

ATTACHMENT C3

**QUALITY ASSURANCE OBJECTIVES AND DATA VALIDATION
TECHNIQUES FOR WASTE CHARACTERIZATION SAMPLING AND
ANALYTICAL METHODS**

Waste Isolation Pilot Plant
Hazardous Waste Permit
January 31, 2012

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2 **QUALITY ASSURANCE OBJECTIVES AND DATA VALIDATION**
3 **TECHNIQUES FOR WASTE CHARACTERIZATION SAMPLING AND**
4 **ANALYTICAL METHODS**

5 C3-1 Validation Methods

6 The Permittees shall require the generator/storage sites (**sites**) to perform validation of all data
7 (qualitative as well as quantitative) so that data used for Waste Isolation Pilot Plant (**WIPP**)
8 compliance programs will be of known and acceptable quality. Validation includes a quantitative
9 determination of precision, accuracy, completeness, and method detection limits (as
10 appropriate) for analytical data (headspace Volatile Organic Compounds (**VOC**), total VOCs,
11 Semivolatile Organic Compounds (**SVOC**), and metals data). Quantitative data validations shall
12 be performed according to the conventional methods outlined below (equations C3-1 through
13 C3-8). These quantitative determinations will be compared to the Quality Assurance Objectives
14 (**QAOs**) specified in Sections C3-2 through C3-9. A qualitative determination of comparability
15 and representativeness will also be performed.

16 The qualitative data or descriptive information generated by radiography and visual examination
17 is not amenable to statistical data quality analysis. However, radiography and visual
18 examination are complementary techniques yielding similar data for determining the waste
19 matrix code. The waste matrix code is determined to ensure that the container is properly
20 included in the appropriate waste stream.

21 Data validation will be used to assess the quality of waste characterization data collected based
22 upon project precision, accuracy, completeness, comparability, and representativeness
23 objectives. These objectives are described below:

24 Precision

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

30
$$RPD = \frac{C_1 - C_2}{\frac{(C_1 + C_2)}{2}} \times 100 \quad (C3-1)$$

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

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

$$3 \quad \%RSD = \frac{s}{y_{mean}} \times 100 \quad (C3-2)$$

4 where s is the standard deviation and y_{mean} is the mean of the replicate sample analyses.

5 The standard deviation, s , is calculated as follows:

$$6 \quad s = \sqrt{\frac{\sum_{i=1}^n (y_i - y_{mean})^2}{n - 1}} \quad (C3-3)$$

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

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

$$12 \quad \%D = \frac{|C_1 - C_2|}{C_1} \times 100 \quad (C3-4)$$

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

14 Accuracy

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

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

$$19 \quad \%R = \frac{C_m}{C_{srm}} \times 100 \quad (C3-5)$$

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

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

$$23 \quad \%R = \frac{S - U}{C_{sc}} \times 100 \quad (C3-6)$$

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

3 Method Detection Limit

4 The method detection limit (**MDL**) is the minimum concentration of an analyte that can be
5 measured and reported with 99 percent confidence that the analyte concentration is greater
6 than zero. The MDL for all quantitative measurements (except for those using Fourier Transform
7 Infrared Spectroscopy [**FTIRS**]) is defined as follows:

$$8 \quad MDL = t_{(n-1, 1-\alpha=.99)} \times s \quad (C3-7)$$

9 where $t_{(n-1, 1-\alpha=.99)}$ is the t-distribution value corresponding to a 99 percent confidence level with n-
10 1 degrees of freedom, n is the number of observations, and s is the standard deviation of
11 replicate measurements.

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

$$13 \quad MDL = 3s \quad (C3-8)$$

14 where s is the standard deviation. Initially, a minimum of seven samples spiked at a level of
15 three to five times the estimated MDL and analyzed on non-consecutive days must be used to
16 establish the MDLs. MDLs should be updated using the results of the laboratory control sample
17 or on-line control samples.

18 Completeness

19 Completeness is a measure of the amount of valid data obtained from the overall measurement
20 system compared to the amount of data collected and submitted for analysis. Completeness
21 must be expressed as the number of samples analyzed with valid results as a percent of the
22 total number of samples submitted for analysis. Completeness, expressed as the percent
23 complete (**%C**), is calculated as follows:

$$24 \quad \%C = \frac{V}{n} \times 100 \quad (C3-9)$$

25 where V is the number of valid sampling or analytical results obtained and n is the number of
26 samples submitted for analysis.

27 Comparability

28 Comparability is the degree to which one data set can be compared to another. Comparability of
29 data generated at different sites will be ensured through the use of standardized, approved
30 testing, sampling, preservation, and analytical techniques and by meeting the QAOs specified in
31 Sections C3-2 through C3-9.

32 The comparability of waste characterization data shall be ensured through the use of
33 generator/storage site data usability criteria. The Permittees shall ensure that data usability

1 criteria are consistently established and used by the generator/storage sites to assess the
2 usability of analytical and testing data. The criteria shall address, as appropriate, the following:

3 Definition or reference of criteria used to define and assign data qualifier flags based on
4 Quality Assurance Objective results,

5 Criteria for assessing the usability of data impacted by matrix interferences,

6 Criteria for assessing the usability of data based upon positive and negative bias as
7 indicated by quality control data, of data qualifiers, and qualifier flags,

8 Criteria for assessing the usability of data due to

9 Severe matrix effects,
10 Misidentification of compounds,
11 Gross exceedance of holding times,
12 Failure to meet calibration or tune criteria

13 Criteria for assessing the usability of data that does not meet minimum detection limit
14 requirements.

15 The Permittees shall be responsible for evaluating generator/storage site data usability and the
16 U.S. Department of Energy (**DOE**) shall assess implementation through the generator/storage
17 site audit.

18 Representativeness

19 Representativeness is the degree to which sample data represent a characteristic of a
20 population, parameter variations at a sampling point, or an environmental condition.
21 Representativeness is a qualitative parameter that concerns the proper design of the sampling
22 program.

23 Representativeness of waste containers from waste streams subjected to headspace gas,
24 homogeneous solids, and soil/gravel sampling and analysis will be validated, through
25 documentation, that a true random sample with an adequate population was identified and
26 collected consistent with Permit Attachment C2, Section C2-1. Since representativeness is a
27 quality characteristic that expresses the degree to which a sample or group of samples
28 represents the population being studied, the random selection of waste containers ensures
29 representativeness on a Program level. The Permittees shall require the Site Project Manager
30 to document that the selected waste containers from within a waste stream were randomly
31 selected. Sampling personnel shall verify that proper procedures are followed to ensure that
32 samples are representative of the waste contained in a particular waste container or a waste
33 stream.

