DOE/CAO-95-1076

PERFORMANCE DEMONSTRATION PROGRAM PLAN FOR ANALYSIS OF SIMULATED HEADSPACE GASES

REVISION 0

June 1995

U.S. DEPARTMENT OF ENERGY
CARLSBAD AREA OFFICE

Performance Demonstration Program Plan for Analysis of Simulated Headspace Gases

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Carlsbad Area Office

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ACRONYMS

CAO	Carlsbad Area Office
COC	Chain of custody
CTAG	Critical target analyte gas
CTC	Critical target compound
DOE	Department of Energy
GC/MS	Gas chromatography/mass spectrometry
IDL	Instrument detection limit
MDL	Method detection limit
PDP	Performance Demonstration Program
PRQL	Program Required Quantitation Limit
QA	Quality Assurance
QAO	Quality Assurance Objective
QAPP	Quality Assurance Program Plan
%R	Percent recovery
RPD	Relative percent difference
RSD	Relative standard deviation
SPC	Standard Preparation Contractor
TC	Target compound
TIC	Tentatively identified compound
TRU	Transuranic
VOC	Volatile organic compound
VTSR	Validated time of sample receipt
WIPP	Waste Isolation Pilot Plant

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PERFORMANCE DEMONSTRATION PROGRAM PLAN for ANALYSIS OF SIMULATED HEADSPACE GASES

1.0 INTRODUCTION

1.1 General

The Performance Demonstration Program (PDP) for analysis of headspace gases will consist of regular distribution and analyses of test standards to evaluate the capability for analyzing VOCs, hydrogen, and methane in the headspace of transuranic (TRU) waste throughout the Department of Energy (DOE) complex. Each distribution is termed a PDP cycle. These evaluation cycles will provide an objective measure of the reliability of measurements performed for TRU waste characterization.

Laboratory performance will be demonstrated by the successful analysis of blind audit samples of simulated TRU waste drum headspace gases according to the criteria set within the text of this Program Plan. Blind audit samples (hereinafter referred to as PDP samples) will be used as an independent means to assess laboratory performance regarding compliance with the QAPP QAOs. The concentration of analytes in the PDP samples will encompass the range of concentrations anticipated in actual waste characterization gas samples. Analyses which are required by the WIPP to demonstrate compliance with various regulatory requirements and which are included in the PDP must be performed by laboratories which have demonstrated acceptable performance in the PDP. These analyses are referred to as WIPP analyses and the samples on which they are performed are referred to as WIPP samples for the balance of this document.

1.2 Purpose

The Performance Demonstration Programs (PDPs) are designed to help ensure compliance with the QAOs identified in the TRU Waste Characterization Quality Assurance Program Plan (QAPP) for the Waste Isolation Pilot Plant (WIPP) (DOE,1994). The PDPs are intended for use by the Carlsbad Area Office (CAO) as part of the assessment and approval process for the measurement facilities supplying services for the characterization of WIPP TRU waste. The other two parts of this approval process include the evaluation of method performance data submitted by the measurement facility and the performance of quality assurance audits. The PDP may also be used by the CAO in qualifying facilities that propose to supply additional analytical services required for other than waste characterization, such as support of site operations.

Each PDP is defined in its respective PDP Plan which describes the detailed elements which comprise the program, including the nature of the test materials and the analyses required. The PDP Plan also identifies the criteria that will be used for the evaluation of laboratory performance, the responsibilities of the Program Coordinator, the responsibilities of the Standard Preparation Contractor (SPC), and the responsibilities of the participating laboratories. The CAO will ensure the implementation of this plan by designating a Program Coordinator and by providing technical oversight and coordination for the program. In addition to the PDP described in the present document, two other PDPs are active. These are described in their respective PDP Plans, the *Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program*, (DOE, 1995a) and the *Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes*, (DOE, 1995b).

1.3 Scope and Frequency

The CAO will ensure the implementation of this plan by designating a Program Coordinator and by providing technical oversight and coordination for the program. The PDP Plan identifies the criteria that will be used for the evaluation of laboratory performance, the responsibilities of the Program Coordinator, and the responsibilities of the participating laboratories.

All laboratories supporting sites that intend to ship TRU waste to the WIPP facility by performing headspace gas analysis of these wastes will participate in this PDP. Satisfactory performance of PDP analyses is a necessary but not sufficient condition for certification to ship TRU waste to WIPP.

Acceptable performance must be demonstrated by all participating laboratories prior to the initial analysis of WIPP samples and on a semi-annual basis. Single blind samples will be distributed to participating laboratories every 26 ± 3 weeks. The criteria for acceptable performance are given in Section 6 of this Program Plan. The PDP samples must be analyzed using the methods the laboratory intends to use for the analysis of WIPP samples. These methods must have been developed and approved within the specifications of the QAPP. Additional guidance on acceptable methods is published in the *Transuranic Waste Characterization Sampling and Analysis Methods Manual*, (DOE, 1995c). Only the methods actually used in the PDP will be considered acceptable to support the analysis of WIPP samples. The data generated as a result of the performance demonstration will indicate the appropriateness of the method used as well as the performance of the laboratory.

There are two components to this characterization program; 1) to analyze for volatile organic compounds (VOCs) and 2) to analyze for hydrogen and methane. The analytes in the VOC sample will be selected from the list in Table 1. The VOC gases have also been divided into two groups by importance, critical and non-critical. Critical VOC gases are those compounds which have been identified in documentation and/or studies of TRU waste as:

a) Critical to performance demonstration for the WIPP, or

b) Of special significance with respect to hazardous waste characterization or supporting the ultimate granting of the no-migration variance from the land disposal ban.

Non-critical VOC gases are those which have been identified as potentially present in the WIPP Waste in sufficient quantities to be of quantitative interest but not identified as critical. The performance criteria for the critical gases are more stringent than those for the non-critical gases (see Section 6). Hydrogen and methane gases have been identified in documentation and/or studies of TRU waste as being of concern regarding flammability and providing information regarding gas generation processes occurring in the waste.

The critical VOC gases are called critical target compounds (CTCs) and the hydrogen and methane gases are called critical target analyte gases (CTAGs). The non-critical VOC gases are called target compounds (TCs) and non-target compounds (NTCs). Of the list of 29 VOCs, eight are CTCs and 21 are TCs. Additional NTCs may also be included in the matrix which will be required to be reported as Tentatively Identified Compounds (TICs).

