum Headspace Gas Sampling Through Manifold

This section describes minimum requirements for the collection of headspace samples through a sampling manifold (see 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 3-6 or a Program required subset of the listed analytes. "Headspace" should be interpreted to mean the VOCs and gas within a layer of confinement.

This 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. QC sample frequencies are specified in Section 9.0. Headspace samples for the determination of the analytes listed in Table 3-5 must include:

- Headspace samples from directly under the drum lid, the 55-gallon poly bag, and the innermost layers of confinement
- Field duplicate samples
- Field blank samples
- Equipment blanks
- Field reference standard samples.

Before collecting the first drum headspace sample, the headspace sampling manifold must be used to collect a field reference standard and then an equipment blank. The concentrations of analytes in these QC samples must be determined in order to evaluate whether the headspace sampling manifold can meet the quality assurance objectives specified in Section 3.3.

4.1.1 Scope and Application

The TRU wastes to be sent to the WIPP as part of the Test Phase are packaged in 55-gallon drums and can contain waste items, residual materials, and/or packaging materials confined by several layers of plastic bags (Figure 4-2). Given this waste packaging heterogeneity, the EPA is requiring that the headspace sample which the DOE collects from within 55-gallon drums be representative of the entire headspace of the drum. EPA expects that all layers of confinement (i.e., the drum, the 55-gallon poly bag, and the innermost layers of confinement) in a drum will have to be sampled until DOE can

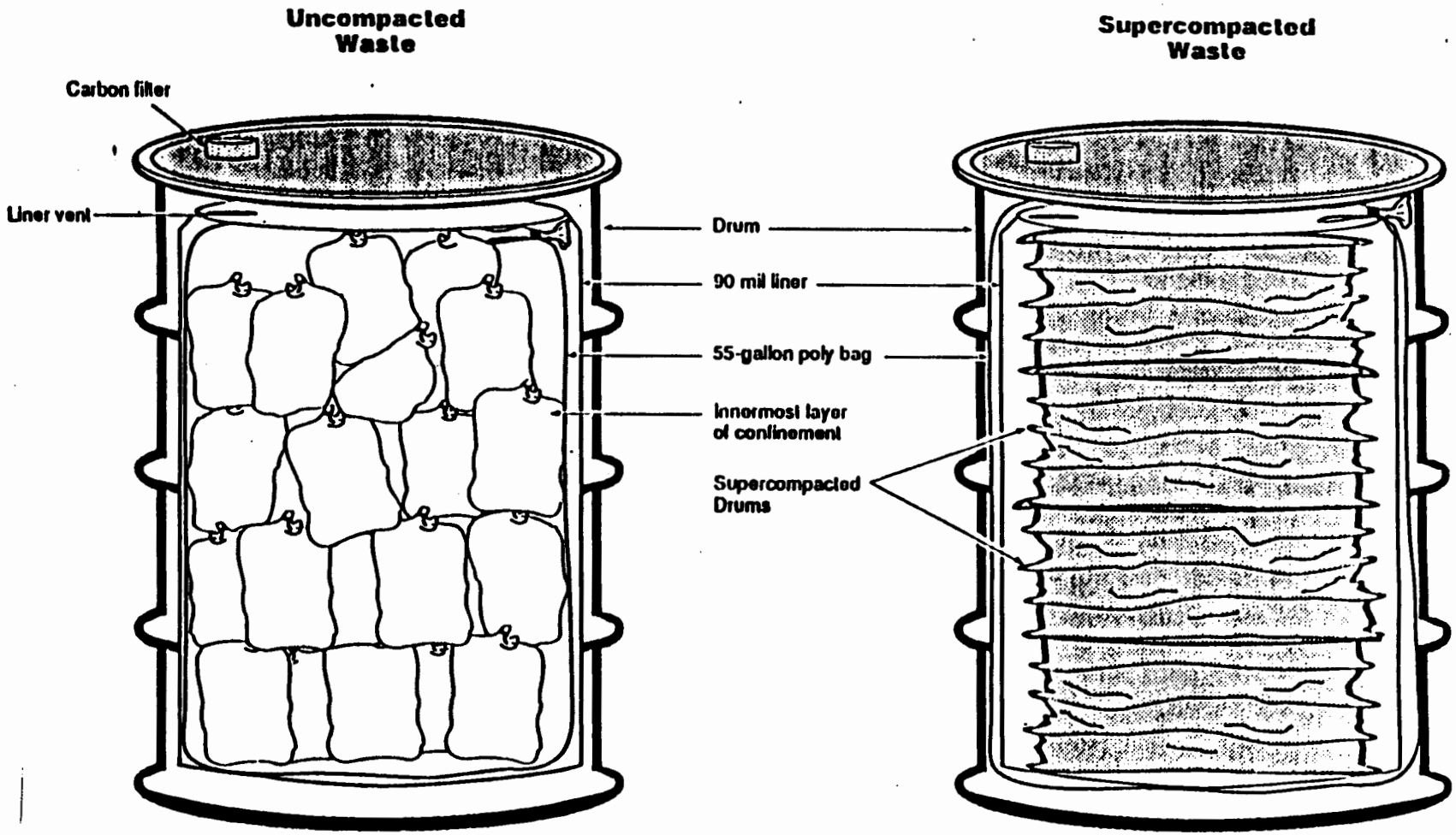


Figure 4-2. Schematic Diagram of Waste Drum with Minimum Layers of Confinement and Sampling Locations

demonstrate to the EPA, based on the data collected, that sampling of all layers is either unnecessary or can be safely reduced.

This protocol for sampling of the headspace within TRU 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 in TRU waste drums containing different waste types. 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

This section summarizes the general method for sampling using a manifold. Sub-sections describe aspects of the method unique to each layer of confinement within a drum.

All sampling equipment 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. Sample holding times and storage conditions must conform to the requirements specified in Table 4-1.

This headspace sampling protocol 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. The headspace sampling manifold and sample canisters must be evacuated to 0.05 mm Hg (canister gauge will read 30 inches Hg vacuum) 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 filter connected to different sampling heads depending on the layer of confinement to be sampled. The sampling head(s) must be capable of punching through the metal lid of the drum or penetrating a carbon composite filter, and puncturing poly bags.

The headspace sampling manifold must also be equipped with a purge assembly that allows equipment blanks, field reference standards, and field 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

Table 4-1. Headspace Sample Containers and Holding Times

Parameter	Container	Minimum Drum Headspace Sample Volume[a]	Holding Temperatures	Field Holding Time[b]	Shipping Allowance	Laboratory Holding Time[c]
Gases	SUMMA® Canister	100 milliliters	Ambient	4 days	2 days	28 days
VOCs	SUMMA® Canister	250 milliliters	0-40°C	4 days	2 days	28 days
NO_x[d]	SUMMA® Canister	100 milliliters	Ambient	4 days	2 days	28 days

[a] Alternatively, if available headspace is limited, a single 100 ml sample may be collected for determination of VOCs and gases.

[b] From time of headspace sample collection to shipment.

[c] This is a programmatic-based maximum holding time. Holding time begins at VTSR.

[d] A separate headspace sample will not be required if mass spectrometry is used for inorganic gas analysis. NO_x analysis is not required for samples collected from within innermost layers of confinement.

sampling area in the immediate vicinity of the layer of confinement to be sampled. Field blanks shall be collected by collecting a sample of room air through the headspace sampling manifold after the sampling manifold has been cleaned and verified to contain 1.0 ppm VOCs or less by using an organic vapor analyzer. The collection of a field blank through the headspace sampling manifold must be immediately preceded by the collection of an equipment blank.

The design of the headspace sampling manifold, associated sampling heads, and the headspace sample volume requirements ensure that a representative sample is collected. When an estimate of the available headspace volume can be made, less than 10% of that volume should be withdrawn. A determination of the headspace sampling manifold internal volume must be made and documented. The total volume of headspace gases collected during each headspace sampling operation can be determined by adding the combined volume of the sample canisters attached to the headspace sampling manifold to the internal volume of the headspace sampling manifold.

4.1.2.1 Drum Sampling

A sample of the drum headspace directly under the lid must be collected from within each drum. Two methods have been developed for collecting this sample in such a way that it is representative of the drum headspace. These two methods, summarized in this section, require specialized sampling heads compatible with the headspace sampling manifold.

Sampling through the Carbon Filter

To sample the drum headspace through the carbon filter, a side port needle (i.e., a hollow needle sealed at the tip with a small opening on its side close to the tip) must be pressed through the drum filter and into the headspace beneath the drum lid. This permits the gas to be drawn into the headspace sampling manifold. This procedure is described in detail in the Guidance Manual (USDOE, 1991c) and is specific to a type of carbon composite filter that permits insertion of the needle. To assure that the sample collected is representative, all of the general method requirements, sampling apparatus requirements, and quality control requirements described in this Section (4.1) must be met in addition to the following requirements which are pertinent to drum headspace sampling through the carbon filter:

- The lid of the drum's 90 mil liner must contain a hole for venting to the drum
- During sampling, the drum's carbon composite filter must be sealed to prevent outside air from entering the drum.

Sampling through the Drum Lid

Sampling through the drum lid, also described in the Guidance Manual (USDOE, 1991c), must be performed when the drum's carbon composite filter does not permit insertion of the side port needle. To sample the drum headspace through the drum lid, the lid must be breached using a sparkless punch and the headspace sample withdrawn to the headspace sampling manifold. A drum punch sampling system that forms an airtight seal between the drum lid and the sampling manifold must be used. To assure that the sample collected is representative, all of the general method requirements, sampling apparatus requirements, and quality control requirements described in this Section (4.1) must be met in addition to the following requirements which are pertinent to drum headspace sampling through the drum lid:

- The lid of the drum to be sampled should be relatively free of dents, scratches, and materials that may affect the drum punch seal to the drum lid and cause leaks to occur during sampling
- All components of the drum punch sampling system that come into contact with sample gases must be purged with humidified zero air prior to sample collection
- Equipment blanks and field reference standards must be collected through all components of the drum punch sampling system that contact the headspace sample
- Pressure must be applied to the sparkless punch until the drum lid has been breached, then the punch must be backed out to expose the headspace to the drum punch sampling system
- Provisions must be made to relieve potential drum pressure increases during drum punch operations. Pressure increases may occur during sealing of the drum punch sampling system to the drum lid
- The lid of the drum's 90 mil liner must contain a hole for venting to the drum
- During sampling, the drum's carbon composite filter must be sealed to prevent outside air from entering the drum.

4.1.2.2 55-Gallon Bag Sampling

Headspace samples must be collected from within the innermost 55-gallon poly bag drum liner after removing the drum lid. A headspace sample must be collected from the 55-gallon poly bag regardless of the condition (breached or torn) of the bag. Headspace gases from within the innermost 55-gallon poly bag must be drawn into the headspace sampling manifold through a needle that punctures the bag to access the bag's headspace.

Holding the needle in the headspace, the operator must open the inlet valve on the headspace sampling manifold to allow headspace to expand into the evacuated sample canister. Care should be taken to prevent placing the tip of the clean needle in an area where solid material or particulate matter might fill or clog the needle. Once the canister has filled, the operator must close the inlet valve and prepare the sample canister for shipment.

4.1.2.3 Innermost Layer Sampling

All of the innermost layers of confinement (i.e., the poly bag layer closest to the waste) that meet the following minimum criteria must be sampled:

- The innermost layer of confinement must have a minimum of 1 liter of headspace from which a representative 100 ml sample must be withdrawn. A 250 ml sample should be collected if there is enough available headspace gas (2.5 liter, minimum). When field duplicates are collected, twice the available headspace shall be present.

- Headspace contained within rigid innermost layers of confinement (e.g., glass, metal, and rigid plastic containers) will not be sampled, but the external poly bag closest to the rigid container must be sampled if a minimum of 1 liter of headspace is available.

The sampling operator must determine and document in writing, in accordance with site SOPs, which innermost layers of confinement are eligible for headspace sampling (see Section 3.3). This documentation must include the innermost layer of confinement identification number (Section 5.4) and estimated available headspace volume (less than 1 liter, greater than 1 liter, or greater than 2.5 liters). If the estimated available headspace volume is less than 1 liter and therefore ineligible for headspace sample collection, then site SOPs must address documenting, in writing, this information.

To accomplish innermost layer of confinement sampling, the operator must first determine whether to use a 100 ml or a 250 ml sample canister. If the innermost layer of confinement to be sampled contains at least 1 liter but not more than 2.5 liters of available headspace, then a 100 ml sample canister must be used. If the innermost layer of confinement to be sampled contains greater than 2.5 liters of available headspace, then a 250 ml sample canister should be used. In all cases, the maximum allowable sample volume should be collected. Once the appropriate sample canister size has been determined, the operator can sample in the same manner as for the 55-gallon poly bag.

4.1.3 Sampling Apparatus

The following sections describe the equipment required to collect headspace samples from within drums of TRU waste. The headspace sampling manifold (Section 4.1) and SUMMA® sample canisters (Section 4.1) are used when sampling each area of confinement in the drum. Three different sampling

heads are described for use with the manifold, allowing sampling through the drum lid carbon composite filter, through the drum lid itself, and through the poly bags inside the drum, both 55-gallon and innermost.

4.1.3.1 Headspace Sampling Manifold

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

- Applicable sampling head that forms a leak tight connection with the headspace sampling manifold
- A flexible hose that will allow movement of the sampling head from the purge assembly (pressurized line) to the waste container
- Headspace sampling manifold pressure sensor(s) that must be pneumatically connected to the headspace sampling manifold. The headspace sampling manifold pressure sensor(s) must be able to measure absolute pressure in the range from 0.05 mm Hg to 1000 mm Hg. Resolution must be ±0.005 mm Hg at 0.05 mm of Hg. The headspace sampling manifold pressure sensor(s) must have an operating range from approximately 15°C to 40°C
- 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. Ports must have VCR® fittings for connection to the sample canister(s)
- 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
- 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 tubing should be 1/8-inch.
- An organic vapor analyzer (OVA) capable of detecting all analytes listed in Table 3-6. The OVA must be capable of measuring total VOC concentrations as low as 0.1 ppm. Detection of 1,1,2-trichloro-1,2,2-trifluoroethane may not be possible if a photoionization detector is used. The OVA measurement must be confirmed by the collection of equipment blanks at the frequency specified in Section 9.0 to check for headspace sampling manifold cleanliness.

Under Revision

Figure 4-3. Headspace Sampling Manifold Schematic

The pressurized line must consist of the following major elements:

- A cylinder of compressed zero air and a cylinder of helium or nitrogen to clean the headspace sampling manifold between headspace samples and provide gas for the collection of equipment blanks. These high purity gases must be certified by the manufacturer to contain less than 1 ppm total VOCs. The gases must be metered into the pressurized line of the headspace sampling manifold by two-stage stainless steel regulators. Alternatively, a zero air generator may be used provided a sample of the zero air is collected and demonstrated to contain less than 1 ppm total VOCs.

- 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 line 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 equipment blanks and field reference standards and to assist with system cleaning between headspace sample collection

- A purge assembly which allows the sampling head (evacuated line) to be connected to the pressurized line 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 cleaning activities and during equipment blank and field reference standard collection. Maintaining excess flow will prevent ambient air from contaminating the equipment 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 line and a pressurized line, the area in which the headspace sampling manifold is operated should contain sensors for measuring ambient pressure and ambient temperature, as follows:

- The ambient pressure sensor should have a full range of at least 500 to 800 mm Hg and should be kept in the sampling area during sampling operations. Its resolution should be 1.0 mm Hg and calibration should be based on NIST standards. A temperature sensor should be near the ambient pressure sensor for temperature corrections, if necessary

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

4.1.3.2 Headspace Sample Canister

Samples must be collected in the SUMMA® passivated 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. Based on data obtained on ambient air samples and mixed gas standards, it is anticipated that headspace samples will be stable longer than the specified holding times. The programmatic 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. NO_x and gas samples may be kept 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 requirements 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. All sample canisters must have VCR® fittings for connection to sampling and analytical equipment. This type of vessel has been used for VOC sample collection with good 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). If the available headspace is limited, 100 mL samples may be collected for the determination of VOCs and gases.

A spiral bourdon-tube type dial pressure/vacuum gauge should be mounted on each headspace sample canister. The vacuum/pressure gauge will indicate if a headspace sample canister has a substantial leak and will ensure that a clean headspace sampling manifold is not exposed to a headspace sample canister that has leaked. This type of gauge should 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 should be able to indicate from 30 psig pressure to a vacuum of 30 inches mercury. Canister gauges are intended to be gross leak-detection devices not vacuum certification devices.