34 Identification of Tentatively Identified Compounds

35 In accordance with SW-846 convention, identification of compounds detected by gas
36 chromatography/mass spectrometry methods that are not on the list of target analytes shall be
37 reported. Both composited and individual container headspace gas, volatile analysis
38 (TCLP/Totals), and semi-volatile (TCLP/Totals) shall be subject to tentatively identified

1 compound (**TIC**) reporting. These TICs for GC/MS Methods are identified in accordance with the
2 following SW-846 criteria:

3 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the
4 most abundant ion) should be present in the sample spectrum.

5 The relative intensities of the major ions should agree within ± 20 percent.

6 Molecular ions present in the reference spectrum should be present in the sample
7 spectrum.

8 Ions present in the sample spectrum but not in the reference spectrum should be reviewed
9 for possible background contamination or presence of coeluting compounds.

10 Ions present in the reference spectrum but not in the sample spectrum should be reviewed
11 for possible subtraction from the sample spectrum because of background
12 contamination or coeluting peaks.

13 The reference spectra used for identifying TICs shall include, at minimum, all of the
14 available spectra for compounds that appear in the 20.4.1.200 NMAC (incorporating
15 40 CFR Part 261) Appendix VIII list. The reference spectra may be limited to VOCs
16 when analyzing headspace gas samples.

17 TICs for headspace gas analyses that are performed through FTIRS analyses shall be
18 identified in accordance with the specifications of SW-846 Method 8410.

19 TICs shall be reported as part of the analytical batch data reports for GC/MS Methods in
20 accordance with the following minimum criteria:

21 a TIC in an individual container headspace gas or solids sample shall be reported in the
22 analytical batch data report if the TIC meets the SW-846 identification criteria listed
23 above and is present with a minimum of 10% of the area of the nearest internal
24 standard.

25 a TIC in a composited headspace gas sample that contains 2 to 5 individual container
26 samples shall be reported in the analytical batch data report if the TIC meets the SW-
27 846 identification criteria listed above and is present with a minimum of 2% of the area
28 of the nearest internal standard.

29 a TIC in a composited headspace gas sample that contains 6 to 10 individual container
30 samples shall be reported in the analytical batch data report if the TIC meets the SW-
31 846 identification criteria listed above and is present with a minimum of 1% of the area
32 of the nearest internal standard.

33 a TIC in a composited headspace gas sample that contains 11 to 20 individual container
34 samples shall be reported in the analytical batch data report if the TIC meets the SW-
35 846 identification criteria listed above and is present with a minimum of 0.5% of the
36 area of the nearest internal standard.

1 TICs that meet the SW-846 identification criteria, are reported in 25 percent of all waste
2 containers sampled from a given waste stream, and that appear in the 20.4.1.200 NMAC
3 (incorporating 40 CFR §261) Appendix VIII list, will be compared to acceptable knowledge data
4 to determine if the TIC is a listed waste in the waste stream. TICs identified through headspace
5 gas analyses that meet the Appendix VIII list criteria and the 25 percent reporting criteria for a
6 waste stream will be added to the headspace gas waste stream target list regardless of the
7 hazardous waste listing associated with the waste stream. TICs reported from the Totals VOC
8 or SVOC analyses may be excluded from the target analyte list for a waste stream if the TIC is a
9 constituent in an F-listed waste whose presence is attributable to waste packaging materials or
10 radiolytic degradation from acceptable knowledge documentation. If a listed waste constituent
11 TIC cannot be attributed to waste packaging materials, radiolysis, or other origins, the
12 constituent will be added to the target analyte list and new hazardous waste numbers will be
13 assigned, if appropriate. TICs subject to inclusion on the target analyte list that are toxicity
14 characteristic parameters shall be added to the target analyte list regardless of origin because
15 the hazardous waste designation for these numbers is not based on source. However, for
16 toxicity characteristic and non-toxic F003 constituents, the site may take concentration into
17 account when assessing whether to add a hazardous waste number. If a target analyte list for a
18 waste stream is expanded due to the presence of TICs, all subsequent samples collected from
19 that waste stream will be analyzed for constituents on the expanded list.

20 C3-2 Headspace-Gas Sampling

21 Quality Assurance Objectives

22 The precision and accuracy of the container headspace-gas sampling operations must be
23 assessed by analyzing field QC headspace-gas samples. These samples must include
24 equipment blanks, field reference standards, field blanks, and field duplicates. If the QAOs
25 described below are not met, a nonconformance report must be prepared, submitted, and
26 resolved (Section C3-13).

27 Precision

28 The precision of the headspace-gas sampling and analysis operation must be assessed by
29 sequential collection of field duplicates for manifold sampling operations or simultaneous
30 collection of field duplicates for direct canister sampling operations for VOCs determination.
31 Corrective actions must be taken if the RPD exceeds 25 percent for any analyte found greater
32 than the PRQL in both of the duplicate samples.

33 Accuracy

34 A field reference standard must be collected using headspace-gas sampling equipment to
35 assess the accuracy of the headspace-gas sampling operation at a frequency of one field
36 reference standard for every 20 containers sampled or per sampling batch. Corrective action
37 must be taken if the %R of the field-reference standard is less than 70 or greater than 130.

38 Field blanks must also be collected at a frequency of 1 field blank for every 20 containers or
39 sampling batch sampled to assess possible contamination in the headspace gas sampling
40 method. Equipment blanks must also be collected at a frequency of 1 equipment blank for each
41 equipment cleaning batch to assess possible contamination in the equipment cleaning method.

1 Corrective actions must be taken if the blank exceeds three times the MDLs listed for any of the
2 compounds listed in Table C3-2.

3 Completeness

4 Sampling completeness shall be expressed as the number of valid samples collected as a
5 percent of the total number of samples collected for each waste stream. A valid sample is
6 defined as a sample collected in accordance with approved sampling methods and the
7 container was properly prepared for sampling (e.g., the polyliner was vented to the container
8 headspace). The Permittees shall require participating sampling facilities to achieve a minimum
9 90 percent completeness. The amount and type of data that may be lost during the headspace-
10 gas sampling operation cannot be predicted in advance. The Permittees shall require the Site
11 Project Manager to evaluate the importance of any lost or contaminated headspace-gas
12 samples and take corrective action as appropriate.

13 Comparability

14 Consistent use and application of uniform procedures and equipment, as specified in Permit
15 Attachment C1 and application of data usability criteria, should ensure that headspace gas
16 sampling operations are comparable when sampling headspace at the different sampling
17 facilities. The Permittees shall require each site to take corrective actions if uniform procedures,
18 equipment, or operations are not followed without approved and justified deviations. In addition,
19 laboratories analyzing samples must successfully participate in the Performance Demonstration
20 Program (**PDP**) (DOE, 2003).

21 Representativeness

22 Specific headspace-gas sampling steps to ensure samples are representative include:

23 Selection of the correct Drum Age Criteria (**DAC**) Scenario and waste packaging
24 configuration and meeting DAC equilibrium times.