Table 1. VOC Headspace Target Compound List (TCL) and Program Required Quantitation Limits (PRQLs).

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

* Critical Target Compounds (CTCs)

Table 2. Hydrogen/Methane Critical Target Analyte Gases (CTAGs) and Program Required Detection Limits (PRQLs)

Hydrogen/Methane Gases	CAS Number	Hydrogen/Methane PRQL (vol%)
1. Hydrogen (H_2)	1333-74-0	0.1
2. Methane (CH_4)	74-82-8	0.1

2. DEFINITIONS

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

ACTION LIMIT - A numerical criterion which must be met for the analysis of an individual analyte, e.g., blank or background concentration. Failure to meet this criterion may result in a conclusion that the laboratory is unable to quantitate for a specific individual analyte.

ACTION LEVEL - A numerical criterion which must be met for a type of analysis e.g., a fraction of %Recoveries which must fall within the respective QAOs. Failure to meet this criterion may result in a conclusion that the laboratory is unable to adequately perform a specific type of analysis.

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 METHOD - The sample preparation and instrumentation procedures or steps that must be performed to estimate the quantity of analyte in a sample.

AUDIT - A planned and documented investigative evaluation of an item or process to determine the adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, and/or other applicable documents.

BLIND AUDIT SAMPLE - A sample of known composition provided as a single-blind sample to the analytical laboratory. Used by DOE to evaluate analytical laboratory performance. Blind audit samples are distributed to participating laboratories as part of the Performance Demonstration Program.

CHAIN OF CUSTODY (COC) - A set of procedures established to ensure that the integrity of the sample and that of the sample data are maintained.

CORRECTIVE ACTION - Measures taken to rectify conditions adverse to quality or schedule and, where necessary, to preclude repetition.

CRITICAL TARGET ANALYTES GASES (CTAGs) - Those gases not analyzable as VOCs which have been identified by the Program as critical analytes. Critical target analytes gases for the Program are listed in Table 2.

CRITICAL TARGET COMPOUNDS (CTCs) - VOCs which have been identified in documentation and/or studies of TRU waste as critical to performance demonstration for the WIPP or of special significance with respect to hazardous waste characterization or supporting the ultimate granting of the no-migration variance from the land disposal ban. Critical target compounds are identified in Table 1.

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

INSTRUMENT DETECTION LIMIT (IDL) - The minimum signal that an instrument can detect with 99-confidence that the analyte concentration is greater than zero.

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

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

METHOD DETECTION LIMIT (MDL) - The minimum concentration of an analyte that can be measured and reported for a given method with 99-percent 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.

PDP SAMPLE - A blind audit sample prepared specifically for use in the PDP.

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

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 additional information.

PROGRAM COORDINATOR - A CAO-designated organization that administers and coordinates PDP functions, such as PDP sample component preparation, subcontractor oversight, scheduling, scoring, and report summary generation.

PROGRAM REQUIRED QUANTITATION LIMIT (PRQL) - Minimum level of analyte quantitation acceptable. An analyte PRQL should be a minimum of three times the method detection limit (MDL) or instrument detection limit (IDL). PRQLs are presented in Tables 1 and 2.

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

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

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 (%R).

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

STANDARDS PREPARATION CONTRACTOR (SPC) - an independent contractor responsible for the actual preparation and shipping of the blind PDP standards. The SPC is responsible for maintaining the canister inventory, cleaning canisters prior to use for PDP samples, and the traceability of the reference standards used to prepare the PDP samples.

TARGET COMPOUNDS - Those VOCs identified by the program as analytes. Target compounds for the program are listed in Table 1.

TENTATIVELY IDENTIFIED COMPOUNDS (TICS) - Nontarget compounds identified using gas chromatography/mass spectrometry (GC/MS). These reported concentrations will have a higher uncertainty associated with them than the reported target analyte concentrations.

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

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the analytical facility, as recorded on the shipper's delivery receipt and sample traffic report.

VOLATILE ORGANIC COMPOUNDS (VOCs) - For the purposes of the program, those VOCs listed in Table 1 and any additional compounds tentatively identified by the VOC analytical procedures used to satisfy program requirements.

3. PROGRAM COORDINATION

The reviewing and approving authority for the PDP is the CAO. The CAO will use the PDP plan to assess, evaluate, and approve DOE facilities for waste measurement and characterization before the waste is shipped to the WIPP facility. The PDP is only one component of an overall evaluation regime maintained by the CAO.

The CAO will be responsible for ensuring the PDP is conducted on a semiannual basis. A CAO-designated organization shall function as the PDP Coordinator and technical advisor to CAO. For the Headspace Gas PDP, the Program Coordinator will

1. Ensure preparation, control, and distribution of PDP standards.

2. Distribute PDP cycle schedules to measurement facility participants.

3. Confirm the impending initiation of a PDP cycle at least 2 weeks prior to the planned start date.

4. Develop ongoing procedures for PDP sample preparation.

5. Receive, review, and compile the analytical data.

6. Report performance data as specified within this document.

7. Ensure that the records of participation and results of all PDP cycles are maintained in a traceable and retrievable condition.

The Program Coordinator will provide independent technical oversight and coordination of the demonstration program to qualify participating measurement facilities.

The Program Coordinator will maintain a controlled list of the facilities participating in the semi-annual testing program. Measurement facilities required to participate in the PDP will be designated by the CAO. Facilities which are not required to participate but which desire to do so may petition the CAO to be permitted to participate in the PDP. Participation by measurement facilities not actively engaged in characterization of TRU wastes for WIPP related programs will be at the discretion of the CAO.

Each participating facility will be required to provide the Program Coordinator with the name, telephone number, fax number, and address of the contact persons responsible for administrative communications for the PDP. Each participating facility will also be required to provide an address suitable for delivery by freight and express package delivery service of the PDP standards.

4.0 PREPARATION OF PDP SAMPLES

The PDP blind audit samples are prepared in three different concentration classes; low, high, and special and will be prepared according to specifications provided by the Program Coordinator (Section 3.0). A blank for each component is also prepared. Table 3 lists suggested concentration limits for various classes for both VOC and hydrogen/methane PDP samples. The listed concentration maxima are for guidance purposes only. Final analyte concentrations in the PDP samples are left to the discretion of the Program Coordinator. Any given distribution may contain canisters from all classes or any subset of classes for each program component.