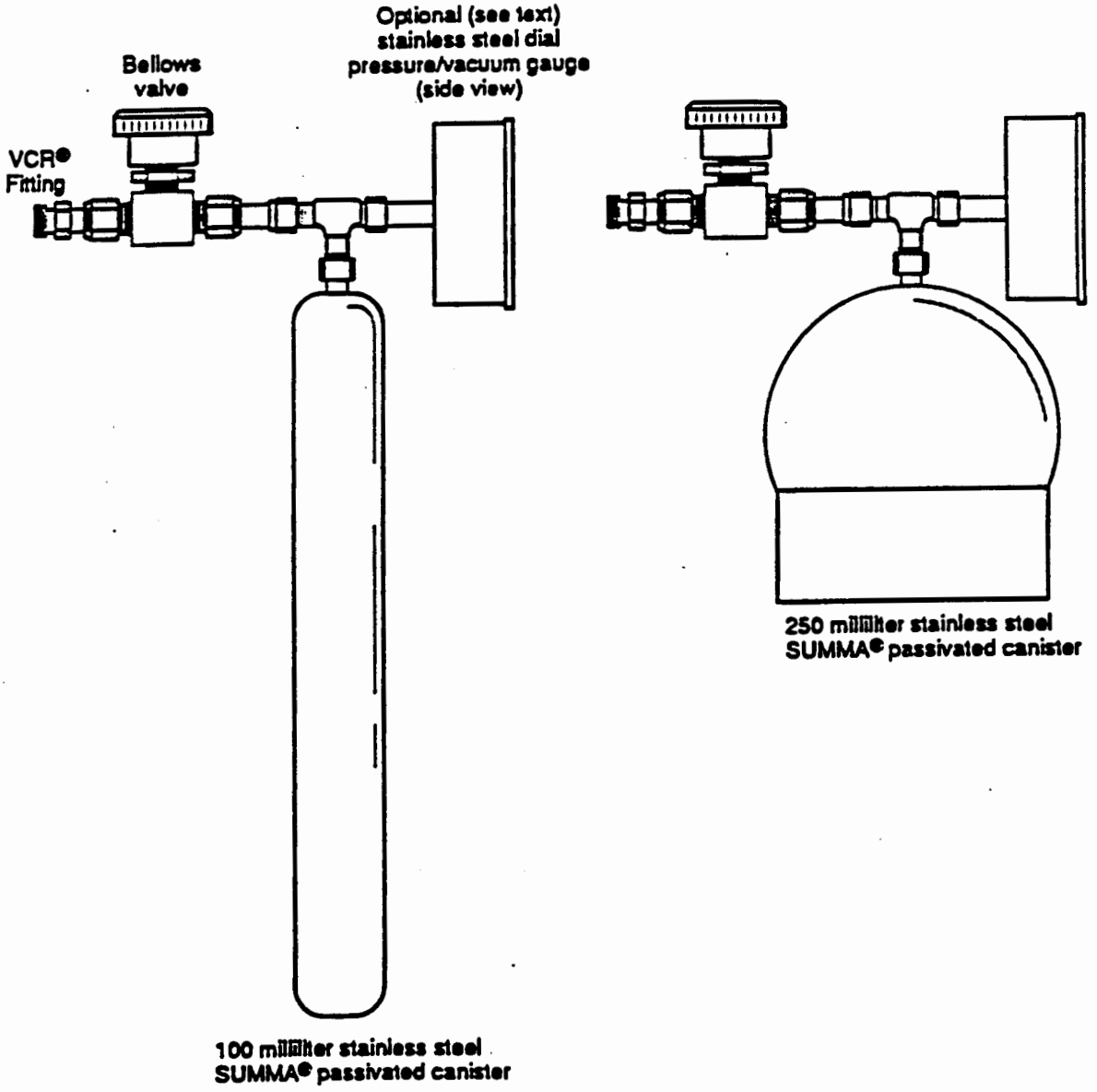


Figure 4-4. SUMMA® Canister Components Configuration (Not to Scale)

4.1.3.3 Apparatus for Sampling through the Carbon Filter

The sampling head for collecting drum headspace by penetrating the carbon composite filter must consist of a side port needle (i.e., a hollow needle sealed at the tip with a small opening on its side close to the tip), a filter to prevent particles from contaminating the headspace sample, and an adapter to connect the two. To prevent cross contamination, the sampling head must be cleaned or replaced after headspace sample collection, after field reference standard collection, and after field blank collection. The following requirements must also be met:

- The housing of the carbon composite filter must allow insertion of the sampling needle through the filter element into the drum headspace.
- The side port needle must be used to reduce the potential for plugging.
- The headspace sampling manifold purge assembly must be modified for compatibility with the side port needle.

4.1.3.4 Apparatus for Sampling through the Drum Lid

Sampling through the drum lid must be accomplished using the drum punch sampling system described in detail in the Guidance Manual (USDOE, 1991c), or equivalent. This system must be sealed to the drum lid and must consist of components that permit a sparkless punch to breach the drum lid and expose the drum headspace to the drum punch sampling system. The same type of sampling head as used for the 55-gallon poly bag sampling must be pneumatically connected to the drum punch sampling system to provide a seal between the drum lid and the headspace sampling manifold. The following requirements must also be met:

- A flow indicating device to verify excess flow of quality control gases must be pneumatically connected downstream of the drum punch sampling system.
- Equipment to adequately secure the drum punch sampling system to the drum lid.
- The punch must be made of non-sparking materials sufficiently hard to puncture the drum lid. Provisions must be made to prevent the punch from rotating as it is pressed through the drum lid.
- The drum punch sampling system must provide an airtight seal between the drum lid and the sampling manifold.

4.1.3.5 Apparatus for Sampling Through 55-gallon and Innermost Poly Bags

The sampling head for 55-gallon and innermost poly bag sampling must allow the bags to be pierced to gain access to their headspace. The head must contain a filter to prevent particulates from contaminating the headspace sample. To prevent cross contamination, the head must be cleaned or

replaced after headspace sample collection, after field reference standard collection, and after field blank collection.

4.1.4 Quality Control Measures

To minimize the potential for cross contamination of samples the headspace sampling manifold and sample canisters must be properly cleaned and leak-checked prior to headspace sampling. Procedures for cleaning and preparing the headspace sampling manifold and sample canisters are provided in the Guidance Manual (USDOE, 1991c). Cleaning requirements are presented below.

4.1.4.1 Headspace Sample Canister Cleaning

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

4.1.4.2 Sampling Equipment Initial Cleaning and Leak-Check

The surfaces of all headspace sampling equipment components that will come into contact with headspace must be thoroughly inspected and cleaned prior to assembly.

The headspace sampling manifold and associated sampling heads must be purged with humidified zero air, nitrogen, or helium and leak-checked after assembly. This cleaning must be repeated if the headspace sampling manifold and/or associated sampling heads are contaminated to the extent that the routine system cleaning is inadequate.

4.1.4.3 Sampling Equipment Routine Cleaning and Leak-Check

The headspace sampling manifold and associated sampling heads which are reused must be cleaned and checked for leaks after headspace sample and field duplicate collection; after field blank collection, if the field blank is collected through the headspace sampling manifold; and after the additional cleaning required for field reference standard collection has been completed (see next section).

The protocol for 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 sampling head be attached to the purge assembly. Humidified zero air, nitrogen, or helium, regulated through the purge assembly, must then be swept through the evacuated line of the headspace sampling manifold.

In addition to sweeping the evacuated line of the headspace sampling manifold, it is recommended that the headspace sampling manifold be heated and periodically evacuated and flushed with humidified zero air, nitrogen, or helium. Each cleaning must remove VOCs from the internal surfaces of the headspace sampling manifold to levels that are or less than the PRQLs of the analytes listed in Table 3-8, as determined by analysis of an equipment blank or the OVA. When not in use, the headspace sampling manifold must be demonstrated clean before storage with a positive pressure of high purity gas (i.e., helium, nitrogen, or zero air) in both the pressurized and evacuated lines.

Sampling must be suspended and corrective actions must be taken when the analysis of an equipment blank indicates these limits have been exceeded. The site Project Manager must insure that corrective action has been taken prior to resumption of sampling.

4.1.4.4 Manifold Cleaning After Field Reference Standard 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 line 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 sampling head, between the flexible hose and the purge assembly. This configuration allows both the evacuated and pressurized line of the headspace sampling manifold to be flushed (evacuated and pressurized) with humidified zero air, nitrogen, 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.

4.2 Protocol for Drum Headspace Sampling by Direct Canister

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 3-6 or a Program required subset of the listed analytes. "Headspace" should be interpreted to mean the VOCs and gas within a layer of confinement.

This section specifies requirements for the collection of headspace samples using the direct canister sampling apparatus from three areas within a TRU waste drum (see Figure 4-2). The three areas are:

- The drum headspace; that is, the headspace directly under the drum lid
- The 55-gallon poly bag headspace
- The innermost layers of confinement headspace.

This 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. QC sample frequencies are specified in Section 9.0. Headspace samples for the determination of the analytes listed in Table 3-5 must include:

- Headspace samples from directly under the drum lid, the 55-gallon poly drum liner bag, and the innermost layers of confinement poly bags
- Field duplicate samples
- Field blank samples
- Equipment blanks
- Field reference standard sample.

Before collecting the first headspace sample, the sampling apparatus described in this section must be used to collect an equipment blank and a field reference standard. The concentrations of analytes in these QC samples must be determined in order to evaluate whether the direct canister method can meet the quality assurance objectives specified in Section 3.3.

4.2.1 Scope and Application

The scope and applicability of the direct canister sampling protocol is identical to that of the manifold sampling protocol described in Section 4.1.

4.2.2 Method Summary

This headspace sampling protocol employs a canister sampling system to collect headspace samples for analysis and quality control purposes without the use of the headspace sampling manifold described in Section 4.1. Rather than attaching the sampling heads discussed in Section 4.1 to a manifold, in this method the sampling heads are attached directly to an evacuated sample canister.

All sampling equipment components that come into contact with headspace samples must be constructed of relatively inert materials such as stainless steel or Teflon®. A passivated interior surface of the stainless steel components is recommended. Sample holding times and storage conditions must conform to the requirements specified in Table 4-1.

Sample canisters must be evacuated to 0.05 mm Hg prior to use and attached to a changeable filter connected to different sampling heads depending on the layer of confinement to be sampled. The sampling head(s) must be capable of punching through the metal lid of the drums or penetrating a carbon composite filter, and puncturing poly bags. Field duplicates must be collected in the same manner and using the same type of sampling apparatus as used for headspace sample collection. Field

blanks must be samples of room air collected in the headspace sampling area in the immediate vicinity of the waste drum prior to removal of the drum lid. Equipment blanks and field reference standards must be collected using a purge assembly equivalent to the pressurized line of the headspace sampling manifold described in Section 4.1.3.1. These samples must be collected from the needle tip through the same components (e.g., needle, filter) that the headspace sample passes through.

The sample canisters, associated sampling heads, and the headspace sample volume requirements ensure that a representative sample is collected. When an estimate of the available headspace volume can be made, less than 10% of that volume should be withdrawn. A determination of the sampling head internal volume must be made and documented. The total volume of headspace gases collected during each headspace sampling operation can be determined by adding the volume of the sample canister(s) attached to the sampling head to the internal volume of the sampling head. Every effort should be made to minimize the internal volume of the sampling heads.

The following sections describe specific aspects of the method pertinent to sampling each area of confinement within a drum. Section 4.2.3 describes the sampling apparatus for each area of confinement and Section 4.2.4 describes the associated sampling quality control measures.

4.2.2.1 Drum Sampling

A sample of the drum headspace directly under the lid must be collected from within each drum. As in Section 4.1, two methods have been developed for collecting this sample in such a way that it is representative of the drum headspace. These two methods, summarized in this section, require that specialized sampling heads be directly attached to each sample canister, as appropriate for the layer of confinement to be sampled.

Sampling through the Carbon Filter

To sample the drum headspace through the carbon filter, a side port needle (i.e., a hollow needle sealed at the tip with a small opening on its side close to the tip) must be pressed through the drum filter and into the headspace beneath the drum lid. This permits the gas to be drawn into the evacuated canister. This procedure is specific to a type of carbon composite filter that permits insertion of the needle. To assure that the sample collected is representative, the sampling apparatus requirements and quality control requirements specified in this Section (4.2) must be met in addition to the following requirements which are pertinent to drum headspace sampling through the carbon filter:

- The lid of the drum's 90 mil liner must contain a hole for venting to the drum
- The drum's carbon composite filter must be sealed to prevent outside air from entering the drum during sampling.

Sampling through the Drum Lid

Sampling through the drum lid must be performed when the drum's carbon composite filter does not permit insertion of the side port needle. To sample the drum headspace through the drum lid, the lid must be punctured using a sparkless punch and the headspace gas withdrawn to an evacuated sample canister. A drum punch sampling system that forms an airtight seal between the drum lid and the sample canister, as described below, must be used. To assure that the sample collected is representative, the sampling apparatus requirements and quality control requirements described in this Section (4.2) must be met in addition to the following requirements which are unique to drum headspace sampling through the drum lid:

- The lid of the drum to be sampled should be relatively free of dents, scratches, and materials that may affect the drum punch seal to the drum lid and cause leaks to occur during sampling
- All components of the drum punch sampling system that come into contact with sample gases must be purged with humidified zero air, nitrogen, or helium prior to sample collection
- Equipment blanks and field reference standards must be collected through all components of the drum punch sampling system that contact the headspace sample
- Pressure must be applied to the sparkless punch until the drum lid has been punctured, then the punch must be backed out to expose the headspace to the drum punch sampling system
- Provisions must be made to relieve potential drum pressure increases during drum punch operations. Pressure increases may occur during sealing of the drum punch sampling system to the drum lid
- The lid of the drum's 90 mil liner must contain a hole for venting to the drum
- The drum's carbon composite filter must be sealed to prevent outside air from entering the drum during sampling.

4.2.2.2 55-Gallon and Innermost Poly Bag Sampling

After removing the drum lid, headspace samples must be collected from within the innermost 55-gallon poly liner bag and the innermost layers of confinement (i.e., the poly bag layer closest to the waste). A headspace sample must be collected from the 55-gallon poly bag regardless of the condition (breached or torn) of the bag. Headspace samples must be collected from within all innermost layers

of confinement that meet the criteria specified in Sections 3.3 and 4.1. Headspace gases from within these poly bags must be drawn into an evacuated canister through a needle that is punctured through the bag and into the headspace.

Holding the needle in the headspace, the operator must open the canister inlet valve to allow headspace to expand into the evacuated sample canister. Care should be taken to prevent placing the tip of the clean needle in an area where solid material or particulate matter might fill or clog the needle. Once the canister has filled, the operator must close the inlet valve and prepare the sample canister for shipment.

4.2.3 Sampling Apparatus

The following sections describe the direct canister sampling equipment required to collect headspace samples from within drums of TRU waste. Each sample canister used with this method must have a dial pressure/vacuum gauge able to indicate from 30 psig pressure to a vacuum of 30 inches Hg. Canister gauges are intended to be gross leak-detection devices not vacuum certification devices. Prior to sampling, canisters must be evacuated to 0.05 mm Hg and the canister gauge must indicate 30 inches of Hg vacuum. This 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 SUMMA® sample canisters must be used when sampling each area of confinement in the drum. Three different sampling heads are described for attachment to the sample canister. These heads must form a leak-tight connection with the sample canister and allow sampling through the drum lid carbon composite filter, through the drum lid itself and through the poly bags, both 55-gallon and innermost. Figure 4-5 illustrates the direct canister sampling equipment with the poly bag sampling head attached.

4.2.3.1 Apparatus for Sampling through the Carbon Filter

The sampling head for collecting drum headspace by penetrating the carbon composite filter must consist of a side port needle (i.e., a hollow needle sealed at the tip with a small opening on its side close to the tip), a filter to prevent particles from contaminating the headspace sample, and an adapter to connect the two. To prevent cross contamination, the sampling head must be cleaned or replaced after headspace sample collection, after field reference standard collection, and after field blank collection. The following additional requirements must also be met:

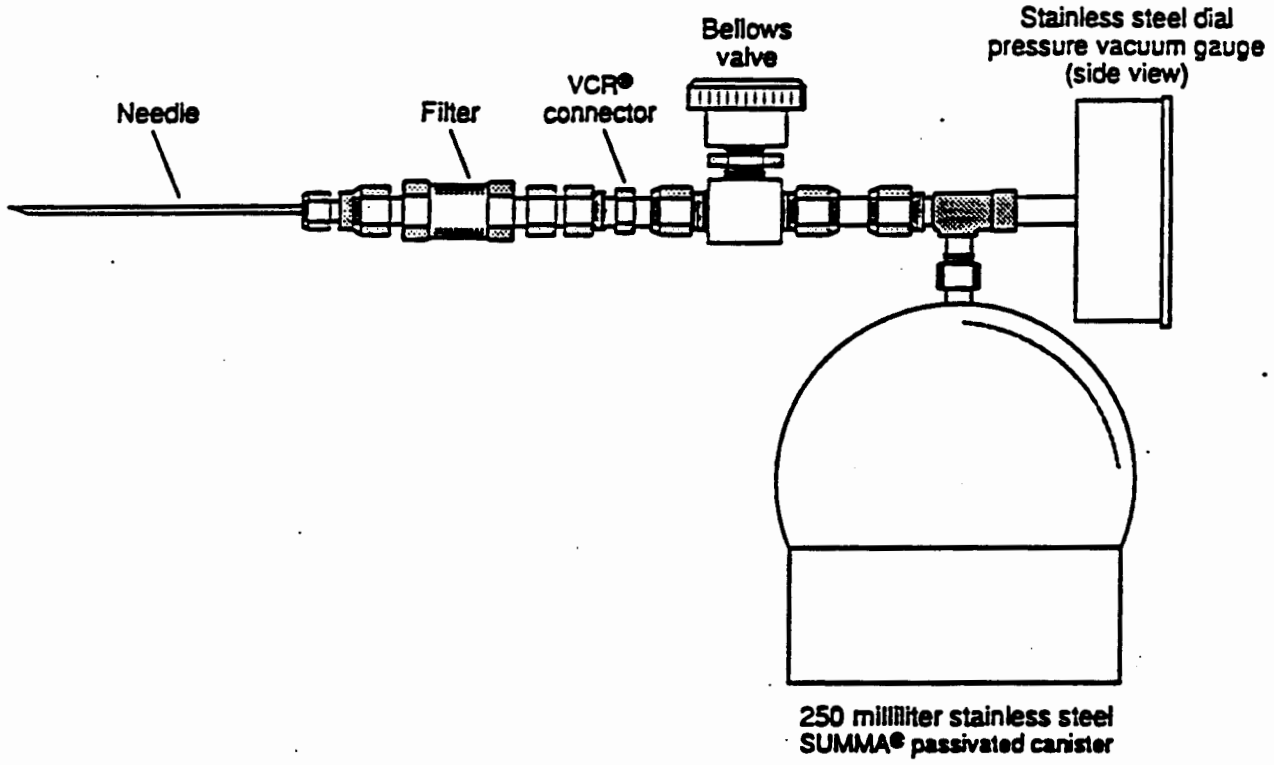


Figure 4-5. Schematic Diagram of Direct Canister Sampling Equipment

- The housing of the carbon composite filter must allow insertion of the sampling needle through the filter element into the drum headspace.
- The side port needle must be used to reduce the potential for plugging.
- The headspace sampling manifold purge assembly must be modified for compatibility with the side port needle.