25 A sample canister cleaning and leak check after assembly

26 Sampling equipment cleaning or disposal after use

27 Sampling equipment leak check after sample collection

28 Use of sample canisters with passivated internal surfaces

29 Use of low-internal-volume sampling equipment

30 Collection of samples with a low-sample volume to available headspace volume ratio (less
31 than 10 percent of the headspace when the headspace can be determined)

32 Careful and documented pressure regulation of all activities specified in Attachment C1,
33 Section C1-1

34 Performance audits

1 Collection of equipment blanks, field reference standard, field blanks, and field duplicates
2 at the specified frequencies.

3 Manifold pressure sensors and temperature sensors calibrated before initial use and
4 annually using NIST, or equivalent standards.

5 OVA calibrated daily, prior to first use, or as necessary according to manufacturer's
6 specifications.

7 Failure to perform the checks at the prescribed frequencies would result in corrective actions.

8 C3-3 Sampling of Homogeneous Solids and Soils/Gravel

9 Quality Assurance Objectives

10 To ensure that sampling is conducted in a representative manner on a waste-stream basis for
11 waste containers containing homogeneous solids and soil/gravel, samples must be collected
12 randomly in both the horizontal and vertical planes of each container's waste. For waste
13 containers that contain homogeneous solids and soil/gravel in smaller containers (e.g., 1 gal
14 [4.0 L] poly bottles) within the waste container, one randomly chosen smaller container must be
15 sampled from each container.

16 Precision

17 Sampling precision must be determined by collecting and sampling field duplicates (e.g., co-
18 located cores or co-located samples as described in Permit Attachment C1-2b(1)) once per
19 sampling batch or once per week during sampling operations, whichever is more frequent. A
20 sampling batch is a suite of homogeneous solids and soil/gravel samples collected
21 consecutively using the same sampling equipment within a specific time period. A sampling
22 batch can be up to 20 samples (excluding field QC samples), all of which must be collected
23 within 14 days of the first sample in the batch. The Permittees shall require the Site Project
24 Manager to calculate and report the RPD between co-located core/samples.

25 The recommended method for establishing acceptance criteria for co-located cores and co-
26 located samples is the F-test method because the F-Test: 1) does not require potentially
27 arbitrary groupings into batches, 2) is based on exact distributions, and 3) is more likely to
28 detect a change in the process. When a sufficient number of samples are collected (25 to 30
29 pairs of co-located cores or samples), control charts of the RPD will be developed for each
30 constituent and for each waste matrix or waste type (e.g., pyrochemical salts or organic
31 sludges). The limits for the control chart will be three standard deviations above or below the
32 average RPD. Once constructed, RPDs for additional co-located pairs will be compared with the
33 control chart to determine whether or not the co-located cores are acceptable. Periodically, the
34 control charts will be updated using all available data.

35 The statistical test will involve calculating the variance for co-located cores and samples by
36 pooling the variances computed for each pair of duplicate results. The variance for the waste
37 stream will be computed excluding any data from containers with co-located cores, because the
38 test requires the variance estimates to be independent. All data must be transformed to
39 normality prior to computing variances and performing the test. The test hypothesis is evaluated
40 using the F distribution and the method for testing the difference in variances.

1 Accuracy

2 Sampling accuracy through the use of standard reference materials shall not be measured.
3 Because waste containers containing homogeneous solids and soil/gravel with known quantities
4 of analytes are not available, sampling accuracy cannot be determined. However, sampling
5 methods and requirements described are designed to minimize sample degradation and hence
6 maximize sampling accuracy.

7 Sampling accuracy as a function of sampling cross-contamination will be measured. Equipment
8 blanks will be collected at a frequency of once per equipment cleaning batch. Corrective actions
9 must be taken if the blank exceeds three times the MDLs (PRDLs for metals) listed for any of
10 the compounds or analytes listed in Tables C3-4, C3-6, and C3-8. Equipment blanks will be
11 collected from the following equipment types:

- 12 Fully assembled coring tools
- 13 Liners cleaned separately from coring tools
- 14 Miscellaneous sampling equipment that is reused (bowls, spoons, chisels)

15 Completeness

16 Sampling completeness shall be expressed as the number of valid samples collected as a
17 percent of the total number of samples collected for each waste stream. A valid sample is any
18 sample that is collected from a randomly selected container using randomly selected horizontal
19 and vertical planes in accordance with approved sampling methods. The Permittees shall
20 require participating sampling facilities to achieve a minimum 90 percent completeness.

21 Comparability

22 Consistent use and application of uniform procedures, sampling equipment, and measurement
23 units must ensure that sampling operations are comparable. Consistent application of data
24 usability criteria will also ensure comparability. In addition, the Permittees shall require
25 laboratories analyzing samples to successfully participate in the PDP (DOE, 2005).

26 Representativeness

27 Specific steps to ensure the representativeness of samples include the following for both waste
28 containers and smaller containers:

29 Coring tools and sampling equipment must be clean prior to sampling.

30 The entire depth of the waste minus a site defined approved safety factor must be cored,
31 and the core collected must have a length greater than or equal to 50 percent of the
32 depth of the waste. This is called the core recovery and is calculated as follows:

33
$$\text{Core recovery (percent)} = \frac{y}{x} \times 100 \quad (\text{C3-10})$$

34 where

35 x = the depth of the waste in the container

1 y = the length of the core collected from the waste.

2 Coring operations and tool selection should be designed to minimize alteration of the in-
3 place waste characteristics. Minimal waste disturbance must be verified by visually
4 examining the core and describing the observation (e.g., undisturbed, cracked, or
5 pulverized) in the field logbook.

6 If core recovery is less than 50 percent of the depth of the waste, a second coring
7 location shall be randomly selected. The core with the best core recovery shall be
8 used for sample collection.

9 One randomly selected container within a container will be chosen if the container contains
10 individual waste containers.

11 C3-4 Non Destructive Examination Methods

12 Quality Assurance Objectives

13 The QAOs for non destructive examination (**NDE**) are detailed in this section. NDE can be either
14 radiography or visual examination (**VE**). If the QAOs described below are not met, then
15 corrective action shall be taken. It should be noted that NDE does not have a specific MDL
16 because it is primarily a qualitative determination. The objective of NDE for the program is to
17 determine the physical waste form, the absence of prohibited items, and additional waste
18 characterization techniques that may be used based on the Summary Category Groups (i.e.,
19 S3000, S4000, S5000). The Permittees shall require each site to describe all activities required
20 to achieve these objectives in the site quality assurance project plan (**QAPjP**) and standard
21 operating procedures (**SOP**).

22 C3-4a Radiography

23 Data to meet these objectives must be obtained from a video and audio recorded scan provided
24 by trained radiography operators at the sites. Results must also be recorded on a radiography
25 data form. The precision, accuracy, completeness, and comparability objectives for radiography
26 data are presented below.