VOCs and the Critical Target Analyte Gases, hydrogen and methane, will be combined in the same PDP samples just as they would be in actual headspace gas samples. The concentration classes will not be correlated between the VOCs and the hydrogen/methane. For example, a canister may contain VOCs from the high concentration class and hydrogen/methane in concentrations from the low concentration class and vice versa. Individual laboratories will qualify independently for the VOCs and the hydrogen/methane analyses. Canisters will be available in two sizes, 6-liter canisters for labs wishing to qualify for VOCs alone or both VOCs and hydrogen/methane and 1-liter canisters for labs wishing to qualify for only hydrogen/methane analysis.

The Program Coordinator shall ensure delivery of the PDP samples to each of the laboratories participating in the Inter-laboratory PDP. The Program Coordinator will give two weeks advance notification of the PDP sample shipping date to all participating laboratories. The PDP canisters will be sent to the attention of those individuals who have been identified by the participating site per Section 3.0 as responsible for VOC and/or hydrogen/methane analyses. Changes may be made to the addressees by written notification to the Program Coordinator (with a copy to CAO) at least 48 hours before the scheduled shipping date.

Table 3. Blind Audit Sample Concentration Ranges By Class.

Canister Class	Concentrations of Target Analytes	Notes
Low Concentration VOC	< 20 ppmv	1
High Concentration VOC	< 1000 ppmv	1
Special VOC	< 1000 ppmv	1, 2
Low Concentration Hydrogen/Methane	< 1.5% v/v	
High Concentration Hydrogen/Methane	< 3% v/v	
Special Gases	< 1.5% v/v	2
Blanks	< 50% PRQLs	3

Notes:

1. May contain VOCs not on target list.
2. May contain interferents or targets with known analytical problems.
3. Pure dilution gas or zero air; helium will not be used as the diluent gas.

5.0 ANALYTICAL AND DATA REPORTING REQUIREMENTS

This section describes activities required of the participating laboratories with respect to PDP sample receipt, analysis, and reporting.

5.1 Canister Receipt / COC

5.1.1 Immediately on receipt of the canisters, locate the Delivery/Chain-of-Custody Record. Appendix A contains a sample of the form which will be used.

5.1.2 Verify that the canisters actually received match those listed on the Chain-of-Custody (COC) form both by serial number and physical description. Verify that the canisters have not leaked during shipping by comparing the pressure on receipt to the recorded shipping pressure.

a) If there is a discrepancy, notify the Program Coordinator immediately. Maintain COC control over the canisters and await further instructions.
b) If there are no discrepancies, indicate receipt by signing the Delivery/Chain-of-Custody Record at the appropriate location.

5.1.3 Return the copy of the Delivery/Chain-of-Custody Record to the shipper. Retain the original as the COC record for the canisters. Issue supplemental, site-specific forms if the set of canisters is split, or the COC record cannot be accommodated on the form for other reasons.

5.2 Analysis

5.2.1 Analyze the contents of each canister in quadruplicate using the procedures which have been internally demonstrated and approved and which are planned for use in the WIPP waste characterization test program. All analytical records and documentation generated during the performance of PDP analyses are QA records and must meet the relevant requirements in the QAPP.

5.2.2 Analyses should be completed and reported as soon as possible, but in any case must be forwarded to the Program Coordinator within 28 days after sample receipt.

5.2.3 If a participant's analyses will not be reported by the due date and the participant desires an extension, he or she must notify the Program Coordinator as soon as possible and request that an extension be granted. The Program Coordinator cannot grant an extension; however, the Program Coordinator will request that the CAO grant an extension. The Program Coordinator will notify the participating laboratory of the status of their request. All extensions must be requested and granted before the due date. If an extension has not been granted prior to the due date, the Program Coordinator may make the actual identity and concentrations of the analytes in the PDP samples known at any time thereafter. Any laboratory that had not yet reported will then not be able to use these data to qualify for analysis of WIPP samples.

5.3 Reporting

5.3.1 Each PDP sample shall be analyzed in quadruplicate as an aid in determining precision. A summary of all analytes listed in Tables 1 and 2 that are detected, for all replicate analyses, will be sent by the participating laboratories to the Program Coordinator. The concentrations of detected analytes are to be reported irrespective of the relationships of those concentrations to detection limits quoted or demonstrated for the program. The following specifications apply to the summary report.

5.3.1.1 Reports shall be forwarded directly to the Program Coordinator. Express mail or overnight delivery service is preferred but in any case all analytical reports to the Program Coordinator shall be postmarked or shipped by an overnight delivery service no later than 28 calendar days after validated time of sample receipt (VTSR).

5.3.1.2 Analytical reports shall be submitted for each canister received and for laboratory blanks run in association with the PDP samples.

5.3.1.3 Reports shall consist of at least the following information for each determination:

a) Identification of the reporting laboratory,
b) Identification of the PDP Distribution Cycle and program component for which the data are being reported,
c) Identity of the canister by the serial number from the COC form,
d) Any additional identification assigned to the canister by the laboratory,
e) Identification of the procedure used for the analysis of each analyte,
f) Identification of the replicate number corresponding to the analytical data,
g) Identity and concentration for each target compound or analyte identified,
h) Identity and estimated concentration for each non-target compound or analyte found, and
i) Date and time of analysis.

5.3.1.4 The results of each of the individual analyses must be reported, not the average of the four determinations.

5.3.1.5 The forms given in Appendix B or a reasonable facsimile should be used to report the data to the Program Coordinator. The total number of pages in the report shall be indicated.

5.3.1.6 The report shall include a copy of the COC forms for the canisters as they existed at the time of reporting.

5.3.1.7 Corrections to data will be accepted if received in writing prior to or on the report due date. Data may also be corrected by FAX up to 8:00 PM (Washington, DC local time) on the report due date, if followed by express mail or overnight courier transmission of the original hard copy. Verbal corrections to data will not be accepted.

5.3.1.8 The reports shall be signed by a lab staff member assigned this responsibility. Reports should contain any other information which the laboratory feels is relevant to the data evaluation.