4.2.3.2 Apparatus for Sampling through the Drum Lid

Sampling through the drum lid must be accomplished using the drum punch sampling system described in detail in the Guidance Manual (USDOE, 1991c), or equivalent. This system must be sealed to the drum lid and must consist of components that permit a sparkless punch to breach the drum lid and expose the drum headspace to the drum punch sampling system. The same sampling head as used for the 55-gallon poly bag sampling must be pneumatically connected to the drum punch sampling system to provide a seal between the drum lid and the direct canister sampling equipment. The following additional requirements must also be met:

- A flow indicating device to verify excess flow of quality control gases must be pneumatically connected downstream of the drum punch sampling system.
- Equipment to adequately secure the drum punch sampling system to the drum lid.
- The punch must be made of non-sparking materials sufficiently hard to puncture the drum lid. Provisions must be made to prevent the punch from rotating as it is pressed through the drum lid.
- The drum punch sampling system must provide an airtight seal between the drum lid and the sampling manifold.

4.2.3.3 Apparatus for Sampling Through Poly Bags

Figure 4-5 shows a stainless steel needle fitted into a reducer, a 0.5 μm filter, and a VCR® fitting that allows the filter to be connected to the 1/4-inch port of the SUMMA® canister's valve. Both the port connector and the reducer are made of stainless steel and have internal volumes of approximately 0.5 ml.

The needle, used to puncture the layer of confinement during headspace sampling, is 1/8-inch stainless steel tubing, approximately 5 inches long and sharpened at the end. It has an inner diameter of approximately 0.1 inch with an estimated internal volume of approximately 0.2 ml. The filter is a particulate filter containing a 0.5 μm sintered stainless steel fritt and has 1/4-inch fittings on both its inlet and outlet. The filter housing is made of stainless steel and is estimated to have an internal volume of approximately 4 ml. The combined internal volume of these components is approximately

5 ml. This low internal volume, approximately 2 percent of the sample volume, should ensure a representative headspace sample is collected.

4.2.4 Quality Control Measures

To prevent cross contamination, the needle, adapters, and filter of the direct canister apparatus must be disposed of or, if reused, cleaned between sample collections. As a further QC measure, the needle and filter, after cleaning, should be purged with zero air, nitrogen, or helium and capped for storage to prevent sample contamination by VOCs potentially present in ambient air. Field duplicates must be collected to assess sampling and analytical precision. Sample canisters must be cleaned, leak-checked, and evacuated in accordance with the requirements in Section 7.4.

4.3 Protocol for Bin Headspace Sampling

This section describes the requirements for the collection of headspace samples from a bin containing TRU wastes. This protocol is designed to ensure that representative headspace samples, including quality control samples, are consistently collected. QC sample frequencies are specified in Section 9.0. The bin samples for determination of the analytes listed in Table 3-5 must include the following:

- Headspace samples from within the bin
- Field Duplicates
- Equipment Blanks
- Field Reference Standards.

4.3.1 Scope and Application

During the WIPP Test Phase, DOE Plans to conduct bin-scale experiments in the WIPP to assess the rates and potential for gas generation that may influence the long-term integrity of the repository. At the generator/storage facilities, four to six drums of TRU wastes will be emptied into each test bin and placed in a WIPP standard waste box (SWB). The experimental bin must be sampled at the generator/storage sites to demonstrate compliance with conditions specified in the NMD as described in Section 1.3. Bin headspace sampling for assessment of compliance with the NMD requirements shall not be done until all drum data have been received by the site Project Manager. The site Project Manager shall notify project personnel when bin headspace sampling can be initiated.

4.3.2 Method Summary

The bin sampling system must be used to recirculate and homogenize bin headspace gas at a preset flow rate for a predetermined length of time. The flow rate and recirculation time must ensure that a minimum of two bin void volume equivalents are recirculated prior to sample collection. The bin void

volume will be a function of waste type, packaging configuration and number of drums needed to load the bin (USDOE, 1991c).

Six-liter bin headspace sample(s) must be collected in evacuated (0.05 mm Hg) SUMMA® passivated canister(s) after the recirculation time has elapsed. The bin sampling system must also pressurize the sample canister(s) to a final pressure of approximately 15 psig. During sample collection, the bin headspace must be continuously recirculated while approximately 10% of the recirculating gas is directed to the sample canister (20% for duplicate collection). Sample holding times and storage conditions must conform to the requirements specified in Table 4-1.

For field duplicate sample collection, a total volume of 24 liters is withdrawn from the bin into two six-liter SUMMA® canisters by pressurizing the canisters to 15 psig. During recirculation and duplicate sample collection, redundant pressure/vacuum relief controls must be provided since the bin must not be subjected to a positive pressure greater than 5.0 psig or a negative pressure less than minus 5.0 psig (Halvarson, 1991).

4.3.3 Sampling Apparatus

The bin headspace sampling equipment, shown in Figure 4-6, must consist of the following components:

- Flow regulating indicating device(s) upstream of the sample canister(s) capable of regulating and indicating the flow rate of gases through the recirculation loop
- Additional flow controlling device(s) capable of adjusting the sample canister fill rate(s) independent of the recirculation loop flowrate.
- Pneumatic recirculation loop and valves constructed of relatively inert materials such as stainless steel, Viton® or Teflon®. A passivated interior surface on the stainless steel components is recommended
- An oil free pump capable of circulating bin headspace at a minimum rate of approximately 500 ml/min and capable of pressurizing the sample canister(s) to a pressure of 15 psig
- Redundant bin pressure regulating equipment capable of maintaining the internal bin pressure between a minimum of minus 5.0 psig and a maximum of 5.0 psig
- SUMMA® passivated sample canister(s)
- Vacuum/pressure gauge(s) capable of measuring sample canister pressure from 30 in. Hg vacuum to a pressure of at least 30 psi
- Timer capable of controlling, manually or automatically, the recirculation duration

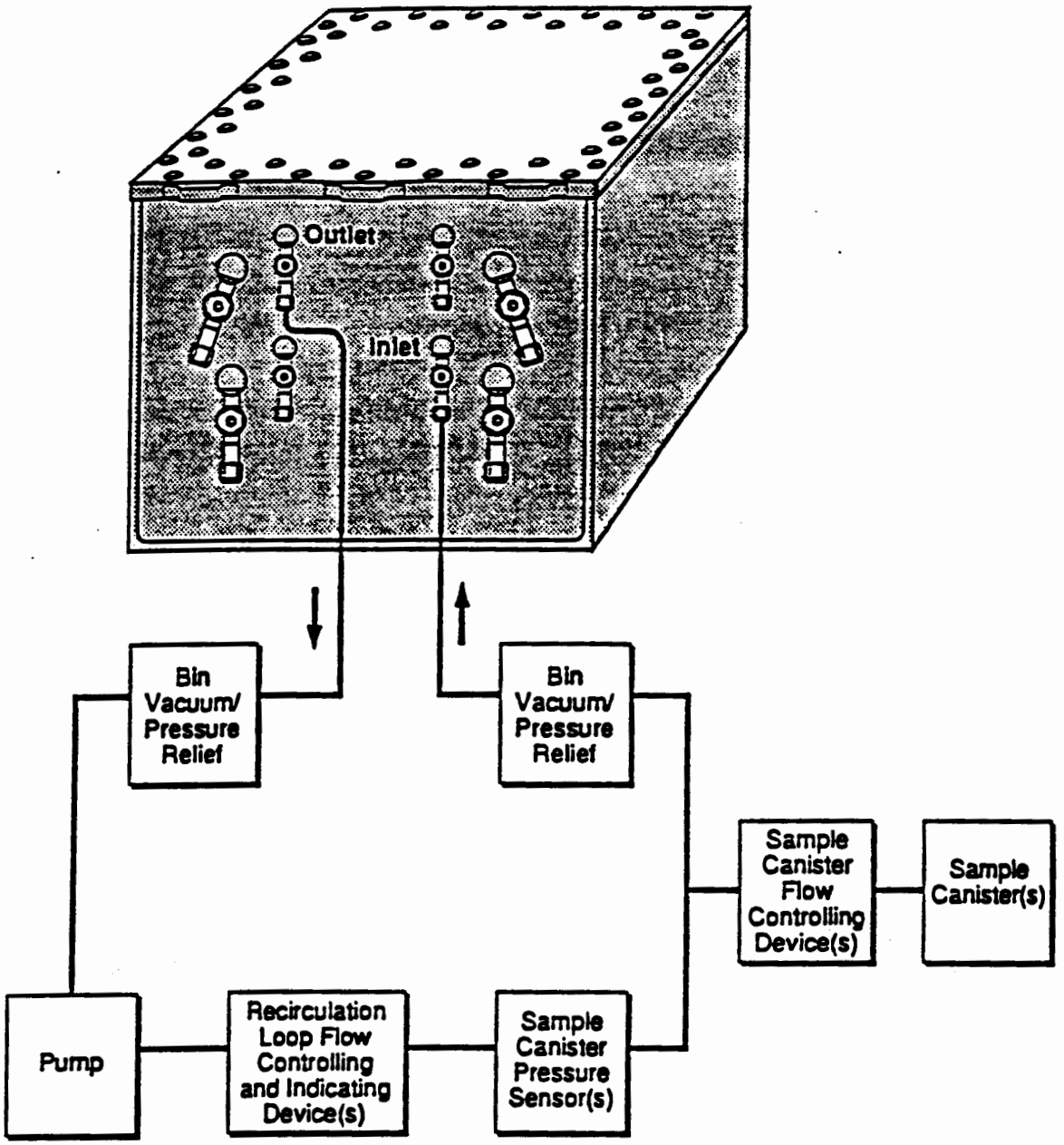


Figure 4-6. Bin Headspace Sampling Equipment Schematic

- The bin headspace sampling equipment should also include ambient pressure and temperature sensors (see Section 4.1.3.1).

4.3.4 Quality Control Measures

Quality control measures must include cleaning and leak checking the bin sampling system. The system must be cleaned by purging with humidified zero air or nitrogen. After purging, a sample of the purge gas, an equipment blank, must be collected through the system and analyzed for the Program required analytes. System cleanliness may also be checked by purging with zero air and monitoring the zero air with an OVA. However, the purge gas used to clean the system must be consistent with the balance of the OVA calibration gas.

The system must also be checked for leaks after each cleaning and prior to use. This must be performed by pressurizing the system with zero air or nitrogen and checking for leaks by monitoring the internal pressure or a soap bubble-type check.

Field reference standards and field duplicates must be collected in accordance with the frequencies specified in Table 9-2. A field reference standard (pressurized cylinder gas containing known analytes at known concentrations) must be collected using the bin headspace sampling apparatus. The results of the analysis of this sample indicate the efficiency of sampling and analytical recovery. Provided the pressure differential across the bin does not exceed a minimum of minus 5.0 psig and a maximum of 5.0 psig, field duplicates must be collected.

5.0 SAMPLE CUSTODY

In accordance with ASME NQA-1, Elements 8 and 13, this section provides identification and control of samples, waste containers, 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 and defensibility, chain of custody (COC) must be maintained and documented for waste containers, 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 container, 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 containers and bins will be considered under effective custody control if they are sealed (i.e., unopened waste containers 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 waste containers, 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. Copies of the original COC forms shall be included in the bin case

report provided to WPSO. The original COC forms shall be maintained in the site project files, unless DOE/WPSO request them.

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

5.1 Waste Container COC

COC on individual waste containers shall be initiated at the time the waste containers are removed from stored inventories. Waste container custody must be maintained until the waste container's contents are placed in a bin or until the waste container is removed from the Test Program as a result of failing to meet program requirements. Custody records must document which waste containers were emplaced in each bin. An example waste container COC form is provided in Figure 5-2. Site QAPjPs must include copies of forms used to document waste container 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 waste container numbers for each waste container under custody;
- Signatures of custodians relinquishing and receiving custody, along with the date and time of the transfer;
- Description of final waste container disposition, along with signature of individual removing waste container 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;

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			

Comments (note any discrepencies): _____

_____

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

- Signatures of custodians relinquishing and receiving custody, along with date and time of the transfer; and
- Comment section.

5.3 Bin COC

COC for bins shall be initiated upon introduction of wastes to the bin. Bin custody must be maintained until the bin is loaded in a TRUPACT-II 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 waste containers, 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.

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

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.

5.4.1 Gas 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 canister prior to shipment to the field. 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 completed tags must include the following:

- Applicable waste container or bin number
- Sample identification number (13 digits, as described later in this section)
- Sampler signature
- Ambient temperature and pressure (°C and mm Hg, respectively), when required
- Comment section
- Requested analyses
- 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.

- When a sampling manifold is used, the sampling manifold pressure must be recorded when required.

- In the analytical laboratory, canister pressure must be recorded within 24 hours of validated time of sample receipt (VTSR) and immediately prior to sample preparation or 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.

WIPP GAS SAMPLE CANISTER TAG

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

Bin Number: _____ Drum Number: _____

Sampling Organization: _____ Sample Description: _____

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

ANALYSIS REQUESTED	Inorganic Gases	NO_x	VOC's	Other

Blank Sample? _____Yes _____No

Remarks: _____

_____

Sampler Signature: _____

Notes: (1) C = Canister pressure gauge reading in psig, M = Manifold pressure gauge reading in mm Hg.
(2) P = pressure in mm Hg; T = Temperature in C

Figure 5-5. Gas Sample Canister Tag
EXAMPLE ONLY

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.

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:

ZZ	MMDDYY	AAXXX
sampling site ID	date	canister ID

where ZZ 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.

5.4.3 Innermost Layer of Confinement Identification Number

Each innermost layer of confinement, as it is removed from within a waste container, must be marked with an identification number according to the following scheme. The first innermost layer of confinement removed from within each waste container must be labeled "1," and the last removed labeled "n," where n corresponds to the total number of innermost layers of confinement in the waste container. The innermost layer of confinement identification number must be recorded on the QC Sample Record Form, Figure 5-6. This record will allow field sample identification numbers to be correlated with both the innermost layer of confinement identification numbers and the waste container number.

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. Field reference standards collected during bin sampling (Section 8.0) do not have to be 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:

FIELD QC SAMPLE RECORD FORM

Date: _____
Bin Number: _____
Sampler: _____

	DRUM/BIN NO.		DRUM/BIN NO.		DRUM/BIN NO.	
SAMPLE TYPE*	Field Sample Number	Time	Field Sample Number	Time	Field Sample Number	Time

Comments (note any discrepancies): _____

*Sample Type Designator

Sx - samples
Dx - duplicate samples
EBx - equipment
FBx - field blanks
FSx - field standards

ISn - innermost layer of confinement samples
IDn - innermost layer of confinement field duplicate
IEBn - innermost layer of confinement equipment blanks
IFBn - innermost layer of confinement field blanks
IFSn - innermost layer of confinement field standards

DSx - drum samples
DDx - drum duplicate
DEBx - drum equipment blanks
DFBx - drum field blanks
DFSx - drum field standards

Where x = O for organic; I for inorganic; and N for oxides of nitrogen
n = innermost layer of confinement number, 1,2,3,...

Figure 5-6. QC Sample Record Form
EXAMPLE ONLY

- Sample identification numbers of each sample collected from each waste container or bin, where waste containers or bins are identified by waste container or bin 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;
- Comment section; and
- Sample type designator.

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.

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 national certifying agencies (e.g., NIST) or manufacturers. All reference standards must have valid relationships to nationally recognized standards. If national standards do not 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, respectively, to ensure and provide documentation that items which fail 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 using procedures provided by manufacturers in equipment manuals.

Calibrated equipment must be uniquely identified. This can be accomplished by the use of the manufacturer's serial number, a calibration system identification number, or some 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 isolated 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 controls established and implemented in site QAPjPs and SOPs, respectively. These procedures must address performance criteria. When RTR equipment is in use, operational checks must be conducted at the beginning of each work shift. These checks shall also include observation of a test pattern to ensure that the RTR system has adequate video quality.

6.2 Radioassay (RA)

RA equipment must be calibrated and maintained in accordance with controls established and implemented in the site QAPjPs and SOPs, respectively. SOPs must cover the system calibration, routine performance checks, and operation of the system. For any types of RA systems which are covered by ANSI, ASTM or other consensus standards, the site SOPs should be consistent with all relevant provisions of these standards.

Complete verification of calibration of RA for at least one counting geometry/sample matrix combination must be repeated at least annually. Calibration standards shall be prepared from primary standards obtained from suppliers maintaining measurement systems traceable to NIST whenever such standards are available. When standards are not available from such suppliers, the actual standards used shall be calibrated against primary standards obtained from suppliers maintaining measurement

systems traceable to NIST. The documentation of this cross-calibration shall be retained as a quality assurance record. Calibration standards shall be prepared using isotopes, geometries, and matrices having characteristics as close as possible to those expected for actual samples without compromising the quantitative integrity or homogeneity of the standard. The commonly accepted techniques of transmission and live-time corrections to compensate for matrix variations present within a drum are acceptable for the SGS gamma-ray assay technique. Computer programs used to calculate activities of radioisotopes may use correction algorithms to compensate for some waste characteristics such as waste density, gamma absorption, neutron moderator and absorption indices for the PAN systems. These programs shall be documented, verified and validated as required in NQA-1, Element 11 and Supplement 11S-2, Supplementary Requirements for Computer Program Testing.

All RA equipment shall receive routine performance checks for such parameters as system counting efficiency and system background. Spectrometry based systems shall also receive routine performance checks for energy calibration and resolution. Routine performance checks shall be performed with check sources which are stable and constant or which change only by well-established and predictable quantities (e.g., radioisotope decay). Site SOPs for performance checks shall state the standards used, frequencies for each test, record keeping, control limits, and corrective actions to be taken when a control limit is exceeded. Control charts (e.g., based on acceptable ranges or variances) should be used to track trends in the parameters measured in performance checks. Performance checks shall be performed and documented at least twice each shift. These checks shall be performed prior to any actual waste measurements on each work shift and after completion of all waste measurements for that shift. When shift operations are contiguous or overlapping, the performance checks for the end of the shift completing work can be the same performance checks as those done at the beginning of the shift starting work.

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.