27 Precision

28 Precision is maintained by reconciling any discrepancies between two radiography operators
29 with regard to identification of the waste matrix code, liquids in excess of TSDF-WAC limits, and
30 compressed gases through independent replicate scans and independent observations.
31 Additionally, the precision of radiography is verified prior to use by tuning precisely enough to
32 demonstrate compliance with QAOs through viewing an image test pattern.

33 Accuracy

34 Accuracy is obtained by using a target to tune the image for maximum sharpness and by
35 requiring operators to successfully identify 100 percent of the items required to meet the DQOs
36 for radiography specified in Permit Attachment C, Section C-4a(1) in a training container during
37 their initial qualification and subsequent requalification.

1 Completeness

2 A video and audio media recording of the radiography examination and a validated radiography
3 data form will be obtained for 100 percent of the waste containers subject to radiography. All
4 video and audio media recordings and radiography data forms will be subject to validation as
5 indicated in Section C3-10.

6 Comparability

7 The comparability of radiography data from different operators shall be enhanced by using
8 standardized radiography procedures and operator qualifications.

9 C3-4b Visual Examination

10 Results must be recorded on a VE data form. The precision, accuracy, completeness, and
11 comparability objectives for VE data are presented below.

12 Precision

13 Precision is maintained by reconciling any discrepancies between the operator and the
14 independent technical reviewer with regard to identification of waste matrix code, liquids in
15 excess of TSDF-WAC limits, and compressed gases.

16 Accuracy

17 Accuracy is maintained by requiring operators to pass a comprehensive examination and
18 demonstrate satisfactory performance in the presence of the VE expert during their initial
19 qualification. VE operators shall be requalified every two years.

20 Completeness

21 A validated VE data form will be obtained for 100 percent of the waste containers subject to VE.

22 Comparability

23 The comparability of VE data from different operators shall be enhanced by using standardized
24 VE procedures and operator qualifications.

25 C3-5 Gas Volatile Organic Compound Analysis

26 Quality Assurance Objectives

27 The development of data quality objective (**DQOs**) specifically for this program has resulted in
28 the QAOs listed in Table C3-2. The specified QAOs represent the required quality of data
29 necessary to draw valid conclusions regarding program objectives. WAP-required limits, such
30 as the program required quantitation limits (**PRQL**) associated with VOC analysis, are specified
31 to ensure that the analytical data collected satisfy the requirements of all data users. A summary
32 of the Quality Control Samples and the associated acceptance criteria is included in Table C3-3.
33 Key data-quality indicators for laboratory measurements are defined below.

1 Precision

2 Precision shall be assessed by analyzing laboratory duplicates and replicate analyses of
3 laboratory-control samples and PDP blind-audit samples. Results from measurements on these
4 samples must be compared to the criteria listed in Table C3-2. These QC measurements will be
5 used to demonstrate acceptable method performance and to trigger corrective action when
6 control limits are exceeded.

7 Accuracy

8 Accuracy as %R shall be assessed for the laboratory operations by analyzing PDP blind-audit
9 samples and laboratory-control samples. Results from these measurements must be compared
10 to the criteria listed in Table C3-2. These QC measurements will be used to demonstrate
11 acceptable method performance and to trigger corrective action when control limits are
12 exceeded.

13 Calibration

14 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated
15 using the procedures and criteria specified in Table C3-3. These criteria will be used to
16 demonstrate acceptable calibration and to trigger corrective action when control limits are
17 exceeded.

18 Method Detection Limit

19 MDLs shall be expressed in nanograms for VOCs and must be less than or equal to those listed
20 in Table C3-2. MDLs shall be determined based on the method described in Section C3-1. The
21 detailed procedures for MDL determination shall be included in site SOPs.

22 Program Required Quantitation Limit

23 Laboratories must demonstrate the capability to quantitate analytes at or below the PRQLs
24 given in Table C3-2. Laboratories shall set the concentration of at least one calibration standard
25 below the PRQL. The detailed procedures for PRQL demonstration shall be included in
26 laboratory SOPs.

27 Completeness

28 Laboratory completeness shall be expressed as the number of samples analyzed with valid
29 results as a percent of the total number of samples submitted for analysis. A composited sample
30 is treated as one sample for the purposes of completeness, because only one sample is run
31 through the analytical instrument. Valid results are defined as results that meet the data usability
32 criteria based on application of the Quality Control Criteria specified in Tables C3-2 and C3-3;
33 and meet the detection limit, calibration representativeness, and comparability criteria within this
34 section. The Permittees shall require that participating laboratories meet the completeness
35 criteria specified in Table C3-2.

1 Comparability

2 For VOC analysis, data generated through analysis of samples from different sites shall be
3 comparable. The Permittees shall require each site to achieve comparability by using
4 standardized methods and traceable standards and by requiring all sites to successfully
5 participate in the PDP (DOE, 2003).

6 Representativeness

7 Representativeness for VOC analysis shall be achieved by collecting sufficient numbers of
8 samples using clean sampling equipment that does not introduce sample bias. Samples must
9 be collected as described in Permit Attachment C1.

10 C3-6 Total Volatile Organic Compound Analysis

11 Quality Assurance Objectives

12 The development of DQOs specifically for this program has resulted in the QAOs listed in Table
13 C3-4. The specified QAOs represent the required quality of data necessary to draw valid
14 conclusions regarding program objectives. WAP-required limits, such as the PRQL associated
15 with VOC analysis, are specified to ensure that the analytical data collected satisfy the
16 requirements of all data users. Key data-quality indicators for laboratory measurements are
17 defined below.

18 Precision

19 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,
20 replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from
21 measurements on these samples must be compared to the criteria listed in Table C3-4. These
22 QC measurements will be used to demonstrate acceptable method performance and to trigger
23 corrective action when control limits are exceeded.

24 Accuracy

25 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control
26 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from
27 these measurements for matrix spikes samples must be compared to the %R criteria listed in
28 Table C3-4. Results for surrogates and internal standards are evaluated as specified in the SW-
29 846 method (EPA 1996) or Table C3-5. These QC measurements will be used to demonstrate
30 acceptable method performance and to trigger corrective action when control limits are
31 exceeded.

32 Laboratory blanks shall be assessed to determine possible laboratory contamination and are
33 evaluated as specified in Table C3-5. These QC measurements will be used to demonstrate
34 acceptable levels of laboratory contamination and to trigger corrective action when control limits
35 are exceeded.

1 Calibration

2 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated
3 using the procedures and criteria specified in Table C3-5 and the SW-846 method (EPA 1996).
4 These criteria will be used to demonstrate acceptable calibration and to trigger corrective action
5 when control limits are exceeded.

6 Method Detection Limit

7 MDLs shall be expressed in milligrams per kilogram (mg/kg) for VOCs and must be less than or
8 equal to those listed in Table C3-4. The detailed procedures for MDL determination shall be
9 included in site SOPs.