5.3.1.9 The concentrations of all Critical Target Compounds (CTCs) and Critical Target Analyte Gases (CTAGs) which exceed the program required quantitation limits (PRQLs) must be quantified even if multiple dilutions of the gas sample must be analyzed. (See Tables 1 and 2 for PRQLs.) There is no requirement that concentrations of gases in the PDP samples be limited to any specific ratio range. (The Program Coordinator will ensure that the ratios of analytes are not so large as to be likely to cause instrument contamination.)

5.3.1.10 Concentrations must be reported in ppmv for VOC and in % volume for the hydrogen/methane headspace test gases using sample reporting criteria specified in the QAPP.

5.3.2 The requirement to submit only summary data for scoring does not relieve the laboratory from the requirement to maintain appropriate analytical records and documentation. The records generated during the analysis of the PDP samples are QA records. They must be maintained in a

traceable and auditable condition. Storage conditions and duration must meet the requirements of the QAPP and other implementing QA documents and procedures.

5.3.3 In each PDP cycle all canisters should be returned to the Standards Preparation Contractor (SPC) for cleaning, certification and inventory within 2 weeks of notification of laboratory performance. Laboratories which are unable to return the canisters or which wish to retain the canisters for an additional period for experimental purposes must make alternate arrangements with the CAO PDP Liaison. These arrangements could possibly include replacement of canisters at the laboratory's expense in order to insure an adequate inventory of canisters in the system.

6.0 EVALUATION OF PERFORMANCE DATA

Laboratory performance will be evaluated on a point score system. Analytical performance will be evaluated separately for VOCs and hydrogen/methane. The acceptance criteria for the laboratories will be based on the requirements of this Program Plan.

6.1 Analysis of Volatile Organic Compounds (VOCs)

VOC analysis performance will be evaluated in the areas of performance on blanks, accuracy, precision, and correct detection and identification of TICs.

6.1.1 Performance on Blanks

6.1.1.1 Purpose: Analytical results for blanks are used to determine the presence of contamination problems and to quantify those problems if any exist.

6.1.1.2 Criteria: The criterion for blank performance is that none of the target compounds should be present in the blank analyses at levels exceeding 50% of the PRQL.

6.1.1.3 Evaluation Method: Acceptable blank performance is based on the data for all detected compounds and the percent of their concentrations relative to the PRQL for that compound calculated as follows:

$$RBT_A = \frac{CB_A}{PRQL_A} x\ 100$$

where:

RBT_A = amount of compound A calculated in blank as percent of the PRQL;
CB_A = concentration of compound A in blank (ppmv);
$PRQL_A$ = required quantitation limit for compound A (ppmv).

6.1.1.4 Actions: Actions will be taken depending on the blank results. If all of the participating laboratories report a specific analyte to be present in the blank at levels exceeding 50% of the PRQL, the blank will be considered contaminated and the analyte data will be judged unusable and deleted from consideration in the performance criteria for that particular performance demonstration.

6.1.1.4.1 For any compound for which the RBT_A exceeds 50%, the laboratory will be judged to have exceeded an action limit for compound A. Data for that compound will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.1.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.1.2 Accuracy of Quantitation

6.1.2.1 Purpose: Analytical results for blind spikes of known concentration will be used to determine the accuracy with which a laboratory can quantitate the target compounds.

6.1.2.2 Criteria: The results reported for the target compounds should not deviate from the reference values by more than 30%.

6.1.2.3 Evaluation Method: The reported analytical data are used to calculate the relative percent accuracy (RPA) for each of the target compounds as follows:

$$RPA_A = \frac{ACS_A}{TC_A} x\ 100$$

where:

RPA_A = relative percent accuracy expressed as percent recovery of compound A in the PDP sample;

ACS_A = average concentration of compound A from quadruplicate determinations of the PDP sample (ppmv);

TC_A = reference value of compound A in the PDP sample (ppmv).

6.1.2.4 Actions: Actions will be taken depending on the recovery of the target analytes. If all of the reporting laboratories report a specific analyte that falls outside the criteria of 6.1.2.2 in the same direction, then that data will be judged as inappropriate for use in the determination of performance for that round of performance demonstration.

6.1.2.4.1 For any compound for which the RPA_A is outside the range of 70 to 130% recovery (i.e., the measured value differs from the reference value by more than ±30%) in any of the blind spikes, the laboratory will be judged unable to quantitate for compound A. Data for that compound will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.1.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.1.3 Precision of Replicate Determinations

6.1.3.1 Purpose: Analytical results for quadruplicate analyses of blind spikes of known concentration will be used to estimate the precision with which a laboratory can quantitate the target compounds.

6.1.3.2 Criteria: The results reported for the target compounds of quadruplicate determinations from the same canister should not exhibit a relative standard deviation greater than 25%.

6.1.3.3 Evaluation Method: The analytical results for the quadruplicate determinations from each canister are used to calculate the relative percent standard deviation for each of the target compounds as follows:

$$\%RSD_A = \frac{s}{AC_A} x\ 100$$

where:

$\%RSD_A$ = relative standard deviation of the quadruplicate determinations from a single canister (percent);

s = standard deviation of the quadruplicate determinations from a single canister;

AC_A = average concentration of compound A in quadruplicate determinations from a single canister (ppmv).

6.1.3.4 Actions: Actions will be taken depending on the performance results for the precision of replicate determinations.

6.1.3.4.1 For any compound for which the $\%RSD_A$ exceeds 25%, the laboratory will be judged unable to quantitate reproducibly for that compound. Data for that compound will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.1.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.1.4 Precision of Quantitation of Duplicates

6.1.4.1 Purpose: Analytical results for duplicate blind spikes of known concentration will be used to determine the precision with which a laboratory can quantitate the target compounds.

6.1.4.2 Criteria: The difference between the results reported for the target compounds for duplicate determinations from different canisters should not exceed 25% of the average of the duplicate results.

6.1.4.3 Evaluation Method: The analytical results for all reported data are used to calculate the relative percent differences for each of the target compounds as follows:

$$RPD_A = \frac{|ACS_A - ACD_A|}{\left[\frac{ACS_A + ACD_A}{2}\right]} \times 100$$

where:

RPD_A = relative percent difference between the averages of quadruplicate determinations of two duplicate canisters;

ACS_A = average concentration of compound A in quadruplicate determinations from duplicate canister 1 (ppmv);

ACD_A = average concentration of compound A in quadruplicate determinations from duplicate canister 2 (ppmv).