6.3.1 Certification Check of the Sampling Area Pressure Sensors

The sampling area pressure sensors(s), if present, must be certified prior to initial use, then annually, using NIST standards. If necessary, the pressure indicated by the pressure sensor(s) must be temperature compensated.

6.3.2 Calibration of Ambient Air Temperature Sensor

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

6.3.3 Calibration of the Organic Vapor Analyzer

The organic vapor analyzer (OVA) must be calibrated once per day, prior to first use, or as necessary according to the manufacturer's specifications. Calibration gases must be manufacturer-certified to contain known analytes at known concentrations. The balance of the OVA calibration gas must be consistent with the sampling manifold purge gas when the OVA is used.

6.4 Visual Examination

The weighing system used during the visual examination must be calibrated using NIST traceable 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 audio/video equipment. Checks of this equipment must include observation of test patterns, prior to each day's use, to ensure adequate video quality.

6.5 Determination of Headspace Gases

There are several methods for the analysis of headspace gas samples collected in SUMMA® passivated canisters for the constituents listed in Table 3-7. 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 listed in Table 3-7, 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 fail to meet the acceptance criteria of ±10% or closures fail to 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

Calibration procedures involve generating a multipoint initial calibration. This calibration curve shall be run initially, when indicated as a QC action, and when a change in the instrument may in any way affect the analytical results. The initial calibration is followed by daily verification with a calibration check standard.

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

Procedures for establishing initial and continuing calibration for gas chromatography are detailed in this section.

Initial Calibration. A multipoint calibration curve, consisting of a minimum of three standards, shall be generated for all analytes using gas standards. The low standard shall be at five times the MDL or at 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. Continuing calibration check standards must be analyzed at the beginning of each 12-hour shift. The laboratory control sample (LCS) may be used for the continuing calibration check. If a standard(s) other than the LCS is used for continuing calibration, then the LCS must be run as a sample during the analytical batch. The calibration check standard must be from a separate source than that used for the initial instrument calibration. Recalibration is required when a calibration verification fails or when a change in the instrument may affect the analytical results. The response factor (or concentration) of the continuing calibration check standard shall be compared to the corresponding average response factor (or concentration) from the most recent valid initial calibration. If the RPD between the average response factor and that of the continuing calibration check is less than 10%, the GC system will be considered calibrated.

6.5.3 Spectrophotometry

A multipoint calibration curve, consisting of a blank and a minimum of 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 0.67 moles of $NaNO_2$ produces an equivalent absorbance 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 five times the MDL 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 Determination of Headspace 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 3-6 which will be collected in SUMMA® passivated canisters. The differences between these methods are the manner in which the gas sample is introduced into 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* (USEPA, 1988a) for gas sample introduction into the GCMS. Calibration procedures used for the analysis of VOC canister samples by these methods are derived from EPA Compendium Method TO-14 (USEPA, 1988b) and the third edition of EPA SW-846, Method 8240/8260 (USEPA, 1986b). 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.

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	Initially and as needed	%RSD all compounds <35%
GCMS	Continuing calibration	Every 12 hours	%D for all compounds within 30% of initial calibration
GC	3-pt initial calibration	Initially and as needed	%RSD all compounds <30%
GC	Continuing calibration	Every 12 hours	%D for all compounds within 30% of initial calibration, RTs within 3 standard deviations of initial calibration

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 3-6. Commercially purchased primary gas standards must be certified by the manufacturer. 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 standards shall be prepared from dilution of the primary standards. 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 3-6. 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. Laboratory SOPs 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 analysis of 50 ng of bromofluorobenzene (BFB). 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 a minimum of five secondary analytical standards that define the linear range of the instrument for the analytes listed in Table 3-6. One of the standards must be at an amount (nanograms) equivalent to the PRQLs specified in Table 3-8.

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

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 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 initial calibration. If the percent difference (%D) between the average response factor and the continuing midpoint response factor is less than 30%, 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 30% 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. This curve shall be verified using a 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 3-6. Commercially purchased primary gas standards must be certified by the manufacturer. 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. 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 initial calibration curve.

Initial Calibration. A multipoint external standard calibration shall be performed. The multipoint calibration must consist of a minimum 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 multipoint 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.

Retention time windows shall be established for all analytes. Retention time windows are determined by injecting a minimum of three standards over a period of 72 hours. Retention time windows shall be calculated as the mean plus or minus three times the standard deviation of the individual retention times for each calibration standard analyzed in the 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight. Retention time windows must be determined for all analytes on each GC column prior to the analysis of any samples and whenever a new GC column is installed.

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 %D 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. 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.

6.7 Flammability Testing

Calibration requirements and frequencies are summarized in Table 6-3. Guidance Manual Procedure 810.1 describes the Program-required procedure for evaluating the flammability of bin headspace samples that contain a summed concentration of flammable VOCs exceeding 500 ppmv. DOE generator/storage sites' analytical laboratories or their contract laboratories shall maintain the most current revision of the Guidance Manual as the source for Procedure 810.1. Site QAPjPs and SOPs shall adopt all calibration and maintenance procedures described in Guidance Manual Procedure 810.1.

In addition to calibration procedures, operational checks shall be performed at the beginning of each working day prior to the testing of standards or samples. Operational checks shall include:

- Examination of the structural integrity of the flame test vessel (no cracks or deformations)
- Examination of the cleanliness of the flame test vessel (no residual material or soot from previous testing)
- Inspection of the ignition source power output (clearly visible spark produced between the electrodes)
- Verification of the pressure stability of the test vessel and all connecting lines (apparatus capable of maintaining a vacuum of 30 ±1.5 mm Hg for five minutes).

6.7.1 Standards Preparation

Standards for flammability testing shall use pure compounds and shall be prepared according to the procedures given in Guidance Manual Procedure 810.1.

6.7.2 Calibration Procedures

Initial calibration of the flame test apparatus shall be performed by testing the flammability of 8.0% (v/v) methanol, 15.0% (v/v) methanol, and 1.0% (v/v) diethyl ether. Calibration of the flame test apparatus shall be maintained on a daily basis with 8.0% (v/v) methanol. Calibration is achieved when, under the test conditions specified in Guidance Manual Procedure 810.1, the 8.0% (v/v) methanol and 15% (v/v) methanol standards are evaluated as flammable, and the 1.0% (v/v) diethyl ether standard is evaluated as nonflammable. The calibration frequency and performance criteria are summarized in Table 6-3.

Table 6-3. Flammability Testing Calibration Requirements

Technique	Procedure	Frequency of Procedure	Acceptance Criteria
Flame Test	3-pt initial calibration	Initially and after every 20 samples	8.0% Methanol - Flammable 15% Methanol - Flammable 1.0% Diethyl ether - Not Flammable
Flame Test	Continuing calibration	Beginning of each working day	8.0% Methanol - Flammable

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 and site-specific Item Description Code (IDC), as applicable
- Waste items, residual materials, packaging materials, and/or waste material categories inventory, as applicable
- Quantity of free liquids
- RCRA Waste Categories, per Waste Analysis Plan (DOE, 1992a).

RTR is a nondestructive 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 such as wave motion of free liquids. An RTR system normally consists 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 and data acquisition 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.

The waste container is scanned while the operator views the television screen. An audio/videotape is made of the waste container scan and is maintained as a permanent record. An RTR data form is also used to document identifiable waste items, residual and packaging materials and their quantities.

7.2 Radioassay (RA)

Three types of RA 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. Software 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, ASTM C853 (ASTM, 1982), ANSI 15.20 (ANSI, 1975), and Nuclear Regulatory Commission (NRC) standard practices and guidelines (Regulatory Guide 5.11, USNRC, 1984) 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 assays shall be performed until the remedial action is satisfactorily completed.

The assay procedure for PAN units specifies the use of a computerized data acquisition system. An operator must insert a waste container into the PAN unit and enter all waste container 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.

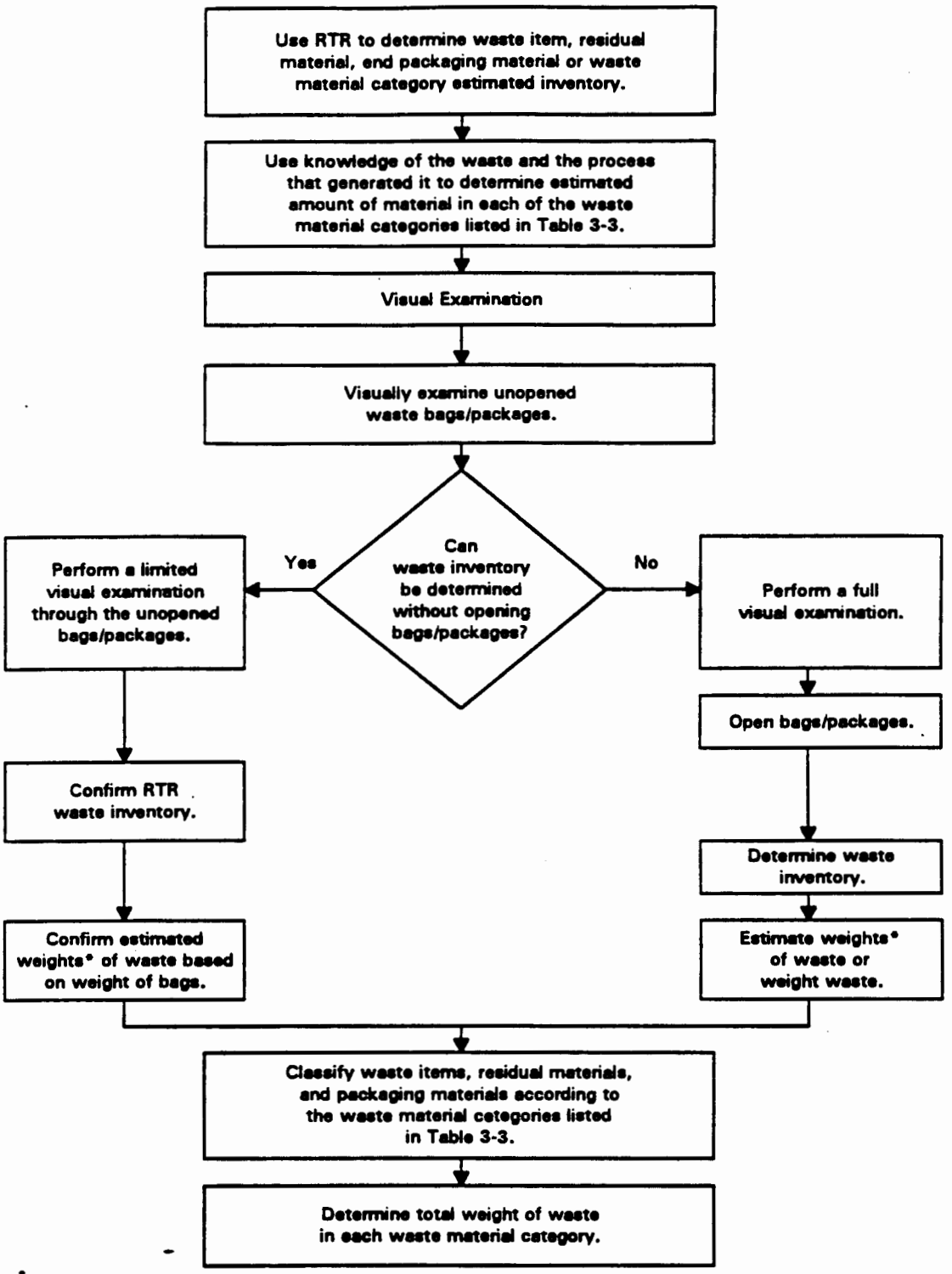
7.2.3 Passive Neutron Coincidence Counting (PNCC) Procedure

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, residual materials, and packaging materials in the waste container 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, residual materials, and packaging materials so that they can be classified according to the waste material categories listed in Table 3-3. Individual bags (or packages) and, if necessary, the contents of each bag within the waste container must be weighed, and the weights must be recorded. In cases where bags are not opened, 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.). Estimated or measured weights must also include process knowledge estimates of sorbed liquids, lead in leaded rubber, etc. for composite materials. 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.



*Include process knowledge estimates of eorbed liquids, lead in leaded rubber, etc. for composite materials.

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

Figure 7-1 illustrates a two-track approach to the visual examination of waste. Subsequent to RTR and waste container opening, 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 the weighing of bags is appropriate. If the waste is heterogeneous, the expert may decide a full visual examination involving determination of a waste item, residual material, and packaging material inventory by opening bags, segregating waste, and weighing is warranted. Various degrees of segregation and weighing are possible based on the expert's judgment. The sites QAPjP must specify the qualifications and decision-making criteria for the visual examination expert. 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 Headspace Gases

Analytical methods for the analysis of 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 (optional) 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. For a positive pressure check, a canister passes if the pressure does not change by more than ±2 psig in 24 hours. Any canister that fails must be checked for leaks and reprocessed. One canister per batch shall be filled with humid zero air or humid high purity 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 Gas 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 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. These methods shall be specified and described in each site's QAPjP and SOPs.

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 mass spectrometer must have a minimum abundance sensitivity of 1 ppm, to allow measurement of low levels of CO in N_2 at m/e 28. 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, nitrogen, oxygen, 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 and retention time windows 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 Determination of Organic Headspace Gases

Analytical methods for the analysis of VOCs collected using SUMMA® passivated canisters (see Section 7.4.1 for canister cleaning requirements) 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, 1988a). Detailed guidance procedures for the analysis of volatile organic compounds listed in Table 3-6 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 3-6.

7.5.1 VOC Analytical Methods

Analytical procedures must use GCMS for the analysis of VOCs listed in Table 3-8 (m-Xylene and p-Xylene cannot be separated by GCMS procedures). 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 Program (Section 10.2), and all other requirements of this QAPP are satisfied. Samples and calibration standards must be analyzed at the same temperature (±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 3-6 and identification of compounds not listed in Table 3-6. 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 3-6, 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 dissimilar 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.

7.6 Flammability Testing

The evaluation of the flammability of bin headspace samples is performed using a modified version of ASTM Method E 681-85. The modified method, which is given in the Guidance Manual as Procedure 810.1, shall be used by DOE generator/storage sites' analytical laboratories or their contract laboratories to perform flammability evaluations. The details of implementing Guidance Manual Procedure 810.1 shall be specified in site QAPjPs and laboratory SOPs.

Samples for flammability testing shall be sent to the laboratory in SUMMA® canisters. The sample introduction portion of the flame test apparatus shall provide a leak-tight seal of the SUMMA® canister to the flame test apparatus. The flame test vessel and all connecting lines shall be capable of being evacuated to less than 30 Torr. Samples shall be transferred to the 5 liter flame test vessel from SUMMA® canisters at ambient temperature.

As samples are expanded into the flame test vessel, the temperature and pressure within the flame test vessel shall be monitored and the final temperature and pressure recorded. Prior to ignition, the final temperature within the test vessel shall be at ambient and the final pressure not less than 400 Torr. At the minimum operating pressure of 400 Torr, a 2.63 L sample must be delivered to the flame test vessel.

Temperature measurements of the ambient air immediately surrounding the flame test vessel shall be accurate to ±4°C. Temperature measurements within the flame test vessel shall be accurate to ±1°C. Pressure measurements within the flame test vessel shall be accurate to ±0.2%. The calibration requirements for temperature and pressure sensors shall be specified in site SOPs.

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 here. To prevent the introduction of errors or the loss or misinterpretation of the data, adequate precautions must be taken. The general requirements discussed here should be considered minimum and will be expanded on throughout the subsections dealing with individual analytical or test techniques. Site QAPjPs must delineate the implementation of the requirements contained in this section. 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, legibly, 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, is defined as a suite of samples of a similar matrix that is processed as a unit within a specific time period. An analytical batch must not exceed 20 samples, all of which must be received by the laboratory within 14 days of the validated time of sample receipt (VTSR) of the first sample of the batch.

Data Reduction - Data Generation Level

Data reduction must be performed at the site of data generation such that all the requirements outlined in relevant 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 relevant subsections.
- The reporting units must be consistent with the appropriate method used and the requirements described in relevant subsections.

- All analytical data must be reported to the significant figures specified in relevant subsections and appropriate to the technique used.
- All data must receive independent technical review to ensure that the reduction is appropriate.

Data Reduction - Project Level

Prior to any data reduction at the site-project level, it should be verified that all data have been validated at the data generation level. The validated data must then be reduced further, when appropriate, to meet the requirements outlined in Section 8.7. Minimum requirements for data reduction shall include the following:

- The technique of data reduction shall be consistent with the equations/procedures outlined in Section 12.6.
- Appropriate unit conversions shall be made prior to comparison of the data to regulatory limits.
- All data must receive independent review to ensure that the reduction is appropriate and correct.

Data Validation

The data shall be validated at three levels. The first level of review is conducted on each analytical batch in the laboratory or on each waste container for the other characterization tests. Next, the site Project Manager shall review data associated with each bin (bin case). Finally, DOE/WPSO 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 are correct and complete.

2. One hundred percent of the data must receive technical supervisory signature release on a per analytical batch or waste container 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-waste container data) is complete and includes raw data, data forms, calculation records, QC summaries, narrative, COCs, and sample tags as appropriate (see text on page 8-4 for requirement regarding sample tag removal).
   - 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. QC criteria that were not met must be documented in a non-conformance report.
   - 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. This verification should occur prior to project level data reduction.
   - 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.

- Project level data reduction has occurred, when appropriate, and is consistent with the requirements outlined in Sections 8.7 and 12.0.
- Project level reduced data are evaluated for compliance with regulatory requirements (Section 8.7).
- Regulatory compliance data have been entered on the appropriate reporting forms.

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 minimum 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), as described in Section 12.6.
   - 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 report. If the Project Manager requests that sample canisters be retained for future use (e.g., experimental holding time study), new sample identification and chain-of-custody procedures should be initiated with a cross reference to the original sample.

Data Validation - DOE/WPSO Level

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

1. One hundred percent check of the bin case and bin case addendum reports for inventory.

   - Regulatory compliance reporting forms are present
   - Waste characterization summary forms are present
   - Result summary for field and laboratory QC samples
   - RTR/RA data report
   - Visual examination data report
   - Laboratory data reports are present
   - Project level signature releases

2. One hundred percent verification that documentation is provided that demonstrates project level validation has occurred.