10 Program Required Quantitation Limit

11 Laboratories must demonstrate the capability to quantitate analytes in samples at or below the
12 PRQLs given in Table C3-4. Laboratories shall set the concentration of at least one calibration
13 standard below the PRQL. The detailed procedures for PRQL demonstration shall be included
14 in laboratory SOPs.

15 Completeness

16 Laboratory completeness shall be expressed as the number of samples analyzed with valid
17 results as a percent of the total number of samples submitted for analysis. Valid results are
18 defined as results that meet the data usability criteria based upon application of the Quality
19 Control Criteria specified in Tables C3-4 and C3-5 and meet the calibration, detection limit,
20 representativeness, and comparability criteria within this section. Participating laboratories must
21 meet the completeness criteria specified in Table C3-4.

22 Comparability

23 For VOC analysis, data generated through analysis of samples from different sites shall be
24 comparable. The Permittees shall require sites to achieve comparability by using standardized
25 SW-846 sample preparation and methods that meet the QAO requirements in Tables C3-4 and
26 C3-5, traceable standards, and by requiring all sites to successfully participate in the PDP
27 (DOE, 2005). Generator/storage sites may use the most recent version of SW-846. Any
28 changes to SW-846 methodology that results in the elimination of sample preparation or
29 analytical methods in use at generator/storage sites must be addressed as a corrective action to
30 address the comparability of data before and after the SW-846 modification.

31 Representativeness

32 Representativeness for VOC analysis shall be achieved by collecting unbiased samples.
33 Samples must be collected as described in Permit Attachment C1.

1 C3-7 Total Semivolatile Organic Compound Analysis

2 Quality Assurance Objectives

3 The development of DQOs specifically for this program has resulted in the QAOs listed in Table
4 C3-6. The specified QAOs represent the required quality of data necessary to draw valid
5 conclusions regarding program objectives. WAP-required limits, such as the PRQLs, are
6 specified to ensure that the analytical data collected satisfy the requirements of all data users. A
7 summary of Quality Control Samples and associated acceptance criteria for this analysis is
8 included in Table C3-7. Key data-quality indicators for laboratory measurements are defined
9 below.

10 Precision

11 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,
12 replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from
13 measurements on these samples must be compared to the criteria listed in Table C3-6. These
14 QC measurements will be used to demonstrate acceptable method performance and to trigger
15 corrective action when control limits are exceeded.

16 Accuracy

17 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control
18 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from
19 these measurements for matrix spikes samples must be compared to the %R criteria listed in
20 Table C3-6. Results for surrogates and internal standards are evaluated as specified in the SW-
21 846 method (EPA 1996) or Table C3-7. These QC measurements will be used to demonstrate
22 acceptable method performance and to trigger corrective action when control limits are
23 exceeded.

24 Laboratory blanks shall be assessed to determine possible laboratory contamination and are
25 evaluated as specified in Table C3-7. These QC measurements will be used to demonstrate
26 acceptable levels of laboratory contamination and to trigger corrective action when control limits
27 are exceeded.

28 Calibration

29 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated
30 using the procedures and criteria specified in Table C3-7 and the SW-846 method (EPA 1996).
31 These criteria will be used to demonstrate acceptable calibration and to trigger corrective action
32 when control limits are exceeded.

33 Method Detection Limit

34 MDLs shall be expressed in mg/kg for SVOCs and must be less than or equal to those listed in
35 Table C3-6. The detailed procedures for MDL determination shall be included in site SOPs.

1 Program Required Quantitation Limit

2 Laboratories must demonstrate the capability to quantitate analytes in samples at or below the
3 PRQLs given in Table C3-6. Laboratories shall set the concentration of at least one calibration
4 standard below the PRQL. The detailed procedures for PRQL demonstration shall be included
5 in laboratory SOPs.

6 Completeness

7 Laboratory completeness shall be expressed as the number of samples analyzed with valid
8 results as a percent of the total number of samples submitted for analysis. Valid results are
9 defined as results that meet the data usability criteria based on application of the Quality Control
10 Criteria specified in Tables C3-6 and C3-7 and meet the detection limit, calibration,
11 representativeness, and comparability criteria within this section. The Permittees shall require
12 participating laboratories to meet the level of completeness specified in Table C3-6.

13 Comparability

14 For SVOC analysis, data generated through analysis of samples from different sites shall be
15 comparable. The Permittees shall require sites to achieve comparability by using standardized
16 SW-846 sample preparation and methods that meet the QAO requirements in Tables C3-6 and
17 C3-7, traceable standards, and by requiring all sites to successfully participate in the PDP
18 (DOE, 2005). Generator/storage sites may use the most current version of SW-846 if the
19 methods are consistent with QAO requirements. Any changes to SW-846 methodology that
20 results in the elimination of sample preparation or analytical methods in use at
21 generator/storage sites must be addressed as a corrective action to address the comparability
22 of data before and after the SW-846 modification.

23 Representativeness

24 Representativeness for SVOC analysis shall be achieved by collecting unbiased samples.
25 Samples must be collected as described in Permit Attachment C1.

26 C3-8 Total Metal Analysis

27 Quality Assurance Objectives

28 The development of DQOs for the program has resulted in the QAOs listed in Table C3-8. The
29 specified QAOs represent the required quality of data necessary to draw valid conclusions
30 regarding program objectives. WAP-required limits, such as the PRQLs associated with metal
31 analysis, are specified to ensure that the analytical data collected satisfy the requirements of all
32 data users. A summary of Quality Control Samples and the associated acceptance criteria for
33 this analysis is provided in Table C3-9. Key data-quality indicators for laboratory measurements
34 are defined below.

35 Precision

36 Precision shall be assessed by analyzing laboratory sample duplicates or laboratory matrix
37 spike duplicates, replicate analyses of laboratory-control samples, and PDP blind-audit
38 samples. Results from measurements on these samples must be compared to the criteria listed

1 in Table C3-8. These QC measurements will be used to demonstrate acceptable method
2 performance and to trigger corrective action when control limits are exceeded.

3 Accuracy

4 Accuracy shall be assessed through the analysis of laboratory matrix spikes, PDP blind-audit
5 samples, serial dilutions, interference check samples, and laboratory-control samples. Results
6 from these measurements must be compared to the criterion listed in Table C3-8 and C3-9.
7 These QC measurements will be used to demonstrate acceptable method performance and to
8 trigger corrective action when control limits are exceeded.

9 Laboratory blanks and calibration blanks shall be assessed to determine possible laboratory
10 contamination and are evaluated as specified in Table C3-9. These QC measurements will be
11 used to demonstrate acceptable levels of laboratory contamination and to trigger corrective
12 action when control limits are exceeded.

13 Calibration

14 Mass Tunes (for ICP MS only), Standards Calibration, Initial Calibration verifications, and
15 Continuing Calibrations will be performed and evaluated using the procedures and criteria
16 specified in Table C3-9 and the SW-846 method (EPA 1996). These criteria will be used to
17 demonstrate acceptable calibration and to trigger corrective action when control limits are
18 exceeded.