6.1.4.4 Actions: Actions will be taken depending on the magnitude of the RPD between field duplicates.

6.1.4.4.1 For any compound for which the RPD_A exceeds 25%, the laboratory will be judged unable to quantitate reproducibly for that compound. Data for that compound will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.1.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.1.5 Overall Performance

6.1.5.1 Purpose: Individual laboratory performance on the set of PDP samples will be used to assess general problems that may affect the laboratory's ability to analyze for the compounds of interest. This conclusion could result in a holding period during which the laboratory would not analyze WIPP samples until the causes of the problems are identified, corrective action taken, and the efficacy of the corrective action demonstrated.

6.1.5.2 Criteria: The criteria used for the evaluation of laboratory overall performance are specified below. Criteria are applied to the data from a single PDP distribution cycle. Performance will be demonstrated by achieving these criteria:

a) Laboratories must pass 95% of the accumulated performance criteria for critical target compounds (CTC) to be considered as qualified to perform VOC analysis on WIPP samples.

b) Any CTCs for which one or more of the performance criteria are failed (as defined in 6.1.2, 6.1.3, or 6.1.4) must:

   i. have been correctly identified;
   ii. have been quantitated with an RPA between 50 and 150%, and with an RSD and RPD (if applicable) of $\leq$50%.

c) Laboratories must also pass 75% of the accumulated performance criteria for those target compounds (TC) not identified as critical to be considered qualified to perform VOC analysis on WIPP samples.

6.1.5.3 Evaluation Methods: Target compounds have been divided into two groups, CTCs and TCs. Table 4 lists the TCs, and those which have been classed as CTCs are identified. CTCs are those compounds which have been identified in documentation and/or studies of TRU waste as:

a) Critical to performance demonstration for the WIPP, or

b) Of special significance with respect to hazardous waste characterization or supporting ultimate granting of the no-migration variance from the land disposal ban. TCs are those compounds identified as potentially present in the WIPP Experimental Waste in sufficient quantities to be of quantitative interest but not identified as critical.

6.1.5.3.1 The reported analyses of CTCs in the PDP samples will be evaluated on a point scoring system. Results will be scored as follows:

a) For CTCs present in the duplicate canisters, the laboratory will receive five points for each evaluated RPA, RSD, and RPD that meet the criteria of 6.1.2.2, 6.1.3.2, and 6.1.4.2, respectively. (Possible 25 points per compound.)

b) For CTCs present in a single canister, the laboratory will receive five points for each evaluated RPA and RSD that meet the criteria of 6.1.2.2 and 6.1.3.2, respectively. (Possible 10 points per compound.)

6.1.5.3.2 The reported analyses of TCs in the PDP samples will be evaluated on a point scoring system. Results will be scored as follows:

a) For TCs present in the duplicate canisters, the laboratory will receive five points for each evaluated RPA, RSD, and RPD that meet the criteria of 6.1.2.2, 6.1.3.2, and 6.1.4.2, respectively. (Possible 25 points per compound.)

b) For TCs present in a single canister, the laboratory will receive five points for each evaluated RPA and RSD that meet the criteria of 6.1.2.2 and 6.1.3.2, respectively. (Possible 10 points per compound.)

c) For each compound which is known to be present in any canister but which is neither a CTC nor TC, the laboratory will receive five points for correctly identifying the compound as a Non-Target Compound (NTC). (Possible 5 points per compound.)

d) Each laboratory will start with 61 points for each blank canister (5 points for each CTC, and one point for each TC). From this total the laboratory will lose five points for each CTC and one point for each TC for which the laboratory fails to meet the blank criteria of 6.1.1.2.

e) Each laboratory will lose one point for each false positive (i.e., identification of a CTC or TC, at or greater than the PRQL) in a canister in which the compound is known to be absent. This criterion does not apply to the blank canister which is evaluated as in (d), above.

6.1.5.3.3 Example calculation

Laboratory A receives five canisters grouped as follows:

Canister 1 is a blank.

Canisters 2 and 3 are duplicates containing 6 CTCs and 5 TCs at the same concentrations in each canister.

Canister 4 contains 5 CTCs and 7 TCs at different concentrations than canisters 2 and 3 and possibly 1 NTC.

Canister 5 contains 1 CTC and 3 TCs.

The Laboratory can score a maximum of 250 CTC points and 251 TC points, broken down as follows:

Canister 1 = 40 CTC points (8 times 5) and 21 TC points (21 times 1)

Canisters 2 and 3 = 150 CTC points (6 times 25) and 125 TC points (5 times 25)

Canister 4 = 50 CTC points (5 times 10), 70 TC points (7 times 10) and 5 NTC points

Canister 5 = 10 CTC points (1 times 10) and 30 TC points (3 times 10)

Laboratory CTC Score = $100 * (LP_{CTC}/250)$

Laboratory TC Score = 100 * (LP_{TC}/251)

where LP_{CTC} is the total number of CTC points and LP_{TC} is the total number of TC points scored by the laboratory.

6.1.5.4 Special Scoring: On occasion, circumstances may dictate that special canisters be distributed for the evaluation of specific analytical conditions or problems.

6.1.5.4.1 Specific canisters may be distributed to test an individual analyte or a small group of analytes. Such circumstances may include incompatibility between the target analyte(s) and other constituents of the main canister distribution; inability to obtain a pure standard of a target analyte(s); or uncertainties of the certification of a target analyte(s) in the main canister distribution, among others. Under these circumstances, the target analyte(s) will be identified to the laboratories and only the target analyte(s) will be scored. Laboratories will be neither credited nor penalized for analytical data submitted for CTCs and TCs not identified as targets in that canister or for data submitted for NTCs or compounds known to be absent in that canister.

6.1.5.4.2 For some program components canisters may be distributed with the same analytes but at lower concentrations than for other program components. These canisters will be scored as blanks for those laboratories which are not attempting to qualify at PRQLs lower than the concentrations of analytes in these canisters.

6.1.5.5 Canister or Analyte Disqualification: If the preponderance of evidence from the participating laboratories supports a conclusion that the concentration of a specific analyte in a canister has not been certified accurately enough to demonstrate compliance with the criteria of the PDP, the Program Coordinator may judge the data for that analyte to be inappropriate for use in the evaluation of performance for that particular performance demonstration.