   - Field QC samples were evaluated for compliance
   - Sample holding times were evaluated for compliance
   - Verify that regulatory compliance forms are complete and correct

DOE/WPSO must validate a bin case report and notify the site of the acceptance status of the data within two weeks of report receipt. DOE/WPSO must validate a bin case report addendum and notify the site of the acceptance status of the data within one week of report receipt.

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. In addition to the data forms, calculation records, QC summaries, COCs, and sample tags the analytical laboratory report must include:

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. The DOE-WIPP cover page form, Cover Page (Figure 8-5), is an example.

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

RTR, RA, and Visual data must appear on forms which meet the requirements identified in subsequent subsections. The original data forms must be forwarded to the site Project Manager as soon as possible following data generation. A copy of the 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, waste container, and regulatory compliance data into one comprehensive report. This report

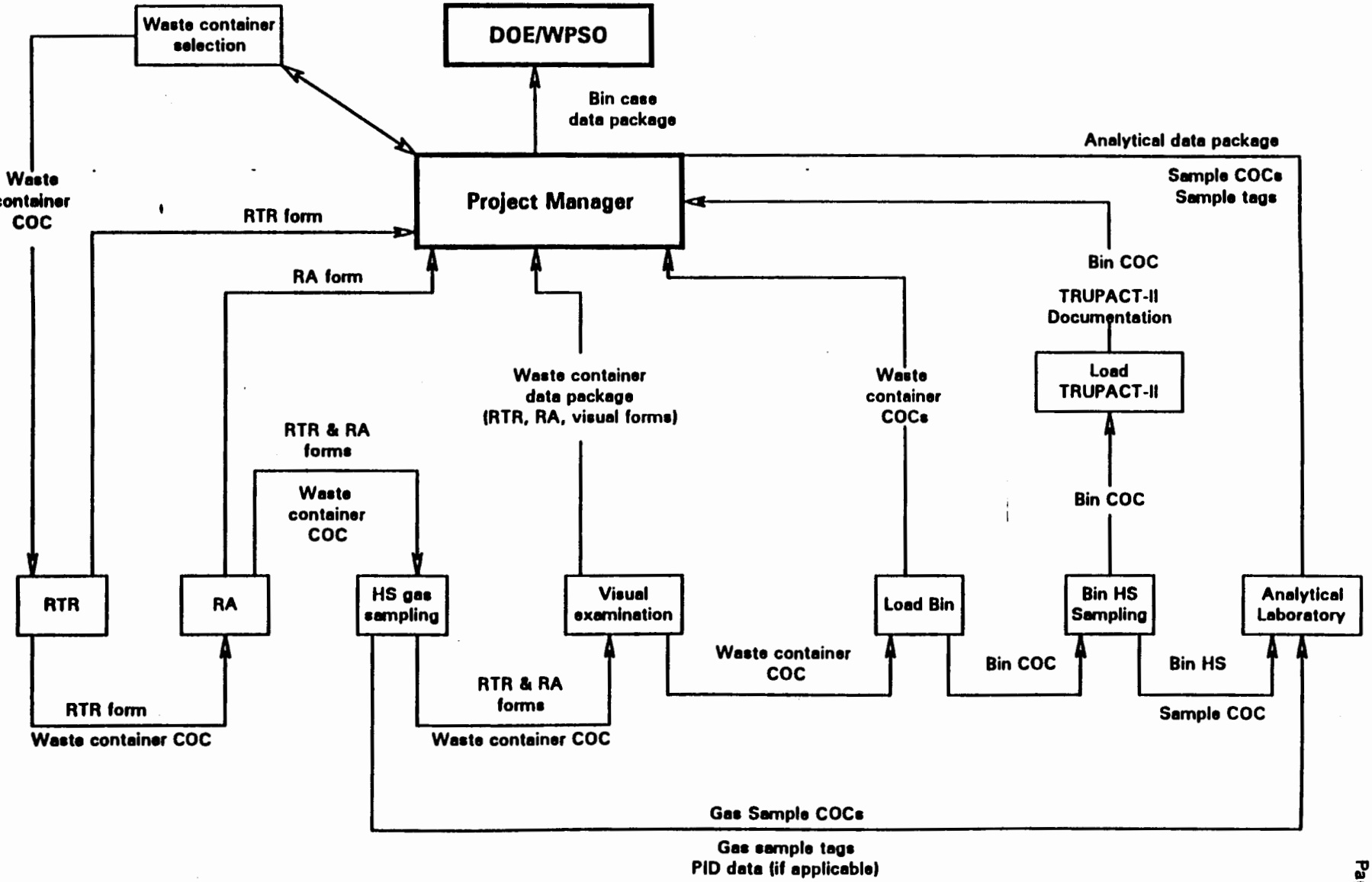


Figure 8-1. Overall Data Reporting Flow Chart

shall entail summarizing the laboratory analytical data and 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/WPSO within two weeks of receipt of all the necessary bin case data. The final bin case report submitted to DOE/WPSO 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 significant deviations from the methods used and occurrences during the sampling process that may impact data quality. Regulatory compliance shall also be discussed.
- Regulatory compliance reporting forms
- A result summary for each waste characterization technique
- A result summary for all field and laboratory QC samples
- RTR/RA data report
- Visual exam data report
- All laboratory data reports.

Each bin case report shall have an addendum report associated with it. This bin case addendum report shall contain all the pertinent information regarding the bin headspace sample collected (Section 4.3) to verify compliance with no-migration (Section 8.7.1.2) and flammability (Section 8.7.1.3). Comparability compliance data shall be submitted as part of the bin case report, not the addendum. Addendum reports shall contain the same type of information that is required for the bin case report (e.g. narrative, result summaries and appropriate reporting forms) pertinent to the compliance bin sample and its analysis and reporting. The bin case report addendum shall contain a cross-reference to the bin case report. A flammability supplement to the bin case report addendum may be required to report a final emplacement time window (Section 1.3.2.4 and Section 8.7.1.3).

A WIPP Waste Profile Form, as described in the WIPP Waste Analysis Plan (USDOE, 1992), shall be sent to DOE/WPSO prior to waste shipment. WIPP Waste Profile Forms must be completed for each waste container and are available, with instructions, from DOE/WPSO.

The site Project Manager must maintain a record of transmittal regarding the submission of the bin case report and the bin case report addendum to the DOE/WPSO. The process by which data are compiled, reviewed, and then shipped to the DOE/WPSO 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, residual materials, and packaging materials documented on an RTR data form must be used in conjunction with site-specific data on standard weights to classify the waste items, residual materials, and packaging materials into the waste material categories listed in Table 3-3 and to estimate the total weight of waste material in each of 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. Involved DOE sites should coordinate these efforts to assure maximum consistency.

8.1.2 RTR Data Reporting

If applicable, the data reporting requirements for RTR are:

- Examining site
- Waste container identification number
- Content Code or IDC, as applicable
- Any changes made to IDC
- Date of RTR examination
- Operator signature/date
- Reviewer signature/date
- Presence/absence of waste container liner (yes/no)
- Estimated inventory of waste container contents, e.g., number/count of a particular waste item, or waste material categories, as applicable
- 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 completed 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.

RTR DATA FORM

Page 1 of 4

Examining Site: _____

Waste Container Identification Number: _____

TRUCON Content Code: _____

IDC: _____

Date of Examination: _____

Audio/videotape ID Number _____

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 8-2a. 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: _____

Audio/videotape ID Number _____

7. Estimated inventory of Waste Container Contents

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

Figure 8-2b. 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: _____

Audio/videotape ID Number _____

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 8-2c. 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	_____	_____
Audio/videotape ID Number:	_____			

WASTE MATERIAL CATEGORIES	Estimated Weight (kg)	Error (kg)
CELLULOSICS	_____	_____
Other organics	_____	_____
Plastics	_____	_____
Rubber	_____	_____
Corroding metal - steel	_____	_____
Corroding metal - aluminum	_____	_____
Noncorroding metal	_____	_____
Solid inorganic wastes	_____	_____
Inorganic sludges	_____	_____
Cements	_____	_____
Totals	_____	_____

Comments (note any descrepancies):

_____

_____

_____

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

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
- 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)

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

- 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)
- 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 3-3 (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 completed 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 and procedures for data reduction and reporting must be detailed in site QAPjPs and/or implementing SOPs to include sample calculations and forms.

8.4.1 Data Reduction

All analyte gas 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.

Quantitative values shall be reported in units of volume percent (vol. %) down to the MDL. Values for analytes analyzed for but not detected shall be reported as the MDL with the required "U" flag (i.e., if the MDL is 0.001 vol. %, report 0.001 U). Analytical results, including MDL values used in reporting, must be corrected for dilution.

The numerical value assigned to the MDL for a given analyte shall include two significant figures when the MDL is greater than or equal to 10% of the corresponding PRDL. If the MDL is less than 10% of the PRDL, its numerical value shall be reported with one significant figure. (Note: Under no

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 and location 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 8-4a. 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 8-4b. 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 8-4c. 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 MATERIAL CATEGORIES	Estimated Weight (kg)	Error (kg)
CELLULOSICS	_____	_____
Other organics	_____	_____
Plastics	_____	_____
Rubber	_____	_____
Corroding metal - steel	_____	_____
Corroding metal - aluminum	_____	_____
Noncorroding metal	_____	_____
Solid inorganic wastes	_____	_____
Inorganic sludges	_____	_____
Cements/Estimated water content (wt%)	_____	_____
Totals	_____	_____

Comments (note any discrepancies):

_____

_____

_____

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

circumstances is a value reported as all zeros acceptable.) Analytical results above the MDL shall be reported to a sufficient number of decimal places to include the least significant figure of the MDL, but to no more than three significant figures.

To illustrate implementation of these reporting requirements, consider the example of an analyte having a PRDL of 0.1 vol. %. If the MDL for this analyte is 0.024 vol. %, then a result of 0.09265 would be reported as 0.093. If the MDL is 0.0008 vol. %, then 0.09265 would be reported as 0.0926.

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.

8.4.2 Data Validation

All gas analysis data must be reviewed and approved prior to being reported. This validation process must include the three levels of review which are 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. The site QAPjPs must include the forms that will be used for reporting. The text identifies the information that must appear on individual data forms. Reporting data forms must include the following:

1. 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. Gas analysis data qualifying flags shall be used. These are as follows:

      Flag qualifier:
      B - Analyte detected in the blank.
      E - Analyte exceeds the calibration curve.
      J - Analyte less than the PRDL and greater than or equal to the MDL.
      U - Analyte was undetected.

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

In addition, laboratory name, analytical batch number, field sample number, laboratory sample ID, date received, and date and time 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 3-7. 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, and date and time the blank 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 and time 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 and time 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.

2. 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, to include sample calculations and forms.

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

DOE-WIPP

COVER PAGE

Lab Name: _____ Project Identification: _____

Lab Code: _____ Analytical Batch No.: _____

Field Sample No.	Lab Sample ID	Laboratory QC Sample ID	Analytical Method

Comments:

_____

_____

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

Signature/Title: _____ Signature/Title: _____

Date: _____ Date: _____

Figure 8-5. Cover Page

EXAMPLE ONLY

DOE-WIPP
FORM 1
GAS ANALYSIS DATA SHEET

FIELD SAMPLE NO.

Lab Name: _____

Lab Code: _____

Analytical Batch No.: _____

Lab Sample ID: _____

Date Received: _____

Analysis Date: _____

Analysis Time: _____

Concentration Units (Vol. %)

Analyte	Concentration	Flag	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: _____

Analysis Time: _____

Analyte	M	ICV			CCV1			CCV2			CCV3		
		True	Found	%R	True	Found	%R	True	Found	%R	True	Found	%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: _____

Analysis Time: _____

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

FIELD SAMPLE NO.

Lab Name: _____

Lab Code: _____

Analytical Batch No.: _____

Analysis Date: _____

Analysis Time: _____

Concentration Units (Vol. %)

Analyte	Sample	Duplicate	RPD
Argon			
Carbon Dioxide			
Carbon Monoxide			
Hydrogen			
Nitrogen			
Nitrogen 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: _____

Analysis Time: _____

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

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. Reporting form flags:
   B - If the analyte was detected in the associated laboratory blank.
   E - If the analyte concentration exceeded the initial calibration curve.
   J - If the analyte was detected at levels less than the PRQL but greater than the MDL.
   U - If the analyte was not detected.

2. 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 listed in Table 3-8 analyzed for by GCMS (p-xylene and m-xylene may be reported as p/m-xylene). For analytes not detected, the analyte MDL corrected for sample volume must be reported. EPA CLP data qualifying flags shall be used. In addition, laboratory name, field sample number, laboratory sample number, dilution factor, date received and date and time 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 and analyzed by GCMS 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 and analyzed by GCMS. 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			
p/m-Xylene			
o-Xylene			

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

i. Copies of data system quantitation reports (including all mass spectra and chromatograms) for all calibration standards, samples and blanks.

j. Copies of mass spectra for all reported TICs.

3. 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. EPA CLP data qualifying flags may be used. In addition, laboratory name, field sample number, laboratory sample number, dilution factor, date received, and date and time 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 listing the response factors for the alcohols and ketones listed in Table 3-8 at all calibration points, the average response factor or for each analyte, the average retention time, the retention time window, and the percent relative standard deviation for each analyte across the 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 average response factor, and absolute retention time 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.

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.

8.6 Flammability Testing

Flammability testing results shall be documented and available to data users on standardized forms approved by DOE/WPSO.

8.6.1 Data Reduction

The qualitative results from flammability testing require no data reduction. Flammability testing results shall be reported with the information required in Section 8.6.3.

8.6.2 Data Validation

The general data validation and review requirements of Section 8.0 are sufficient to ensure that the criteria in Table 6-3 have been satisfied for all flammability testing results.

8.6.3 Data Reporting

The results of flammability testing shall be reported on a standardized form approved by DOE/WPSO. An example form is detailed in Figure 8-8. At a minimum, the form shall contain the following information:

- Laboratory name
- Bin identification number
- Bin field sample identification number
- Flammability test date and time
- Initial calibration data
- Continuing calibration data
- Field sample test data.

Flammability test results shall be included in bin case data packages/reports when the summed concentration of flammable VOCs in a bin headspace sample exceeds 500 ppmv at the 90% confidence level.

8.7 Regulatory Compliance Reduction and Reporting Requirements

Prior to shipment of experimental waste to the WIPP for bin-scale and alcove testing, it must be demonstrated that all applicable regulatory requirements addressed under this Program have been met. Report documentation and all supporting data must be submitted to and approved by DOE/WPSO before experimental waste may be shipped to the WIPP facility. Report documentation shall include the bin case report and the associated addendum report.

Bin ID Number:_____ Flammable: ____ Yes ____ No

Laboratory Name: _____

Laboratory Code:_____

Initial Calibration Information:[a]

Standard	Date	Time	Flammability Evaluation[b]
8.0% Methanol			
15.0% Methanol			
1.0% Diethyl ether			

Continuing Calibration Data:[c]

Standard	Date	Time	Flammability Evaluation
8.0% Methanol			

Sample Data:

Field Sample ID No.	Date	Time	Flammability Evaluation

[a] Valid initial calibration data
[b] Yes/No
[c] If applicable

Figure 8-8. Flammability Testing Reporting Form
EXAMPLE ONLY

Experimental waste characterization data must be validated at the site project level and then subjected to another data reduction step. Section 12.6 details the reduction calculations that must be applied to regulatory compliance data prior to reporting. These calculations adjust the measured data to the upper confidence limit (X_{UL}) to enable comparison with the regulatory limits. In addition, data must be expressed in appropriate units before it is compared to regulatory limits in order to demonstrate compliance.

Procedures for regulatory compliance data reporting are identified in this section. Specific procedures for the implementation of reporting requirements must be detailed in site-specific QAPjPs and SOPs, as appropriate.

8.7.1 Reporting Requirements for the WIPP No-Migration Determination (NMD)

Procedures for the evaluation of NMD compliance data and minimum reporting requirements are identified in this subsection. Regulatory compliance data must be reported on DOE/WPSO approved forms. Example forms are provided throughout subsequent subsections.

8.7.1.1 Comparability

In order to comply with the maximum concentration comparability requirement of the NMD, as outlined in Section 1.3, the upper 90% confidence limit values (X_{UL}) of the measured headspace concentrations from the drum, 55-gallon poly bag, and innermost layers of confinement of each waste container must be compared to the maximum allowable VOC concentrations in EPA's NMD. If the regulatory requirement is satisfied, the waste container may be included in the bin-scale test.

Table 1-3 lists the maximum allowable concentrations (herein referred to as the regulatory limits) of the five major VOC hazardous constituents of concern.

Data obtained (X_{UL}) for headspace samples must be compared to the regulatory limits by the method described below:

- For each waste container, identify the highest X_{UL} data point for each constituent. (Consider the drum, 55-gallon poly bag, and the innermost layer headspace data.)
- Compare this highest X_{UL} constituent concentration to the regulatory limits listed in Table 1-3.
- If the highest X_{UL} constituent concentration is below the regulatory limit, then the waste container passes for that constituent.
- Repeat the comparison for all constituents.
- If all the highest X_{UL} constituent concentrations are below the regulatory limits, then the waste container has met the regulatory requirement and may be included in the bin-scale test.

Comparability compliance data shall be reported for each waste container to be included in the bin-scale test and must include the following minimum information:

- Bin identification number
- Waste container identification number
- Waste Type (as identified in USDOE, 1989)
- All the layers of confinement (drum, 55-gallon poly bag, and innermost layers of confinement) headspace data for the five hazardous constituents, X_{UL}
- The appropriate regulatory limits for each constituent (i.e., as identified by waste type in Table 1-3)
- Identification of the highest X_{UL} data point for each constituent (for all the headspace samples pertaining to the waste container)
- Decision indicator regarding the comparison of headspace data to the regulatory limits.

Comparability data shall be submitted as part of the bin case report. Figure 8-9 is provided as an example of a comparability reporting form.