19 Program Required Detection Limits

20 PRDLs, expressed in units of micrograms per L ($\mu\text{g/L}$), are the maximum values for instrument
21 detection limits (**IDL**) permissible for program support under the WAP. IDLs must be less than or
22 equal to the PRDL for the method used to quantitate a specific analyte. Any method listed in
23 Table C-5 of the Waste Analysis Plan (Permit Attachment C) may be used if the IDL meets this
24 criteria. For high concentration samples, an exception to the above requirements may be made
25 in cases where the sample concentration exceeds five times the IDL of the instrument being
26 used. In this case, the analyte concentration may be reported even though the IDL may exceed
27 the PRDL. IDLs shall be determined semiannually (i.e., every six months). Detailed procedures
28 for IDL determination shall be included in laboratory SOPs.

29 Program Required Quantitation Limit

30 The Permittees shall require participating laboratories to demonstrate the capability of analyte
31 quantitation at or below the PRQLs in units of mg/kg wet weight (given in Table C3-8). The
32 PRDLs are set an order of magnitude less than the PRQLs (assuming 100 percent solid sample
33 diluted by a factor of 100 during preparation). The Permittees shall require participating
34 laboratories to set the concentration of at least one QC or calibration standard at or below the
35 solution concentration equivalent of the PRQL. Detailed calibration procedures shall be included
36 in site SOPs.

37 Completeness

38 Laboratory completeness shall be expressed as the number of samples analyzed with valid
39 results as a percent of the total number of samples submitted for analysis. Valid results are

1 defined as results that meet the data usability criteria based upon application of the Quality
2 Control Criteria specified in Tables C3-8 and C3-9 and meet the detection limit, calibration,
3 representativeness, and comparability criteria within this section. The Permittees shall require
4 participating laboratories to meet the completeness specified in Table C3-8.

5 Comparability

6 For metals analysis, data generated through analysis of samples from different sites shall be
7 comparable. Comparability will be achieved by using standardized SW-846 sample preparation
8 and methods that meet QAO requirements in Tables C3-8 and C3-9, demonstrating successful
9 participation in the PDP (DOE, 2005), and use of traceable standards. Generator/storage sites
10 may use the most recent SW-846 update. Any changes to SW-846 methodology that results in
11 the elimination of sample preparation or analytical methods in use at generator/storage sites
12 must be addressed as a corrective action to address the comparability of data before and after
13 the SW-846 modification.

14 Representativeness

15 Representativeness for metals analysis shall be achieved by the collection of unbiased samples
16 and the preparation of samples in the laboratory using representative and unbiased methods.
17 Samples must be collected as described in Permit Attachment C1.

18 C3-9 Acceptable Knowledge

19 Acceptable knowledge documentation provides primarily qualitative information that cannot be
20 assessed according to specific data quality goals that are used for analytical techniques. QAOs
21 for analytical results are described in terms of precision, accuracy, completeness, comparability,
22 and representativeness. Appropriate analytical and testing results may be used to augment the
23 characterization of wastes based on acceptable knowledge. To ensure that the acceptable
24 knowledge process is consistently applied, The Permittees shall require sites to comply with the
25 following data quality requirements for acceptable knowledge documentation:

26 Precision - Precision is the agreement among a set of replicate measurements without
27 assumption of the knowledge of a true value. The qualitative determinations, such as
28 compiling and assessing acceptable knowledge documentation, do not lend
29 themselves to statistical evaluations of precision. However, the acceptable knowledge
30 information will be addressed by the independent review of acceptable knowledge
31 information during internal and external audits.

32 Accuracy - Accuracy is the degree of agreement between an observed sample result and
33 the true value. The percentage of waste containers which require reassignment to a
34 new waste matrix code and/or designation of different hazardous waste numbers
35 based on sampling and analysis data and discrepancies identified by the Permittees
36 during waste confirmation will be reported as a measure of acceptable knowledge
37 accuracy.

38 Completeness - Completeness is an assessment of the number of waste streams or
39 number of samples collected to the number of samples determined to be useable
40 through the data validation process. The acceptable knowledge record must contain

1 100 percent of the required information (Permit Attachment C4-3). The usability of the
2 acceptable knowledge information will be assessed for completeness during audits.

3 Comparability - Data are considered comparable when one set of data can be compared
4 to another set of data. Comparability is ensured through sites meeting the training
5 requirements and complying with the minimum standards outlined for procedures that
6 are used to implement the acceptable knowledge process. All sites must assign
7 hazardous waste numbers in accordance with Permit Attachment C4-3b and provide
8 this information regarding its waste to other sites who store or generate a similar waste
9 stream.

10 Representativeness - Representativeness expresses the degree to which sample data
11 accurately and precisely represent characteristics of a population. Representativeness
12 is a qualitative parameter that will be satisfied by ensuring that the process of
13 obtaining, evaluating, and documenting acceptable knowledge information is
14 performed in accordance with the minimum standards established in Permit
15 Attachment C4. Sites also must assess and document the limitations of the acceptable
16 knowledge information used to assign hazardous waste numbers (e.g., purpose and
17 scope of information, date of publication, type and extent to which waste parameters
18 are addressed).

19 The Permittees shall require each generator/storage site to comply with the nonconformance
20 notification and reporting requirements of Section C3-13 if the results of sampling and analysis
21 specified in Permit Attachment C are inconsistent with acceptable knowledge documentation.

22 The Permittees shall require each site to address quality control by tracking its performance with
23 regard to the use of acceptable knowledge by: 1) assessing the frequency of inconsistencies
24 among information, and 2) documenting acceptable knowledge inconsistencies identified
25 through radiography, visual examination, headspace-gas analyses, and solidified waste
26 analyses. In addition, the acceptable knowledge process and waste stream documentation must
27 be evaluated through internal assessments by generator/storage site quality assurance
28 organizations and assessments by auditors external to the organization (i.e., the Permittees).

29 C3-10 Data Review, Validation, and Verification Requirements

30 Procedures shall be developed for the review, validation, and verification of data at the data
31 generation level; the validation and verification of data at the project level; and the verification of
32 data at the Permittee level. Data review determines if raw data have been properly collected
33 and ensures raw data are properly reduced. Data validation verifies that the data reported
34 satisfy the requirements of this WAP and is accompanied by signature release. Data verification
35 authenticates that data as presented represent the sampling and analysis activities as
36 performed and have been subject to the appropriate levels of data review. The requirements
37 presented in this section ensure that WAP records furnish documentary evidence of quality.

38 The Permittees shall require the sites to generate the following Batch Data Reports for data
39 validation, verification, and quality assurance activities:

40 A Testing Batch Data Report or equivalent includes all data pertaining to radiography or
41 visual examination for up to 20 waste containers without regard to waste matrix. Table
42 C3-11 lists all of the information required in Testing Batch Data Reports (identified with

1 an "X") and other information that is necessary for data validation, but is optional in
2 Testing Batch Data Reports (identified with an "O").