6.1.5.6 Actions: The site Project Manager shall have the responsibility of ensuring that appropriate corrective action measures are implemented when a laboratory exceeds an action level. The following are considered minimum mandatory measures that must be implemented when action levels are exceeded.

6.1.5.6.1 If a laboratory obtains a score less than 95% of the total possible CTC points, the laboratory will be judged to have exceeded an action level.

6.1.5.6.2 If a laboratory obtains a score of greater than 95% but less than 100% of the total possible CTC points, the laboratory will be judged to have exceeded an action level unless those analytical results which failed the criteria of 6.1.2.2, 6.1.3.2, or 6.1.4.2 were able to meet criteria of $\geq$50% and $\leq$150% for the RPA and $\pm$50% for the RSD and RPD (as applicable).

6.1.5.6.3 If a laboratory obtains a score less than 75% of the total possible TC points, the laboratory will be judged to have exceeded a control level. For those laboratories that are presently qualified from a previous WIPP Performance Demonstration, the laboratory will be placed on probation. Probationary status will be removed if the laboratory scores greater than 75% on the next PDP sample set. Laboratories that score less than 75% on the initial PDP sample set or score less than 75% on two consecutive sample sets (after initially qualifying) will be judged to have exceeded an action level.

6.1.5.6.4 Any laboratory which has exceeded an action level shall cease analytical operations for the analysis of WIPP samples. The laboratory may not begin analytical operations regarding the analysis of WIPP samples until the laboratory has completed the following actions:

a) Investigated the cause(s) of the failure and taken corrective action, and

b) Generated sufficient data to demonstrate that the same problems will not recur, and

c) Demonstrated adequate performance, i.e., met the scoring criteria described in 6.1.5.2 on another set of PDP samples obtained through CAO and the Program Coordinator.

6.1.5.6.5 CAO may elect to grant conditional approval for a laboratory to perform waste characterization analyses for this program if such conditional approval will not compromise the overall quality of the data being generated for the program. Such a conditional approval may be granted if:

a) the laboratory's failure to meet criteria was limited to a very few compounds (possibly even a single compound);

b) CAO has reason to believe that the error is systematic and likely to be correctable after appropriate corrective actions; and,

c) limitations and conditions can be placed on the approval to guarantee that suspect data will not be used in the program.

6.1.5.6.6 CAO may waive the required demonstration of performance on a new set of PDP samples as a condition of laboratory approval if:

a) the laboratory can prove that the cause of its failure to meet performance criteria was due purely to calculational errors, and

b) the laboratory can demonstrate that appropriate control measures have been initiated to prevent recurrence of the errors.

6.2 Analysis of Hydrogen/Methane Gases

Gas analysis performance will be evaluated in the areas of performance on blanks, accuracy, and precision.

6.2.1 Performance on Blanks

6.2.1.1 Purpose: Analytical results for blanks are used to determine the presence of contamination problems if any exist.

6.2.1.2 Criteria: None of the target analytes should be present in the blank at levels exceeding the method detection limit (MDL).

6.2.1.3 Evaluation Method: The analytical results for all reported blanks are reviewed. Data for all detected analytes will be used to calculate the percent of their concentrations relative to the PRQL for that analyte as follows:

$$RBT_A = \frac{CB_A}{PRQL_A} x\ 100$$

where:

RBT_A = amount of analyte A calculated in the blank as percent of the PRQL;
CB_A = concentration of analyte A in the blank (vol%);
$PRQL_A$ = required quantitation limit for analyte A (vol%).

6.2.1.4 Actions: Actions will be taken depending on the blank results and are discussed below.

6.2.1.4.1 If all of the participating laboratories report a specific analyte to be present in the blank at levels exceeding the method detection limit (MDL), the blank will be considered contaminated and the analyte data will be judged unusable and deleted as part of the performance criteria for that performance demonstration.

6.2.1.4.2 For any analyte for which the RBT_A exceeds 50%, the laboratory will be judged unable to quantitate for analyte A at the required PRQL. Data for that analyte will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.2.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.2.2 Accuracy of Quantitation

6.2.2.1 Purpose: Analytical results for blind spikes of known concentration will be used to determine the accuracy with which a laboratory can quantitate the target analytes.

6.2.2.2 Criteria: The results reported for the target analytes should not deviate from the reference values by more than 30%.

6.2.2.3 Evaluation Method: The analytical results for all reported data are used to calculate the recovery for each of the target analytes as follows:

where:

$$RPA_A = \frac{ACS_A}{TC_A} x\ 100$$

RPA_A = relative percent accuracy expressed as percent recovery of analyte A in the PDP sample;
ACS_A = average concentration of analyte A from quadruplicate analyses of the PDP sample (vol%);
TC_A = reference concentration of analyte A in the PDP sample (vol%).

6.2.2.4 Actions: If all of the reporting laboratories report a specific analyte that falls outside the criteria of 6.2.2.2 in the same direction, then that data will be judged as inappropriate for use in the determination of performance for that round of performance demonstration.

6.2.2.4.1 For analytes which the RPA_A is outside the range of 70 to 130% recovery (differs from the reference by more than ±30%) in the blind spikes, the laboratory will be judged as unable to quantitate for that analyte. Data for these analytes will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.2.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.2.3 Precision of Replicate Determinations

6.2.3.1 Purpose: Analytical results for quadruplicate analyses of blind spikes of known concentration will be used to determine the precision with which a laboratory can quantitate the target analytes.

6.2.3.2 Criteria: The results reported for the target analytes of quadruplicate determinations from the same canister should not exhibit a standard deviation of greater than 25%.

6.2.3.3 Evaluation Method: The analytical results for the quadruplicate determinations from each canister are used to calculate the relative standard deviation for each of the target analytes as follows:

$$\%\ RSD_A = \frac{s}{AC_A} x\ 100$$

where:
$\%RSD_A$ = percent relative standard deviation of the quadruplicate determinations with a single canister;
AC_A = average concentration of analyte A from quadruplicate determinations of a single canister (vol%);
s = standard deviation of the quadruplicate determinations of analyte A from a single canister.

6.2.3.4 Actions Actions will be taken depending on the performance results for the precision of replicate determinations.