8.7.1.2 No-Migration Demonstration

In order to comply with the demonstration of no-migration during the bin-scale tests, the bin headspace concentrations of three VOC hazardous constituents must not exceed the NMD mean values (regulatory limits). These hazardous constituents and the associated regulatory limits are listed in Table 1-4.

The upper 90% confidence limit values (X_{UL}) of the measured bin headspace concentrations of these constituents must not exceed the regulatory limits for a bin to be shipped to the WIPP. Compliance with this regulatory requirement must be verified and documented on a reporting form for inclusion in the bin case report. DOE/WPSO must verify that the no-migration demonstration requirements have been met before the bin can be shipped. The no-migration demonstration reporting form must consist of the following minimum information:

- Bin identification number
- Bin headspace sample identification number
- Waste Type
- X_{UL} sample data for the three hazardous constituents
- Constituent regulatory limits as identified in Table 1-4, as appropriate to the waste type
- Decision indicator regarding whether the bin headspace data (X_{UL}) are less than the regulatory limits.

Figure 8-10 depicts an example No-Migration Demonstration Compliance Reporting Form. No-migration demonstration compliance data shall be sumbitted to DOE/WPSO as part of an addendum report with the appropriate bin case report cross-referenced. (See Data Reporting-Project level, Section 8.0.)

Bin ID Number: _____

Comparability Criteria Met: ____ Yes ____ No

Waste Type[a]: _____

Waste Container ID Number: _____

Constituent	Carbon Tetrachloride	Methylene Chloride	1,1,1-Trichloroethane	Trichloro-ethylene	1,1,2-Trichloro-1,2,2-trifluoroethane
Field Sample ID#[b]	Sample Data[c] (X_{UL})				
Regulatory Limit[d]					
Highest Constituent Concentration (X_{UL})					
Decision[e]					

[a]Waste Types are identified in USDOE, 1989.

[b]To include the drum and 55-gallon poly bag as the first entries on form and all innermost layer of confinement samples.

[c]Sample constituent concentrations (volume %) at the upper confidence limit (X_{UL}).

[d]Maximum allowable VOC headspace concentrations (volume %) as appropriate for the waste type (Table 1-3).

[e]Pass if constituent concentration (X_{UL}) is less than the regulatory limit.

Fail if constituent concentration (X_{UL}) is greater than the regulatory limit.

Figure 8-9. Comparability Compliance Reporting Form

EXAMPLE ONLY

Bin ID Number: _____ NMD Criteria Met: ____ Yes ____ No

Bin Sample ID Number: _____

Waste Type[a]: _____

Constituent	Regulatory Limit[b]	Bin Sample Data[c] (X_{UL})	Decision[d]
Carbon Tetrachloride			
Methylene Chloride			
Trichloroethylene			

[a]Waste types are identified in USDOE, 1989.

[b]Allowable mean volatile organic headspace concentrations (volume %) as appropriate for the waste type (Table 1-4).

[c]Bin sample constituent concentrations (X_{UL}) in volume %.

[d]Pass if constituent concentration (X_{UL}) is less than the regulatory limit.
Fail if constituent concentration (X_{UL}) is greater than the regulatory limit.

Figure 8-10. No-Migration Demonstration Compliance Reporting Form
EXAMPLE ONLY

8.7.1.3 Flammability

DOE/WPSO must verify that all flammability requirements have been met before the bin can be shipped to the WIPP facility. VOC concentration data for bin flammability are sent to DOE/WPSO in the bin case addendum report. Hydrogen and methane concentration data are sent either in the bin case addendum report or, in the case of a final emplacement time window determination (Section 1.3.2.4), as a supplement to the bin case addendum report. All bin flammability compliance must be documented on DOE/WPSO-approved reporting forms. Example Flammability Compliance Reporting Forms are provided in Figure 8-11 and 8-12.

The data reporting requirements for VOC flammability are:

- Bin identification number
- Bin sample identification number
- Waste Type
- Date bin sampled
- Date bin loaded
- Upper confidence limit (X_{UL}) concentration of flammable VOCs
- Indication of whether the upper confidence limit of the summed flammable VOC concentrations is greater than 500 ppmv
- Indication of whether flame test was positive or negative, if applicable.

The data reporting requirements for hydrogen and methane flammability are:

- Bin identification number
- Bin sample identification number
- Waste Type
- Date bin sampled
- Date bin loaded
- Indication of whether this is a supplement to the addendum report
- Cross-reference to addendum report, if applicable
- Upper confidence limit (X_{UL}) concentration of hydrogen and methane
- Result of Le Chatelier for hydrogen and methane; fraction of LEL or result of calculation for hydrogen , methane and flammable VOCs, if applicable
- Indication of whether hydrogen/methane or hydrogen/methane/flammable VOCs are present at concentrations less than 50% of the mixture LEL
- Number of days remaining until 50% of the LEL is exceeded, specifying method of determination, i.e., sampled or not sampled
- Date by which the bin may be emplaced at the WIPP without sampling and/or purging
- Date by which the bin must be emplaced or resampled for hydrogen and methane, i.e., emplacement date minus four weeks.

Bin ID Number: _____ Date Bin Sampled: _____

Bin Sample ID Number: _____ Date Bin Loaded: _____

Waste Type[a]: _____ Flammability Criteria Met: ____ Yes ____ No

Flammable VOCs	Volume %[b]
Acetone	
Benzene	
n-Butanol	
2-Butanone	
Chlorobenzene	
Cyclohexane	
1,1-Dichloroethane	
1,2-Dichloroethane	
1,1-Dichloroethene	
cis-1,2-Dichloroethene	
Ethyl benzene	
Diethyl ether	
Methanol	
4-Methyl-2-pentanone	
Toluene	
1,2,4-Trimethylbenzene	
1,3,5-Trimethylbenzene	
o-Xylene	
p/m-Xylene	
Total	
Total (X_{UL})	

Regulatory Criteria	Decision[c]
≥500 ppmv Flammable VOCs	
Flame test	

[a] Waste types are identified in USDOE, 1989.
[b] A value of one-half the MDL must be used when a flammable VOC is not detected.
[c] Yes/No - exceeds 500 ppm or is positive flame test.

Figure 8-11. Volatile Organic Compounds Flammability Compliance Reporting Form EXAMPLE ONLY

Bin ID Number: _____ Date Bin Sampled: _____

Bin Sample ID Number: _____ Date Bin Loaded: _____

Waste Type:[a] _____ Flammability Criteria Met: ____ Yes ____ No

Supplement to Addendum Report: ____ Yes ____ No

Addendum Report Cross-Reference _____

Gases	X_{UL} (Vol. %)
H_2	
CH_4	

Le Chatelier Calculation	Result
H_2/CH_4	
H_2/CH_4/Flammable VOCs	

50% of the LEL Evaluation for H_2/CH_4	Emplacement Time (Days)[b]	Emplacement Date[c]	Emplacement Time Window[d]
Sampled[e]			
Not sampled[f]			

[a] Waste types are identified in USDOE, 1989.

[b] The time from when the waste is loaded on a bin and the bin is closed and placed in a Standard Waste Box through emplacement in the WIPP underground without exceeding 50%.

[c] Date by which the bin may be emplaced at the WIPP without sampling and/or purging.

[d] The emplacement date minus four weeks.

[e] Value from Tables 12-3 through 12-7, Section 12.6.

[f] Value from Table 12-2, Section 12.6.

Figure 8-12. Hydrogen and Methane Flammability Compliance Reporting Form
EXAMPLE ONLY

The method for determining emplacement time is:

1. For sampled bins,

   - Obtain the bin headspace hydrogen and methane concentrations.
   - Identify the TRUPACT-II shipping category.
   - Based on these values, the number of days remaining until 50% of the LEL is exceeded is determined from Tables 12-3 through 12-7. The times reported in Tables 12-3 through 12-7 include an allowance for a three-day shipping period.

2. For bins not sampled,

   - Identify the TRUPACT-II shipping category.
   - Determine the number of days using Table 12-2 and the time since the bin was loaded.

Flammability compliance data shall be submitted to DOE/WPSO as part of the addendum report, with the appropriate bin case report cross-referenced. (See Data Reporting - Project Level, Section 8.0.)

In addition, a DOE/WPSO approved Flammability Quality Assurance Objectives Reporting Form (Figure 8-13) must be submitted as part of the addendum report. This form will verify that the ±10% precision and accuracy requirements for flammable VOCs have been met. Flammability QAO determination procedures are described in Section 12.6.2.5. QAOs must be determined initially and then updated according to the frequency specified in Section 12.6.2.5. The data reported on the form must reflect the updates when appropriate. All data supporting these flammable VOC QAO determinations must be documented and available for auditing purposes.

VOC Flammability Quality Assurance Objectives [a]

Total Measured VOCs	Value (ppmv)
T_1	
T_2	
T_3	
T_4	
T_5	
.	
.	
.	
.	
.	
T_{30}	
Average of Total VOCs ($\bar{T}$)[b]	
Total of the known VOCs (KT)[b]	
Standard Deviation (S_T)	
Bias[c] ($\bar{T}$/KT)	
Precision[d] ($S_T/\bar{T}$)	

[a] As described in QAPP Section 12.6.2.5 and updated semiannually.

[b] Expressed in ppmv.

[c] $\leq 0.90 \leq$ Bias ≤ 1.10.

[d] Precision ≤ 0.10.

Figure 8-13. VOC Flammability Quality Assurance Objectives Reporting Form
EXAMPLE ONLY

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 internal QC tests including routine measurements of performance standards, replicates, and blanks. These internal 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 allowed 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 performed 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 results of this verification must be available to the RTR operator. 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. The system shall be operated in statistical control as determined by the control limits established by site SOPs.

As discussed in Section 6, routine performance checks shall be performed on all RA systems according to approved SOPs. Performance checks are required 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 in accordance with the site QAPjP and SOPs. 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 allowed to operate an RA system.

9.1.3 Drum Headspace Sampling

In the following text, "drum" is used to represent all layers of confinement that must be sampled within a drum (i.e., directly under the drum lid, the 55-gallon poly bag, and the innermost layers of confinement). If sites use the same equipment (e.g., manifold with the poly bag sampling head) to collect headspace samples from within the 55-gallon poly bags and the innermost layers of confinement of drums designated for one bin, then those samples constitute a sample batch and the QC sample frequencies specified in Table 9-1 apply to that batch. If different equipment (e.g., manifold with the carbon composite filter sampling head) is used to collect samples through the carbon composite filters of drums designated for the same bin, these samples constitute a second sample batch for which QC samples must also be collected in accordance with the frequencies specified in Table 9-1.

Field Blanks (FBs). FBs shall be used to evaluate background levels. FBs must be collected prior to sample collection at a frequency one per bin or one per twenty samples collected, whichever is more frequent, for VOC analysis. The site Project Manager shall use the FB data to assess its impact on the sample results.

Equipment Blanks (EBs). EBs shall be used to assess the cleanliness of the sampling equipment prior to first use of the equipment for sample collection. After the initial cleanliness check, EBs must be collected at a frequency of one per bin or one per twenty samples collected, whichever is more frequent, for VOC analysis. The site Project Manager shall use the EB data to assess its impact on the sample results and have the responsibility to ensure corrective action measures are taken when EB data indicates equipment contamination.

Field Reference Standards (FRSs). FRSs shall be used to assess the accuracy with which the sampling equipment can collect VOC, hydrogen, methane, and NO_x (if applicable) samples into SUMMA® canisters prior to first use of the equipment for sample collection. After the initial accuracy check, FRSs must be collected at a frequency of one per bin. FRS results shall be acceptable if the QAOs specified in Section 3.3 are met. 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 that corrective action measures are taken when the QAOs are not achieved.

Field Duplicates (FDs). Field 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 one per twenty samples collected, whichever is more frequent. Field

Table 9-1. Summary of Drum Field QC Headspace Sample Frequencies

QC Samples	Routine
Field blanks[a]	1/bin or 1/20 samples collected, whichever is more frequent
Equipment blanks[b]	1/bin or 1/20 samples collected, whichever is more frequent
Field Reference Standards[c]	1/bin
Field duplicates	1/bin or 1/20 samples collected, whichever is more frequent

[a] Analysis of field blanks for VOCs, only, is required.

[b] One equipment blank sample must be collected, analyzed for VOCs, and demonstrated clean prior to first use of the headspace sampling equipment with each of the sampling heads, then at the specified frequency, for VOCs only thereafter. Daily, prior to work, the sampling manifold, if in use, must be verified clean using an organic vapor analyzer.

[c] One field reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Section 3.3 prior to first use, then at the specified frequency thereafter.

duplicate results shall be acceptable if the QAOs specified in Section 3.3 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 Bin Headspace Sampling

Quality control checks shall be accomplished in the field or in the laboratory. Required bin QC sample frequencies are summarized in Table 9-2 for initial and routine operations.

Equipment Blanks (EBs). EBs shall be used to assess the cleanliness of the bin sampling equipment prior to first use of the equipment for sample collection. After the initial cleanliness check, EBs must be collected at a frequency of one per bin, prior to sample collection. The site Project Manager shall use the EBs data to assess its impact on the sample results and have the responsibility to ensure that corrective action measures are taken when EB data indicates the bin sampling equipment is contaminated.

Field Reference Standards (FRSs). FRSs shall be used to assess the accuracy with which the bin sampling equipment can collect headspace samples into SUMMA® canisters prior to first use of the bin sampling equipment for sample collection. After the initial accuracy check, FRSs must be collected at a frequency of one per bin for the first two bins, then one per five bins thereafter. FRS results shall be acceptable if the QAOs specified in Section 3.3 are met. 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 that corrective action measures are taken when the QAOs are not achieved.

Field Duplicates (FDs). Field duplicate bin samples must 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 during both initial and routine operations. Field duplicate results shall be acceptable if the QAOs specified in Section 3.3 are met. 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 that corrective action measure are taken when the QAOs are not achieved.

9.1.5 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).

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

QC Samples	First Two Bins	Routine
Equipment Blanks[a]	1/bin, prior to work	1/bin, prior to work
Field Reference Standards[b]	1/bin	1/5 bins
Field Duplicates	1/bin	1/bin

[a] One equipment blank must be collected, analyzed for VOCs, and demonstrated clean prior to first use then at the specified frequency thereafter.

[b] One field reference standard must be collected, analyzed, and demonstrated to meet the QAOs specified in Section 3.3 and Section 3.6 prior to first use then at the specified frequency thereafter.

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 item, material (residual or packaging), or waste material category 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 in order 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 QA program (Section 1.10). Laboratory QA programs must specify qualitative and quantitative acceptance criteria for the QC checks for this program and corrective action measures to be taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, procedure 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 QA program. Laboratory QA programs must specify qualitative and quantitative acceptance criteria for the QC checks for this program and corrective action measures to be taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, procedure proficiency samples, and blind audit samples. In addition, acceptable method performance must be demonstrated prior to the analysis of samples.

9.2.1.1 Initial and Continuing Procedure Demonstration

All laboratories analyzing samples in support of this Program must demonstrate acceptable performance prior to the analysis of any samples. Demonstration of acceptable performance shall be achieved by analyzing commercially available gas standards and demonstrating acceptable performance in the

Performance Demonstration Program. These standards must contain all of the analytes listed in Table 3-7 at concentrations appropriate to determine parameters specified in Table 3-7. Initially, 30 replicate standards shall be analyzed in order to demonstrate that the criteria specified for precision and accuracy listed in Table 3-7 can be met. In addition, method detection limits must be initially determined for all analytes listed in Table 3-7 according to Glaser (1981). Demonstration of acceptable procedure performance shall be repeated semiannually by the analysis of seven replicates. Procedures for the evaluation of procedure performance data are given in Section 12. All procedure performance standards (replicates) used to determine precision and accuracy must be at the same concentration.

It shall be the responsibility of the laboratory QA Officer to monitor and document procedure performance. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when acceptable procedure performance is not met.

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 H_2, CH_4, and some, if appropriate, of the remaining 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 one per analytical batch. 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 the 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.

Equipment Blanks (EBs). SUMMA® canisters shall be taken to the field to monitor the cleanliness of the sampling equipment. EBs shall be collected by attaching a clean canister to the sampling equipment and filling it with 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 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

The laboratory shall analyze field samples in duplicate at a minimum frequency of one per analytical batch. 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 they meet the QAOs specified in Table 3-7. 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 QA program. Laboratory QA programs must specify qualitative and quantitative acceptance criteria for the QC checks for this program and corrective action measures to be taken when these criteria are not satisfied. Specific QC practices shall include laboratory duplicates, laboratory blanks, laboratory control samples, procedure proficiency samples, and blind audit samples.

9.2.2.1 Initial and Continuing Procedure Demonstration

All laboratories analyzing samples for this program must demonstrate acceptable procedure 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 or laboratory-prepared standards and demonstrate acceptable performance in the Performance Demonstration Program. These standards must contain all of the analytes listed in Table 3-8 at concentrations appropriate to determine the parameters specified in Table 3-6. Initially, 30 replicate standards shall be analyzed in order to demonstrate that the criteria for precision and accuracy listed in Table 3-6 can be met. In addition, method detection limits must be initially determined for all analytes listed in Table 3-6 according to Glaser (1981). Demonstration of acceptable procedure

performance shall be repeated semiannually by the analysis of seven replicates. Procedures for the evaluation of procedure performance data are given in Section 12. All procedure performance standards (replicates) used to determine precision and accuracy must be at the same concentration.

It shall be the responsibility of the laboratory QA Officer to monitor and document procedure performance. The laboratory QA Officer and technical supervisor shall have responsibility to implement corrective actions when acceptable procedure 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 a minimum of six of the analytes listed in Table 3-6. 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 one per analytical batch. 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 equipment blanks must be analyzed.

Equipment Blanks (EBs). EBs shall be collected by attaching a clean SUMMA® canister to the sampling equipment and filling it with high purity zero air or nitrogen. 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.

Laboratory Blanks (LBs). SUMMA® canisters must be filled with high purity zero air or nitrogen 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.

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 samples shall be analyzed in duplicate at a minimum frequency of one per analytical batch.