3 A Sampling Batch Data Report or equivalent includes all sample collection data pertaining
4 to a group of no more than 20 headspace gas or homogeneous waste samples that
5 were collected for chemical analysis. Table C3-12 lists all of the information required in
6 Sampling Batch Data Reports (identified with an "X") and other information that is
7 necessary for data validation, but is optional in Sampling Batch Data Reports
8 (identified with an "O").

9 An Analytical Batch Data Report or equivalent includes analytical data from the analysis of
10 TRU-mixed waste for up to 20 headspace gas or homogeneous waste samples.
11 Analytical Batch Data Reports or equivalent that contain results for composited
12 headspace gas samples must contain sufficient information to identify the containers
13 that were composited for each composite sample and the sample volume that was
14 taken from each waste container. Because Analytical Batch Data Reports are
15 generated based on the number of samples analyzed, an Analytical Batch Data Report
16 may contain results that are applicable to more than 20 containers depending on how
17 many composite samples are part of the report, but may not exceed a total of 20
18 samples analyzed. Table C3-13 lists all of the information required in Analytical Batch
19 Data Reports (identified with an "X") and other information that is necessary for data
20 validation, but is optional in Analytical Batch Data Reports (identified with an "O").

21 Raw analytical data need not be included in Analytical Batch Data Reports, but must
22 be maintained in the site project files and be readily available for review upon request.
23 Raw data may include all analytical bench sheet and instrumentation readouts for all
24 calibration standard results, sample data, QC samples, sample preparation conditions
25 and logs, sample run logs, and all re-extraction, re-analysis, or dilution information
26 pertaining to the individual samples. Raw data may also include calculation records
27 and any qualitative or semi-quantitative data collected for a sample and that has been
28 recorded on a bench sheet or in a log book.

29 An On-line Batch Data Report or equivalent contains the combined information from the
30 Sampling Batch Data Report and Analytical Batch Data Report that is relevant to the
31 on-line method used.

32 C3-10a Data Generation Level

33 The following are minimum requirements for raw data collection and management which the
34 Permittees shall require for each site:

35 All raw data shall be signed and dated in reproducible ink by the person generating it.
36 Alternately, unalterable electronic signatures may be used.

37 All data must be recorded clearly, legibly, and accurately in field and laboratory records
38 (bench sheets, logbooks), and include applicable sample identification numbers (for
39 sampling and analytical labs).

40 All changes to original data must be lined out, initialed, and dated by the individual making
41 the change. A justification for changing the original data may also be included. Original

1 data must not be obliterated or otherwise disfigured so as not to be readable. Data
2 changes shall only be made by the individual who originally collected the data or an
3 individual authorized to change the data.

4 All data must be transferred and reduced from field and laboratory records completely and
5 accurately.

6 All field and laboratory records must be maintained as specified in Table C-6 of
7 Attachment C.

8 Data must be organized into a standard format for reporting purposes (Batch Data Report),
9 as outlined in specific sampling and analytical procedures.

10 All electronic and video data must be stored appropriately to ensure that waste container,
11 sample, and associated QC data are readily retrievable. In the case of classified
12 information, additional security provisions may apply that could restrict retrievability.
13 The additional security provisions will be documented in generator/storage site
14 procedures as outlined in the QAPjP in accordance with prevailing classified
15 information security standards.

16 Data review, validation, and verification at this level involves scrutiny and signature release from
17 qualified independent technical reviewer(s) not involved in the generation or recording of the
18 data under review, as specified below. Individuals conducting this data review, validation, and
19 verification must use checklists that address all of the items included in this section. Checklists
20 must contain or reference tables showing the results of sampling, analytical or on-line batch QC
21 samples, if applicable. Checklists must reflect review of all QC samples and quality assurance
22 objective categories in accordance with criteria established in Tables C3-2 through C3-9 (as
23 applicable to the methods validated). Completed checklists must be forwarded with Batch Data
24 Reports to the project level. Analytical raw data must be available and reviewed by the data
25 generation level reviewer.

26 C3-10a(1) Independent Technical Review

27 The independent technical review ensures by review of raw data that data generation and
28 reduction are technically correct; calculations are verified correct; deviations are documented;
29 and QA/QC results are complete, documented correctly, and compared against WAP criteria.
30 This review validates and verifies all of the work documented by the originator.

31 One hundred percent of the Batch Data Reports must receive an independent technical review
32 by a trained and qualified individual who was not involved in the generation or recording of the
33 data under review. This review shall be performed by an individual other than the data generator
34 who is qualified to have performed the initial work. The independent technical review must be
35 performed as soon as practicably possible in order to determine and correct negative quality
36 trends in the sampling or analytical process. However at a minimum, the independent technical
37 review must be performed before any waste associated with the data reviewed is managed,
38 stored, or disposed at WIPP, unless the data are being obtained from waste sampling and
39 analysis as containers are being retrieved or generated after initial WSPF approval as described
40 in Attachment C2, Section C2-1. The reviewer(s) must release the data as evidenced by
41 signature, and as a consequence ensure the following:

1 Data generation and reduction were conducted in a technically correct manner in
2 accordance with the methods used (procedure with revision). Data were reported in
3 the proper units and correct number of significant figures.

4 Calculations have been verified by a valid calculation program, a spot check of verified
5 calculation programs, and/or 100 percent check of all hand calculations. Values that
6 are not verifiable to within rounding or significant difference discrepancies must be
7 rectified prior to completion of independent technical review.

8 The data have been reviewed for transcription errors.

9 The testing, sampling, or analytical data QA documentation for Batch Data Reports is
10 complete and includes, as applicable, raw data, DAC and equilibrium calculations and
11 times, calculation records, chain-of-custody (**COC**) forms, calibration records (or
12 references to an available calibration package), QC sample results, and copies or
13 originals of gas canister sample tags. Corrective action will be taken to ensure that all
14 Batch Data Reports are complete and include all necessary raw data prior to
15 completion of the independent technical review.

16 QC sample results are within established control limits, and if not, the data have been
17 appropriately qualified in accordance with data usability criteria. Data outside of
18 established control limits will be qualified as appropriate, assigned an appropriate
19 qualifier flag, discussed in the case narrative, and included as appropriate in
20 calculations for completeness. QC criteria that were not met are documented.

21 Reporting flags (Table C3-14) were assigned correctly.

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

23 Radiography tapes have been reviewed (independent observation) on a waste container
24 basis at a minimum of once per testing batch or once per day of operation, whichever
25 is less frequent (Attachment C1, Section C1-3). The radiography tape will be reviewed
26 against the data reported on the radiography form to ensure that the data are correct
27 and complete.

28 Field sampling records are complete. Incomplete or incorrect field sampling records will be
29 subject to resubmittal prior to completion of the independent technical review.