6.2.3.4.1 For any sample for which the $\%RSD_A$ exceeds 25% for any analyte, the laboratory will be judged unable to quantitate reproducibly for that analyte. Data for that analyte will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.2.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.2.4 Precision of Quantitation of Duplicates

6.2.4.1 Purpose: Analytical results for duplicate blind spikes of known concentration will be used to determine the precision with which a laboratory can quantitate the analytes.

6.2.4.2 Criteria: The difference between the results reported for target analytes for duplicate determinations from different canisters should not exceed 25% of the average of the duplicate results.

6.2.4.3 Evaluation Method: The analytical results for all reported data are used to calculate the relative percent difference for each of the target analytes as follows:

$$RPD_A = \frac{|ACS_A - ACD_A|}{\frac{(ACS_A + ACD_A)}{2}} x\ 100$$

where:

RPD_A = relative percent difference between the average of quadruplicate determinations of two duplicate canisters;

ACS_A = average concentration of analyte A in quadruplicate determinations from duplicate canister 1 (vol%);

ACD_A = average concentration of analyte A in quadruplicate determinations from duplicate canister 2 (vol%).

6.2.4.4 Actions: For any duplicate set for which the RPD_A exceeds 25% for any analyte, the laboratory will be judged unable to quantitate for that analyte. Data for that analyte will be identified as unacceptable by the Program Coordinator. The impact of exceeding an action limit on overall laboratory performance is given in Section 6.2.5. In accordance with Section 6.0, the site Project Manager shall have responsibility to ensure that appropriate corrective action measures are taken when necessary.

6.2.5 Overall Performance

6.2.5.1 Purpose: Laboratory performance on the entire set of PDP samples will be used to assess general problems that may affect the laboratory's ability to analyze for the analytes of interest. This conclusion could result in a holding period during which the laboratory would not analyze WIPP samples until the causes of the problems are identified, corrective action taken, and the efficacy of the corrective action demonstrated.

6.2.5.2 Criteria: The criteria used for the evaluation of overall laboratory performance shall be demonstrated by passing all (100%) of the performance criteria for each critical target analyte gas (CTAG) to be considered qualified to perform gas analysis on WIPP samples.

6.2.5.3 Evaluation Methods: CTAGs are those analytes which have been identified in documentation and/or studies of TRU waste as being of concern regarding flammability and provide information regarding gas generation processes occurring in the waste.

6.2.5.3.1 The results reported for the analysis of CTAGs for the PDP samples must meet all of the criteria identified in sections 6.2.1.2, 6.2.2.2, 6.2.3.2, and 6.2.4.2 of this Program Plan.

6.2.5.3.2 The reported analyses of CTAGs in the PDP samples will be evaluated on a point scoring system. Results will be scored as follows:

a) For target analytes present in the duplicate canisters, the laboratory will receive five points for each evaluated RPA, RSD, and RPD that meet the criteria of 6.2.2.2, 6.2.3.2, and 6.2.4.2, respectively. (Possible 25 points per analyte)

b) For target analytes present in a single canister, the laboratory will receive five points for each evaluated RPA and RSD that meet the criteria of 6.2.2.2 and 6.2.3.2, respectively. (Possible 10 points per analyte)

c) Each laboratory will start with 10 points for each blank canister (5 points for each CTAG). From this total the laboratory will lose five points for each CTAG which fails to meet the blank criteria of 6.2.1.2.

d) Each laboratory will lose five points for each false positive (i.e., identification of a target analyte in a canister in which the analyte is known to be absent). This criterion does not apply to the blank canister which is evaluated as in (c), above.

6.2.5.3.3 The calculation of the PDP sample analysis score will be by the following equation:

$$\text{Laboratory CTAG Score} = 100 * (LP_{CTAG}/TP_{CTAG})$$

where:

LP_{CTAG} is the total number of CTAG points scored by the laboratory, and TP is the total points possible for the PDP sample set.

6.2.5.4 Special Scoring: On occasion, circumstances may dictate that special canisters be distributed for the evaluation of specific analytical conditions or problems.

6.2.5.4.1 Specific canisters may be distributed to test an individual analyte or a small group of analytes. Such circumstances may include incompatibility between the target analyte gas(es) and other constituents of the main canister distribution; inability to obtain a pure standard of a target analyte gas(es); or uncertainties of the certification of a target analyte gas(es) in the main canister distribution, among others. Under these circumstances, the target analyte gas(es) will be identified and only the target analyte gas(es) will be scored. Laboratories will be neither credited nor penalized for analytical data submitted for CTAGs not identified as targets in that canister or for data submitted for target analyte gases known to be absent in that canister.

6.2.5.4.2 For some program components canisters may be distributed with the same analytes but at lower concentrations than for other program components. These canisters will be scored as blanks for those laboratories which are not attempting to qualify at PRQLs lower than the concentrations of analytes in these canisters.

6.2.5.5 Canister or Analyte Disqualification: If the preponderance of evidence from the participating laboratories supports a conclusion that the concentration of a specific analyte in a

canister has not been certified accurately enough to demonstrate compliance with the criteria of the PDP, the Program Coordinator may judge the data for that analyte to be inappropriate for use in the evaluation of performance for that particular performance demonstration.

6.2.5.6 Actions: The site Project Manager shall have the responsibility of ensuring that appropriate corrective action measures are implemented when a laboratory exceeds an action level. The following are considered minimum mandatory measures that must be implemented when action levels are exceeded.

6.2.5.6.1 If a laboratory fails to meet all the criteria of 6.2.2.2, 6.2.3.2, or 6.2.4.2 for the CTAGs, the laboratory will be judged to have exceeded an action level.

6.2.5.6.2 Any laboratory which has exceeded an action level shall cease analytical operations for WIPP samples. The laboratory may not begin analytical operations for WIPP samples until the laboratory has completed the following actions:

a) Investigated the cause of the failure(s) and taken corrective action, and

b) generated sufficient data to demonstrate that the same problems will not recur, and

c) demonstrated adequate performance, i.e., met the scoring criteria described in 6.2.5.2 on another set of PDP samples obtained through CAO and the Program Coordinator.