Field canisters analyzed in duplicate shall be used to assess laboratory precision. Laboratory duplicate results shall be acceptable if the QAOs specified in Table 3-6 are satisfied. 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 for the continuing calibration standards are within the range of 50% to 200% of the average internal standard area counts from the most recent five-point calibration, and for samples if the area counts are within 50% to 200% of the latest continuing calibration standards. 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.

9.2.3 Flammability Testing

Flammability testing provides a qualitative evaluation of whether or not bin headspace samples are flammable. Operator training and experience are critical for assuring the quality of flammability test results, and only trained personnel shall operate the flame test apparatus. Training requirements shall be specified in site QAPjPs and SOPs.

The samples of an analytical batch shall be analyzed by the same flame test operator who performed the initial calibration and continuing calibrations associated with that analytical batch. Calibration requirements and frequencies are summarized in Table 6-3. All field sample duplicates shall be analyzed. Laboratory duplicates shall be analyzed at a frequency of one per analytical

batch. For flammability testing, an analytical batch shall consist of not more than 20 samples. The 1.0 v/v% diethyl ether initial calibration standard is a functional blank, and no additional blanks are 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 WIPP 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 PDP (Section 1.11). DOE/WPIO shall be responsible for administering the PDP.

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/WPIO 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 applicable QAPjP. 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 determine the cause of the adverse finding and 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 the 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 Field Office.

Copies of all WACCC audit reports identifying any nonconformances shall be sent to the appropriate DOE Field Office, DOE/EM-34, and DOE/WPSO. 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/WPSO and DOE/EM-34.

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 Field 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 be responsible for ensuring that a minimum of two internal audits of program activities are conducted annually. 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 analyzing 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 (USDOE, 1991a). The DOE-designated, independent organization shall compile, review, and report the results of the performance evaluation to DOE/WPIO.

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/WPIO shall provide written notification of the adequacy of an analytical laboratory and approval of its participation in the Program to the appropriate DOE Field Office management.

11.0 PREVENTIVE MAINTENANCE

As specified by ASME NQA-1, Element 10, inspection required to verify comformance of an item or activity shall be planned, executed, and documented. Preventive maintenance must have two aspects: (1) a schedule of preventive maintenance activities to ensure the accuracy of measurement systems 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, and MDL for the program are as follows.

12.1 Precision

Precision is 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:

$$RPD = \frac{C_1 - C_2}{(C_1 + C_2) / 2} * 100 \qquad (12\text{-}1)$$

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:

$$RSD = s / \bar{y} * 100 \qquad (12\text{-}2)$$

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

The standard deviation, s, is defined as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n - 1}} \qquad (12\text{-}3)$$

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 determined as the percent recovery (%R) and may be expressed as relative percent accuracy (RPA).

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

$$\%R = \frac{C_m}{C_{srm}} * 100 \qquad (12\text{-}4)$$

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:

$$\%R = \frac{S - U}{C_{ss}} * 100 \qquad (12\text{-}5)$$

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

Relative percent accuracy is given as,

$$100 - \%R \tag{12-6}$$

12.3 Completeness

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

$$\%C = \frac{V}{n} * 100 \tag{12-7}$$

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:

$$Comparability = \frac{CC}{TD} * 100 \tag{12-8}$$

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:

$$MDL = t_{(\eta-1,1-\alpha=.99)} * s \tag{12-9}$$

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 (Glaser, 1981).

12.6 Procedures for Assessing Compliance With Data Quality Objectives and Regulatory Compliance Limits

Statistical procedures are needed to assess compliance with NMD requirements for potential headspace gas flammability, waste comparability, and no-migration finding. These procedures must be applied to laboratory analytical data prior to comparing these data with the regulatory limits specified in Section 1.3.2.

12.6.1 Statistical Procedure for Assessing Compliance With NMD Requirements

The statistical procedure that must be used to assess compliance with regulatory limits established in the NMD is based on the following:

- Laboratory precision and accuracy data resulting from procedure performance testing
- Normal distribution of laboratory procedure performance data (population standard deviation (σ) is approximately equivalent to the sample standard deviation (s) for 30 laboratory procedure performance samples)
- Homogeneous nature of the sample matrix
- Majority of the quantifiable sampling and measurement error due to laboratory error.

Upper confidence limit values (concentration or LEL) are calculated using the following equations:

$$X_{UL} = \Sigma\, a_i\, X_{ci} + \Sigma\, a_i\, S_i\, Z_{\alpha=0.1} \qquad (12\text{-}10)$$

$$X_{ci} = \frac{100\, X_{mci}}{\%R_i} \qquad (12\text{-}11)$$

$$S_i = \frac{(\%RSD_i)\, X_{mci}}{\%R_i} \qquad (12\text{-}12)$$

where

X_{UL}	=	90% upper confidence limit value of the measured concentrations
a_i	=	Independent variable (for flammability calculations, this is the reciprocal of the LEL; for comparability and no-migration finding, the variable is one)
X_{ci}	=	Measured concentration of compound i, corrected for recovery
S_i	=	Standard deviation of compound i
$Z_{\alpha=0.10}$	=	Standard normal deviate (Z-value) at 90% confidence level (one-tail) is 1.29

$\%RSD_i$ = Percent relative standard deviation of compound i (obtained from laboratory procedure performance data)

$\%R_i$ = Percent recovery of compound i (obtained from laboratory procedure data)

X_{mci} = Experimentally measured concentration of compound i.

Equation 12-10 must be used to determine compliance with the NMD waste characterization requirements. Upper confidence limit values shall be calculated using Equation 12-10 and compared to regulatory limits as described in Sections 1.3 and 8.7. A value of one-half the MDL must be used when flammable VOCs are not detected.

12.6.2 Procedure for Obtaining Precision and Accuracy Data Used in Determining X_{UL}

The overall approach to evaluating procedure performance data is shown in Figure 12-1. Initially, precision and accuracy shall be assessed using procedure performance data obtained from the analysis of 30 laboratory reference standards (initial laboratory procedure demonstration). The precision and accuracy obtained from this initial data set shall be used to determine X_{UL}. Precision and accuracy data for all procedures shall be continuously monitored by the analysis of seven replicates every six months.

The precision and accuracy obtained from the first continuing data set must be compared with the initial procedure performance data set. Comparison of the initial and continuing data sets must be made at the 95% confidence level using established procedures (Taylor, 1987; Dowdy, 1983), as described below. If no statistical differences exist between the initial and continuing data sets, the two sets of precision and accuracy data shall be pooled. The precision and accuracy from the pooled data shall then be used to determine X_{UL}. Subsequent continuing data sets must be statistically compared to the most recent pooled data set and the two sets combined to form a new pooled data set if no statistical difference exists.

If a significant statistical difference does exist at the 95% confidence level between the initial or pooled and the continuing data sets, another set of seven replicates must be analyzed. If no statistical difference exists at the 95% confidence level between the two sets of seven replicates, the precision and accuracy of these two seven-replicate sets shall be pooled. The precision and accuracy from this pooled data shall be used to determine X_{UL}. Corrective actions must be taken if two consecutive data sets of seven replicates are not statistically equivalent.

The process of analyzing seven replicates and comparing the resulting precision and accuracy data to the most recent pooled data must be repeated semiannually. If the resulting pooled data set has less

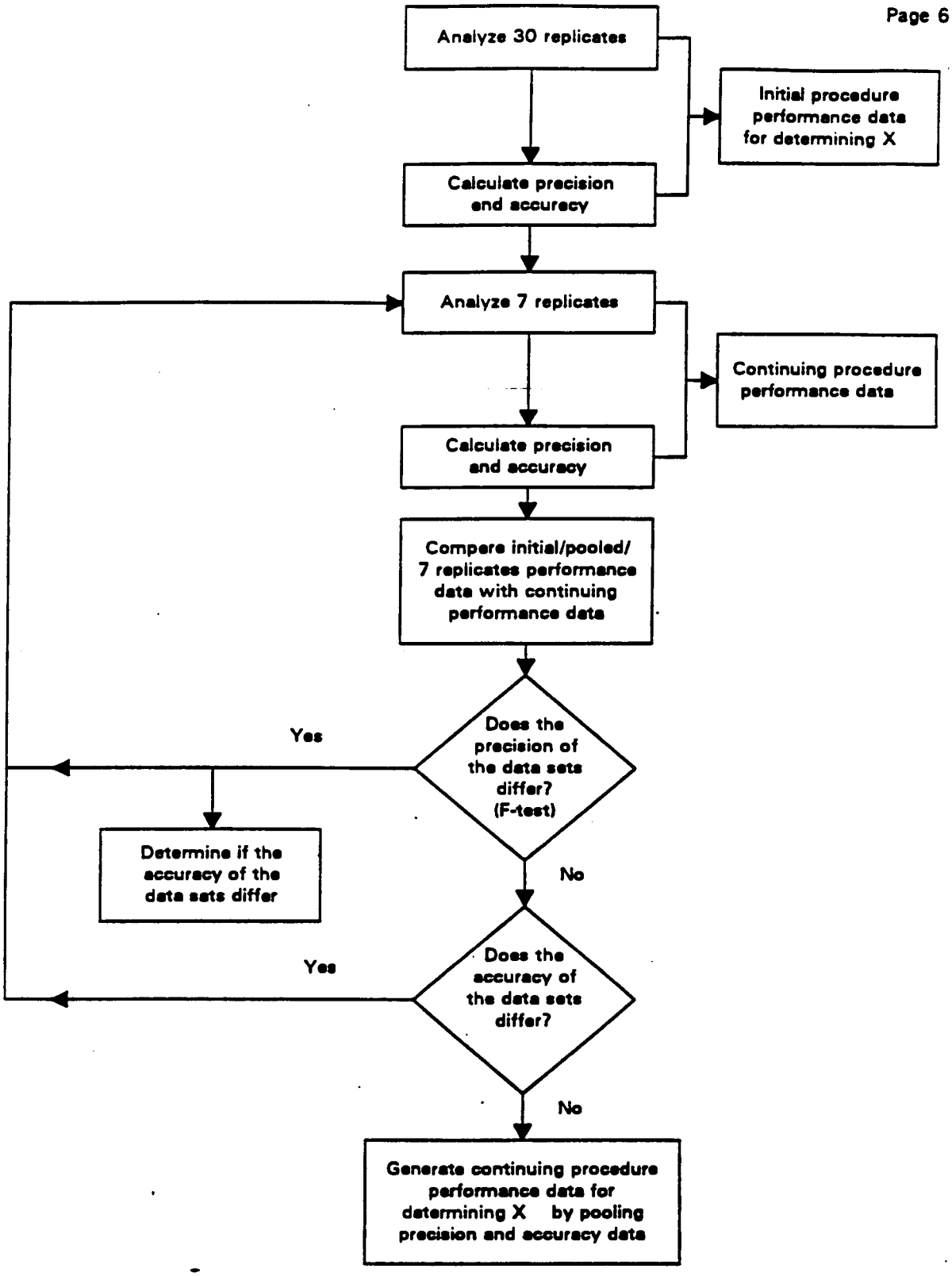


Figure 12-1. Statistical Analysis of Procedure Performance Data

than 30 samples, the impact on calculating X_{UL} values must be evaluated. Procedures should be implemented to ensure that the pooled data set contains only data generated within a two-year period.

12.6.2.1 Analysis of Precision

The F-test shall be used to determine if the precisions obtained from different data sets are statistically the same. Comparison of data sets shall be done at the 95% confidence level (two-tailed). A calculated F-value is determined by taking the ratio of the two data set variances:

$$F_{v1,\,v2} = s_1^2 \,/\, s_2^2 \qquad (12\text{-}13)$$

where

v_1 = number of degrees of freedom for data set 1

v_2 = number of degrees of freedom for data set 2

s_1 = standard deviation of data set 1

s_2 = standard deviation of data set 2.

When calculating F values, s_1 and s_2 must be chosen so that s_1 is greater than s_2, and therefore s_1^2/s_2^2 is always greater than one. This calculated F value is then compared to the critical F_c value found in tables of critical F values (Abramowitz, 1972). The F_c value must be obtained at the 95% confidence level with the degrees of freedom (v_1,v_2) such that v_1 is degrees of freedom for the numerator and v_2 is the degrees of freedom of the denominator. The precision obtained from two data sets is statistically equivalent if the calculated F value is less than the critical F_c value.

12.6.2.2 Analysis of Accuracy

The accuracy obtained from two data sets shall be evaluated at the 95% confidence level (two-tailed) by comparing the data set average using the t-statistic. If the standard deviations of two data sets are statistically equivalent by the F-test, a pooled estimate of the standard deviation can be calculated as follows:

$$s^2 = [(n_1 - 1)\, s_1^2 + (n_2 - 1)\, s_2^2\,]/(n_1 + n_2 - 2) \qquad (12\text{-}14)$$

where

n_1 = number of samples in data set 1

n_2 = number of samples in data set 2.

The t value is given by:

$$t_v = (\bar{X}_1 - \bar{X}_2) / [s\,(1/n_1 + 1/n_2)^{1/2}] \qquad (12\text{-}15)$$

where

v = number of degrees of freedom $(n_1 + n_2 - 2)$

$\bar{X}_1$ = mean concentration of data set 1

$\bar{X}_2$ = mean concentration of data set 2.

If the standard deviations of the two data sets are significantly different, the t value is given by:

$$t_v = (\bar{X}_1 - \bar{X}_2) / \left[\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right]^{1/2} \qquad (12\text{-}16)$$

With the degrees of freedom, v, given as:

$$v = \left[\frac{\left[\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right]^2}{\frac{(s_1^2/n_1)^2}{n_1 + 1} + \frac{(s_2^2/n_2)^2}{n_2 + 1}}\right] - 2 \qquad (12\text{-}17)$$

For either case, the mean concentrations of the two data sets are not significantly different if the t value obtained is less than the critical value of t_c (Abramowitz, 1972; Taylor, 1987; Dowdy, 1983). If the mean concentrations do not differ significantly, then the accuracies for the two data sets do not differ.

12.6.2.3 Procedures to Obtain Pooled Data Sets

Estimates of the standard deviation shall be pooled using the following equation:

$$s_{pooled} = \left[\frac{v_1 s_1^2 + v_2 s_2^2}{v_1 + v_2}\right]^{1/2} \qquad (12\text{-}18)$$

Mean values shall be pooled using the following equation:

$$\bar{X}_{pooled} = \frac{n_1\bar{X}_1 + n_2\bar{X}_2}{n_1 + n_2} \qquad (12\text{-}19)$$

12.6.2.4 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 of all values.

Q = (suspect value - nearest value)/(largest value - 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 12-1.

12.6.2.5 Procedures for Evaluating NMD Flammability Quality Assurance Objectives

This section describes the procedure that must be used to demonstrate compliance with the NMD requirement that all flammability testing meets the quality assurance objectives (QAOs) of ±10% precision and accuracy. This requirement must be met to demonstrate compliance with QAOs for flammable VOCs as a class.

The following procedure must be followed:

1. Obtain 30 sets of analytical data for flammable VOCs, as described in Section 12.6.2.
2. Sum the measured concentrations of each of the flammable VOCs to yield 30 values for the sum of flammable VOCs.
3. Average the results of the 30 summations from step 2.
4. Sum the concentrations of the flammable VOC standards that are obtained for the same set of flammable VOC samples.
5. To determine the accuracy, divide the average sum of measured flammable VOCs by the sum of the standard or known concentrations of flammable VOCs.
6. To determine the precision, calculate the relative standard deviation of the 30 values for the sum of flammable VOCs using Equation 12-23.

Table 12-1. 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

7. This procedure must be applied to the seven sets of analytical data collected semiannually, as described in Section 12.6.2.

Figure 12-2 provides an example of how the data should be reported. Equations 12-20 through 12-23 are used to calculate the required information related to the performance data using the 30 replicates for each flammable VOC.

$$T_i = \sum_{j=1}^{m} X_{ij} \tag{12-20}$$

$$\bar{T} = \frac{\sum_{i}^{n} T_i}{n} \tag{12-21}$$

$$KT = \sum_{j=1}^{m} K_j \tag{12-22}$$

$$S_T = \sqrt{\frac{\sum_{i=1}^{n} (T_i - \bar{T})^2}{n - 1}} \tag{12-23}$$

where:

$X_{i,j}$ = Measured concentration (ppmv) of the ith determination of flammable VOC "j"

T_i = Total measured flammable VOC concentration (ppmv) of the ith determination

$\bar{T}$ = Average of the total measured flammable VOC concentration (ppmv) for all determinations

K_j = Known concentration of flammable VOC "j"

KT = Total of the known flammable VOC concentrations (ppmv)

S_T = Standard deviations of the total measured flammable VOC concentrations for all "i" determinations.

Assume there are "m" different, flammable VOCs and there a total of "n" analyses performed of each standard.