6.2.5.6.3 CAO may elect to grant conditional approval for a laboratory to perform waste characterization analyses for this program if such conditional approval will not compromise the overall quality of the data being generated for the program. Such a conditional approval may be granted if:

a) CAO has reason to believe that the error is systematic and likely to be correctable after appropriate corrective actions; and,

b) limitations and conditions can be placed on the approval to guarantee that suspect data will not be used in the program.

6.2.5.6.4 CAO may waive the required demonstration of performance on a new set of PDP samples as a condition of laboratory approval if:

a) the laboratory can prove that the cause of it's failure to meet performance criteria was due purely to calculational errors, and

b) the laboratory can demonstrate that appropriate control measures have been initiated to prevent reoccurrence of the errors.

7.0 REPORTING OF PERFORMANCE DATA

7.1 Summary of Data

The Program Coordinator shall review and evaluate the results, compile them into a master summary, and deliver this summary to the CAO within three weeks post-receipt of the last laboratory data set or within nine weeks of the last VTSR, whichever occurs first. The report due date will be extended by a time equivalent to any extension granted by CAO under section 5.2.3. The report summary shall include the values reported by the laboratories, the reference concentration values, the acceptance ranges per analyte, and the pass/fail status of each individual laboratory.

7.2 Distribution of Data

The CAO, in conjunction with the Program Coordinator, will evaluate individual laboratory performance and approve individual laboratories for participation in the WIPP waste characterization program. Depending on the results of the PDP, the generator site Project Manager(s) shall have the responsibility of ensuring that appropriate corrective action measures are taken. The semiannual QA reports (TRU Waste Characterization Quality Assurance Program Plan, CAO-94-1080, Section 2.2) must assess the impact of corrective action measures taken.

Copies of the summary report shall be distributed to each of the DOE Operations Offices involved, to each of the participating laboratories, and to such other individuals and organizations as the CAO shall deem appropriate. The identification of individual laboratories shall be coded in copies of the master summary distributed by the CAO. The CAO shall also provide written notification to the DOE operations offices regarding the adequacy and approval status of their participating laboratories.

7.3 Backup PDP Samples

A backup set of blind audit canisters can be prepared by the Program Coordinator approximately four weeks after laboratories are notified of their status. Laboratories that do not pass on the initial set of blind audit canisters may request to have these canisters prepared for their facility. Requests must be submitted in writing to CAO and be accompanied by a report stating the reasons for the failures and any corresponding corrective actions which were taken. The schedule of distribution, analysis, scoring, and approval/disapproval actions by CAO will be negotiated for each supplemental distribution. The schedule will be based on discussions with the potential participants and a review of impacts on the overall WIPP schedule. Timing of and selection of laboratories for participation in supplemental distributions will be entirely at the discretion of CAO. Primary consideration will be given to preventing adverse impacts on WIPP waste characterization and compliance schedules.

7.4 Laboratory Status

Once the CAO has made a determination of laboratory status with respect to analyses which are required by the WIPP to demonstrate compliance with regulatory requirements, such status shall remain in effect until a new determination is made by the CAO. Laboratories obtaining approved status through a supplemental distribution cycle must participate in the next regular distribution cycle to maintain their approved status.

8.0 REFERENCES

DOE. 1994. *TRU Waste Characterization Quality Assurance Program Plan.* DOE/CAO 94-1010, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE. 1995a. *Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program.* DOE/CAO 95-1045, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE. 1995b. *Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes.* DOE/CAO 95-1077, Revision 0, June 1995. Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE. 1995c. *Transuranic Waste Characterization Sampling and Analysis Methods Manual.* DOE/WIPP-91-043, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

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APPENDIX A

Sample Chain of Custody Form

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WIPP EXPERIMENTAL WASTE CHARACTERIZATION PROGRAM
LABORATORY PERFORMANCE DEMONSTRATION
Delivery/Chain-of-Custody Record

Program Segment:	**Headspace Gas Analysis**
Sample Type:	**Single Blind, Standard Distribution**
Distribution Month/Year:	

Canister ID	Canister Volume & Units	Shipping Pressure & Units	Scheduled Analysis		Comments:
			VOC	Gas	

All entries of names in the sections below should be signatures!

Shipped By:	Date/Time	Received By:	Date/Time

After completion to this point, return attached copy to Shipper!

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

Final Disposition By:		Disposition:

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APPENDIX B

Sample Data Reporting Forms

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PERFORMANCE DEMONSTRATION PROGRAM REPORT FORM
HEADSPACE GAS ANALYSIS - VOLATILES

Laboratory Name :	Report Page _____ of _____ Pages
PDP Distribution (Mo/Yr) :	Laboratory Sample ID :
Canister No. :	Replicate Number : _____ of _____

Analyte	Result (ppmv)	Flag	Method Identification	Analysis		Comment
				Date	Time	
Acetone						
Benzene						
Bromoform						
n-Butanol						
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						
Methyl ethyl ketone						
Methyl isobutyl ketone						
1,1,2,2-Tetrachloroethane						
Tetrachloroethene						
Toluene						
1,1,1-Trichloroethane						
Trichloroethene						

Analyte	Result (ppmv)	Flag	Method Identification	Analysis		Comment
				Date	Time	
1,1,2-Trichloro-1,2,2-trifluoroethane						
1,3,5-Trimethylbenzene						
1,2,4-Trimethylbenzene						
m-Xylene						
o-Xylene						
p-Xylene						

ADDITIONAL COMMENTS: _____

_____

_____

_____

_____

APPROVAL: _____ _____ _____

SIGNATURE TITLE DATE

PERFORMANCE DEMONSTRATION PROGRAM REPORT FORM
HEADSPACE GAS ANALYSIS - GASES

Laboratory Name :	Report Page _____ of _____ Pages
PDP Distribution (Mo/Yr) :	Laboratory Sample ID :
Canister No. :	

Analyte	Result (% Vol.)	Flag	Method Identification	Analysis		Comment
				Date	Time	
Replicate Number: _____ of 4						
Hydrogen (H_2)						
Methane (CH_4)						
Replicate Number: _____ of 4						
Hydrogen (H_2)						
Methane (CH_4)						
Replicate Number: _____ of 4						
Hydrogen (H_2)						
Methane (CH_4)						
Replicate Number: _____ of 4						
Hydrogen (H_2)						
Methane (CH_4)						

ADDITIONAL COMMENTS: _____

_____

_____

APPROVAL: _____ _____ _____

SIGNATURE TITLE DATE