Flammable Analytes (j)	Determination Number (i)				Avg	Std Dev (S_j)	Known Value (K_j)
	1	2	3	4... \ \ n			
1	$X_{1,1}$	$X_{2,1}$	$X_{3,1}$	$X_{4,1}$...\ \ $X_{n,1}$	$\bar{X}_1$	S_1	K_1
2	$X_{1,2}$	$X_{2,2}$	$X_{3,2}$	$X_{4,2}$...\ \ $X_{n,2}$	$\bar{X}_2$	S_2	K_2
3	$X_{1,3}$	$X_{2,3}$	$X_{3,3}$	$X_{4,3}$...\ \ $X_{n,3}$	$\bar{X}_3$	S_3	K_3
4	$X_{1,4}$	$X_{2,4}$	$X_{3,4}$	$X_{4,4}$...\ \ $X_{n,4}$	$\bar{X}_4$	S_4	K_4
\	\	\	\	\	\	\	\
\	\	\	\	\	\	\	\
m	$X_{1,m}$	$X_{2,m}$	$X_{3,m}$	$X_{4,m}$...\ \ $X_{n,m}$	$\bar{X}_m$	S_m	K_m
SUMS:	T_1	T_2	T_3	T_4 ...\ \ T_n	$\bar{T}$	S_T	KT

Figure 12-2. Calculation Method for Evaluating Performance Data In Relation to QAOs for Flammability Testing

To meet accuracy QAO, the following must be demonstrated:

$$0.90 \leq \frac{\bar{T}}{KT} \leq 1.10 \qquad (12\text{-}24)$$

$$\frac{S_T}{\bar{T}} \leq 0.10 \qquad (12\text{-}25)$$

12.6.3 Procedures for Determining Emplacement Time During Which H_2/CH_4 Remain Below 0.5 LEL in Experimental Bins

The emplacement time during which H_2/CH_4 concentrations remain below 0.5 LEL levels inside a bin is calculated iteratively using the numerical solutions to the differential equations which describe the mass balances on H_2/CH_4 within each confinement volume (bin, SWB or TRUPACT-II) prior to emplacement in the WIPP. The analysis to determine the maximum emplacement times is similar to the analysis presented in the TRUPACT-II Safety Analysis Report (SAR) for determining aspiration times (NuPac, 1989). The emplacement times derived in this section refer to compliance with the NMD criteria, and do not indicate that the H_2/CH_4 concentrations exceed flammable levels beyond these times. In fact, the conservative analysis used in deriving the decay heat limits in the TRUPACT-II SAR ensure that waste containers are safe (i.e., less than H_2/CH_4 LEL) at all times with respect to H_2/CH_4 concentrations. Two sets of differential equations have been solved numerically for the following configurations:

1. An experimental bin inside a standard waste box (SWB) venting to the atmosphere at the bin preparation site. This represents the time the bin is in interim storage at the DOE generator/storage site.

2. Two experimental bins each inside a standard waste box which are present inside the Inner Containment Vessel (ICV) of a TRUPACT-II. This represents the time the bin is being transported.

The following equations describe mass balance of H_2/CH_4 for bins in interim storage:

$$dX_{hdspc}/dt = C_1 - C_2 (X_{hdspc} - X_{bin}) \qquad (12\text{-}26)$$

$$dX_{bin}/dt = C_3(X_{hdspc} - X_{bin}) - C_4(X_{bin} - X_{SWB}) \qquad (12\text{-}27)$$

$$dX_{SWB}/dt = C_5(X_{bin} - X_{SWB}) - C_6 X_{SWB} \qquad (12\text{-}28)$$

The following equations describe mass balance of H_2/CH_4 during transport in TRUPACT-II:

$$dX_{hdspc}/dt = C_1 - C_2(X_{hdspc} - X_{bin}) \quad (12\text{-}29)$$

$$dX_{bin}/dt = C_3(X_{hdspc} - X_{bin}) - C_4(X_{bin} - X_{SWB}) \quad (12\text{-}30)$$

$$dX_{SWB}/dt = C_5(X_{bin} - X_{SWB}) - C_6(X_{SWB} - X_{TRU}) \quad (12\text{-}31)$$

$$dX_{TRU}/dt = C_7(X_{SWB} - X_{TRU}) \quad (12\text{-}32)$$

where

C_1 = $CG \times R \times T/(P \times V_1)$

X_{hdspc} = mole fraction H_2/CH_4 in the void volume within the two bin liners (i.e., the bin headspace)

X_{bin} = mole fraction H_2/CH_4 in the annular volume between the bin liners and the bin

X_{SWB} = mole fraction H_2/CH_4 in the SWB void volume outside the experimental bin

X_{TRU} = mole fraction H_2/CH_4 in the TRUPACT-II ICV outside the SWBs

C_2 = The effective release rate of H_2/CH_4 across the two bin liners in series divided by the void volume within the bin liners (day^{-1})

C_3 = The effective release rate of H_2/CH_4 across the two bin liners in series divided by the void volume outside of the bin liners (i.e., the volume between the bin and the liners) (day^{-1})

C_4 = The effective release rate of H_2/CH_4 across two bin filters in parallel divided by the void volume outside of the bin liners (day^{-1})

C_5 = The effective release rate of H_2/CH_4 across two bin filters in parallel divided by the SWB void volume outside the experimental bin (i.e., the volume between the bin and the SWB) (day^{-1})

C_6 = The effective release rate of H_2/CH_4 across two SWB filters in parallel divided by the SWB void volume outside the experimental bin (day^{-1})

C_7 = The effective release rate of H_2/CH_4 across four (two from each SWB) SWB filters in parallel divided by the TRUPACT-II ICV void volume outside the SWBs (day^{-1})

t = Time (day)

CG = The allowable flammable gas generation rate per experimental bin (mole/day)

R = The gas law constant (0.08206 atm liter/mole °K)

T = Absolute temperature (294 °K) (NuPac, 1989)

P = Absolute pressure (1 atm) (NuPac, 1989)

V_1 = The void volume within the bin liners (liter).

These systems of differential equations were solved numerically using the Runge-Kutta Fourth Order numerical integration method (Perry et al., 1984; Reklaitis, et al., 1983). Numerical solution implies obtaining the mole fractions of H_2/CH_4 in each confinement volume as a function of time and by waste type.

12.6.3.1 Input Parameters

Several parameters determine the maximum allowable emplacement times for each of the waste types. The values of the parameters are identical to those used in the TRUPACT-II SAR (NuPac, 1989). These parameters are pressure, temperature, allowable H_2/CH_4 generation rates, H_2/CH_4 release rates across layers of confinement, void volumes, and transportation time inside the TRUPACT-II.

Shipping Period Duration Inside TRUPACT-II

The TRUPACT-II SAR shipping Package (NuPac, 1989) provides an analysis of transit times which indicates that "The normal transit time ranges from 0.6 day for shipments from Rocky Flats Plant to 1.7 days for shipments from Hanford." For the purpose of conservatism, three days is assumed for a maximum normal transit time. In determining the emplacement times, a transit period of three days has been used in the calculations.

Flammable Gas Release Rates

The release rates of H_2/CH_4 across each confinement layer are based on the lowest measured release rates (NuPac, 1989) and provide a margin of safety by predicting the smallest allowable emplacement times.

Void Volumes for Waste Types

The void volumes used in the calculations were based on the data from NuPac (1989), and Molecke (1990a).

H_2/CH_4 Generation Rates

The allowable H_2/CH_4 generation rates used in the calculations are the bounding values for each waste type, and are dependent on the packaging configurations within the waste containers (NuPac, 1989). These gas generation rates are the bounding values used to determine the decay heat limits in the TRUPACT-II SAR, and ensure that the gas concentrations in the bins remain below flammable levels at all times (NuPac, 1989). The emplacement times derived in this section refer to compliance with the NMD criteria, and are not meant to imply that gas concentrations exceed flammable levels beyond these times. In fact, the conservative analysis used in deriving the decay heat limits in the TRUPACT-II SAR ensure that waste containers are safe at all times with respect to H_2/CH_4 concentrations.

12.6.3.2 Tables of Allowable Emplacement Times

Two options are available to determine the emplacement time during which $H2/CH_4$ will remain below 50% of the mixture LEL. The first option is based on the length of time which has elapsed since the

bin was prepared. Table 12-2 summarizes the emplacement times at the time of bin preparation for the various shipping categories. The second option is based on sampling the bin headspace for H_2/CH_4. The emplacement times for use with this option are summarized in Tables 12-3 through 12-7. It should be noted that the transportation time has been included in these emplacement times.

Table 12-2. Allowable Emplacement Times Based on the Date of Bin Preparation[a]

Waste Type	Shipping Category	Emplacement Time (Days)
I	I.1D2, I.2D2, I.3D2	123
II	II.1D2	152
III	III.1D2	140
III	III.1D5	160
IV	IV.1D2	125

[a] This option and the corresponding times should be used if no sampling of headspace gases is to be performed after bin preparation.

Table 12-3. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type I, Shipping Categories I.1D2, I.2D2, I.3D2

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	123
0.1	119
0.2	114
0.3	110
0.4	105
0.5	100
0.6	95
0.7	90
0.8	84
0.9	78
1.0	73
1.1	66
1.2	60
1.3	54
1.4	47
1.5	39
1.6	32
1.7	24
1.8	16
1.9	8

Table 12-4. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type II, Shipping Category II.1D2

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	152
0.1	147
0.2	142
0.3	136
0.4	130
0.5	124
0.6	118
0.7	112
0.8	105
0.9	99
1.0	91
1.1	84
1.2	76
1.3	68
1.4	60
1.5	51
1.6	41
1.7	31
1.8	21
1.9	11

Table 12-5. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type III, Shipping Category III.1D2

Mole Percent H_2/CH_4 Gas	Emplacement Time (Days)
0.0	140
0.1	135
0.2	130
0.3	125
0.4	120
0.5	115
0.6	109
0.7	103
0.8	97
0.9	91
1.0	85
1.1	78
1.2	71
1.3	63
1.4	55
1.5	47
1.6	38
1.7	29
1.8	20
1.9	10

Table 12-6. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type III, Shipping Category III.1D5

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	160
0.1	154
0.2	147
0.3	141
0.4	134
0.5	127
0.6	120
0.7	113
0.8	106
0.9	98
1.0	91
1.1	83
1.2	75
1.3	66
1.4	57
1.5	48
1.6	39
1.7	30
1.8	20
1.9	9

Table 12-7. Allowable Emplacement Times Based on Headspace Gas Sampling for Waste Type IV, Shipping Category IV.1D2

Mole Percent H_2/CH_4	Emplacement Time (Days)
0.0	125
0.1	121
0.2	116
0.3	111
0.4	107
0.5	101
0.6	96
0.7	91
0.8	85
0.9	80
1.0	74
1.1	67
1.2	61
1.3	54
1.4	47
1.5	40
1.6	32
1.7	24
1.8	16
1.9	7

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 the 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 memoranda, 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 Field Office. Documentation of nonconformances shall be made available to the site Project Manager, who is responsible for notifying project personnel of the nonconformance. Completion of the corrective action for 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 4-1) 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 Field 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 Field 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 Field Office (Section 10.0). DOE/WPIO shall provide the results of the PDP and notification of the analytical laboratory's adequacy in meeting program requirements to the DOE Field Office (Section 10.0).

15.0 DEFINITIONS

ABSOLUTE CANISTER PRESSURE - Pressure measured relative to absolute 0 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 a similar matrix that is processed as a unit within a specific time period. An analytical batch must not exceed 20 samples, all of which must be received by the laboratory within 14 days of the validated time of sample receipt (VTSR) of the first sample of the batch.

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.

BINARY MIXTURE - A mixture of hydrogen and methane in air.

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

CALIBRATION BLANK - A sample volume containing undetectable quantities of analytes.

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

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

COMPLETENESS - The percentage of measurements made 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 (e.g., concentration and 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 DOE goals.

EMPLACEMENT - When the bin instrumentation is connected to the Radiological Control Barrier as required for conducting the bin-scale tests.

EMPLACEMENT ACTIVITIES - Those activities associated with waste handling within the Salado Formation (i.e., within the unit boundary as defined in the NMD).

EMPLACEMENT TIME - The time from when the waste is loaded in a bin and the bin is closed and placed in a Standard Waste Box through emplacement in the WIPP underground without exceeding 50% of the LEL for the binary mixture of H_2/CH_4.

EMPLACEMENT TIME WINDOW - The emplacement date minus four weeks.

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

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. 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.

FLAME TEST - The procedure specified in Guidance Manual Procedure 810.1 for determining the flammability of headspace samples containing concentrations of flammable VOCs greater than 500 ppmv.

FLAMMABLE MIXTURE - (1) A binary mixture with concentrations of hydrogen and methane exceeding 50% of the theoretical LEL, or (2) a mixture of flammable VOCs in air that propagates a flame during a flame test.

FLAMMABLE VOC - A VOC identified as noncombustible or considered, by EPA, not to be a significant fire hazard under WIPP repository conditions. Flammable VOCs that are evaluated for the Program are listed in Table 1-5.

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.

GASES - The analytes listed in Table 3-6 or the Program-required subset of the Table 3-6 analytes.

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.

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

HIGH PURITY GAS - Gas certified by the manufacturer to contain less than 1 ppm total VOCs.

HOLDING TIME - The maximum permissible time allowed between time of sample collection and time of analysis.

INDEPENDENT STANDARD - Laboratory prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

INNERMOST LAYER OF CONFINEMENT - Within a waste container, a plastic bag that is closest to waste that may be a source of VOCs and/or hydrogen and methane and has a minimum of one liter of headspace.

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

INSTRUMENT DETECTION LIMIT (IDL) - The minimum signal that the instrument can detect with 99% confidence that the analyte concentration is greater than zero. Determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INTERFERENTS - Substances which affect the analysis for the element or compound of interest.

ITEM DESCRIPTION CODE - A site-specific, internal numerical code applied to individual waste forms to provide identification which is used for physical segregation and computerized recordkeeping and tracking.

LABORATORY BLANK - An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sampling processing. The laboratory blank is used to document contamination resulting from the laboratory sample preparation and analytical process.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition. Gas laboratory control samples are analyzed using the same analytical methods employed for the DOE-WIPP samples received.

LABORATORY 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.

LABORATORY RECEIPT DATE - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

LE CHATELIER FORMULA - A simple formula of additive character that connects the lower explosive limits of two or more fuels (flammable gases and/or VOCs) with the lower explosive limit of any mixture of them.

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the analytical curve remains linear.

LOWER EXPLOSIVE LIMIT - The lowest concentration of a fuel (flammable gas and/or VOC) in air that will propagate a flame from an ignition source. The lower explosive limit (LEL) is synonymous with the lower limit of flammability (LLF). LEL values are typically expressed as the volume of fuel per volume of air (v/v%).

MANAGEMENT APPRAISAL - A determination of managerial effectiveness in establishing and implementing quality assurance program plans which conform to Departmental policy requirements. It is based on an analysis of functional appraisals, internal audits, and other information, and on the application of appropriate criteria. It is a review and evaluation of management performance covering all quality assurance and management responsibilities to assure proper quality assurance program balance.

METHOD BLANK - An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank must be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

METHOD DETECTION LIMIT (MDL) - The minimum concentration of a substance that can be measured and reported for a given method with 99% confidence that the analyte concentration is greater than zero. MDL is determined from analysis of a sample in a given matrix type containing the analyte of interest.

NARRATIVE (Case Narrative) - Portion of the data package which includes laboratory, contract, Case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete Case Narrative specifications are included in Exhibit B.

NITROGEN OXIDES - The sum of the measured concentrations of NO and NO_2.

OUT OF CONTROL - One or more of several conditions relating to the plotting of control data and indicating unacceptable results.

PACKAGING MATERIAL - Flexible containment materials, e.g., plastic bags.

PERCENT DIFFERENCE (%D) - The difference between the average initial calibration response factor and the continuing calibration response factors divided by the average initial calibration response.

PERFORMANCE ASSESSMENT - A determination of the long-term performance of the WIPP disposal system in accordance with the requirements of the EPA Standard, 40 CFR 191, Subpart B.

PERFORMANCE EVALUATION (PE) SAMPLE - A sample of known composition provided as a single-blind sample to the analytical laboratory. Used by DOE to evaluate analytical laboratory performance.

PRECISION - A measure of mutual agreement among individual measurements of the same property, made under prescribed similar conditions; often expressed in terms of a standard deviation.

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

PROGRAM REQUIRED DETECTION LIMIT (PRDL) - Minimum level of detection acceptable under the WIPP-QAPP.

PROGRAM REQUIRED QUANTITATION LIMIT (PRQL) - Minimum level of analyte quantitation acceptable under the WIPP-QAPP. An analyte PRQL should be a minimum of three times the MDL.

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 the use of 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, defensible, 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) - The 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.

RCRA WASTE CATEGORY - A waste classification established in the WIPP Waste Analysis Plan to describe TRU mixed waste and to provide a basis for RCRA waste verification efforts by specifying sampling and analytical requirements.

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

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

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 or packaging material.

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.

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.

SUPERCOMPACTED WASTE - Supercompaction is a volume reduction process.

TARGET COMPOUNDS - Those gases and VOCs identified by the Program as analytes. Gas target compounds are listed in Table 3-6; VOC target compounds in Table 3-7.

VOLATILE ORGANIC COMPOUNDS - The target compounds listed in Table 3-7 and any additional compounds tentatively identified by the VOC analytical procedures used to satisfy Program requirements, or Program-required subset of the Table 3-7 analytes.

WASTE CONTAINER - A disposable containment vessel for waste materials including integral liner or shielding materials intended for emplacement at the WIPP (i.e., 55-gallon waste drums or standard waste boxes).

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

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

WASTE TYPE - The classification system describing the physical types of waste [i.e., solidified inorganics (Waste Type I), solid inorganics (Waste Type II), solidified organics (Waste Type III), and solid organics (Waste Type IV) as established by the TRUPACT-II Safety Analysis Report.

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 Albuquerque Operations Office
DOE/WPIO	DOE WIPP Project Integration Office
DOE/WPSO	DOE WIPP Project Site Office
DOT	Department of Transportation
DQO	Data Quality Objective
EB	Equipment Blank
EI	EG&G, Idaho VOC Analytical Laboratory
EM-1	Assistant Secretary of the Office of Environmental Restoration and Waste Management
EM-20	Office of Oversight and Self Assessment
EM-30	Office of Waste Management
EM-34	Office of Waste Management Projects
EM-342	WIPP Project Division
EPA	Environmental Protection Agency
FB	Field Blank
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 Blank
LCS	Laboratory Control Sample

LEL	Lower Explosive Limit
MDL	Method Detection Limit
MMDDYY	Month-Day-Year Format
MS	Mass Spectrometry
NDA	Nondestructive Assay
NEIC	National Enforcement Investigation Center
NFPA	National Fire Protection Association
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
PDP	Performance Demonstration Program
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
RPA	Relative Percent Accuracy
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
TRU	Transuranic
TRUCON	TRUPACT-II Content Codes
TRUPACT-II	Transuranic Package Transporter
VOC	Volatile Organic Compound
VTSR	Validated Time of Sample Receipt
WAC	Waste Acceptance Criteria
WACCC	Waste Acceptance Criteria Certification Committee
WIPP	Waste Isolation Pilot Plant

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