30 QAOs have been met according to the methods outlined in Sections C3-2 through C3-9.

31 C3-10b Project Level

32 Data validation and verification at this level involves scrutiny and signature release from the Site
33 Project Manager (or designee). The Permittees shall require each site to meet the following
34 minimum requirements for each waste container. Any nonconformance identified during this
35 process shall be documented on a nonconformance report (Section C3-13).

36 The Site Project Manager shall ensure that a repeat of the data generation level review,
37 validation, and verification is performed on the data for a minimum of one randomly chosen
38 waste container quarterly (every three months). This exercise will document that the data

1 generation level review, validation, and verification is being performed according to
2 implementing procedures.

3 C3-10b(1) Site Project Manager Review

4 The Site Project Manager Review is the final validation that all of the data contained in Batch
5 Data Reports from the data generation level are complete and have been properly reviewed as
6 evidenced by signature release and completed checklists.

7 One hundred percent of the Batch Data Reports must have Site Project Manager signature
8 release. At a minimum, the Site Project Manager signature release must be performed before
9 any waste associated with the data reviewed is managed, stored, or disposed at WIPP, unless
10 the data are being obtained from waste sampling and analysis as containers are being retrieved
11 or generated as described in Permit Attachment C2, Section C2-1. This signature release must
12 ensure the following:

13 The validity of the DAC assignment made at the data generation level based upon an
14 assessment of the data collection and evaluation necessary to make the assignment.

15 Testing batch QC checks (e.g., replicate scans, measurement system checks) were
16 properly performed. Radiography data are complete and acceptable based on
17 evidence of videotape review of one waste container per day or once per testing batch,
18 whichever is less frequent, as specified in Permit Attachment C1, Section C1-3.

19 Sampling batch QC checks (e.g., equipment blanks, field duplicates, field reference
20 standards) were properly performed, and meet the established QAOs and are within
21 established data usability criteria.

22 Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix spikes,
23 matrix spike duplicates, laboratory control samples) were properly performed and meet
24 the established QAOs and are within established data usability criteria.

25 On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates, on-line
26 control samples) were properly performed and meet the established QAOs and are
27 within established data usability criteria.

28 Proper procedures were followed to ensure representative samples of headspace gas and
29 homogeneous solids and soil/gravel were taken.

30 Data generation level independent technical review, validation, and verification have been
31 performed as evidenced by the completed review checklists and appropriate signature
32 releases.

33 Independent technical reviewers were not involved in the generation or recording of the
34 data under review.

35 Batch data review checklists are complete.

1 Batch Data Reports are complete and data are properly reported (e.g., data are reported
2 in the correct units, with the correct number of significant figures, and with qualifying
3 flags).

4 Verify that data are within established data assessment criteria and meet all applicable
5 QAOs (Sections C3-2 through C3-9).

6 C3-10b(2) Prepare Site Project Manager Summary and Data Validation Summary

7 To document the project-level validation and verification described above, the Permittees shall
8 require each Site Project Manager (or designee) to prepare a Site Project Manager Summary
9 and a Data Validation Summary. These reports may be combined to eliminate redundancy. The
10 Site Project Manager Summary includes a validation checklist for each Batch Data Report.
11 Checklists for the Site Project Manager Summary must be sufficiently detailed to validate all
12 aspects of a Batch Data Report that affect data quality. The Data Validation Summary provides
13 verification that, on a per waste container or sample basis as evidenced by Batch Data Report
14 reviews, all data have been validated in accordance with the site QAPjP. The Data Validation
15 Summary must identify each Batch Data Report reviewed (including all waste container
16 numbers), describe how the validation was performed and whether or not problems were
17 detected (e.g., nonconformance reports), and include a statement indicating that all data are
18 acceptable. Summaries must include release signatures.

19 Once the data have received project-level validation and verification or when the Site Project
20 Manager decides the sample no longer needs to be retained, the Site Project Manager must
21 ensure that the laboratory is notified. Samples must be retained by the laboratory until this
22 notification is received. Gas sample canisters may then be released from storage for cleaning,
23 recertification, and subsequent reuse. Sample tags must be removed and retained in the project
24 files before recycling the canisters. If the Site Project Manager requests that samples or
25 canisters be retained for future use (e.g., an experimental holding time study), the same sample
26 identification and COC forms shall be used and cross-referenced to a document which specifies
27 the purpose for sample or canister retention.

28 C3-10b(3) Prepare Waste Stream Characterization Package

29 In the event the Permittees request detailed information on a waste stream, the Site Project
30 Manager will provide a Waste Stream Characterization Package. The Site Project Manager
31 must ensure that the Waste Stream Characterization Package (Section C3-12b(3)) will support
32 waste characterization determinations.

33 C3-10c Permittee Level

34 The final level of data verification occurs at the Permittee level and must, at a minimum, consist
35 of reviewing a sample of the Batch Data Reports during audits of generator/storage sites and
36 DOE approved laboratories to verify completeness. During such audits, DOE is responsible for
37 the verification that Batch Data Reports include the following:

38 Project-level signature releases

39 Listing of all waste containers being presented in the report

1 Listing of all testing, sampling, and analytical batch numbers associated with each waste
2 container being reported in the package

3 Analytical Batch Data Report case narratives

4 Site Project Manager Summary

5 Data Validation Summary

6 Complete summarized qualitative and quantitative data for all waste containers with data
7 flags and qualifiers.

8 For each Waste Stream Profile Form (**WSPF**) submitted for approval, DOE must verify that each
9 submittal (i.e., WSPF and Characterization Information Summary) is complete and notify the
10 originating site in writing of the WSPF approval. DOE will maintain the data as appropriate for
11 use in the regulatory compliance programs. For subsequent shipments made after the initial
12 WSPF approval, the verification will also include WWIS internal limit checks (Attachment C,
13 Section C-5a(1)).

14 C3-11 Reconciliation with Data Quality Objectives

15 Reconciling the results of waste testing and analysis with the DQOs provides a way to ensure
16 that data will be of adequate quality to support the regulatory compliance programs.

17 Reconciliation with the DQOs will take place at both the project level and the Permittees' level.
18 At the project level, reconciliation will be performed by the Site Project Manager, while at the
19 Permittees' level, reconciliation will be performed as described below.

20 C3-11a Reconciliation at the Project Level

21 The Permittees shall require each Site Project Manager to ensure that all data generated and
22 used in decision making meet the DQOs provided in Section C-4a(1) of Permit Attachment C.
23 To do so, the Site Project Manager must assess whether data of sufficient type, quality, and
24 quantity have been collected. The Site Project Manager must determine if the variability of the
25 data set is small enough to provide the required confidence in the results. The Site Project
26 Manager must also determine if, based on the desired error rates and confidence levels, a
27 sufficient number of valid data points have been determined (as established by the associated
28 completeness rate for each sampling and analytical process). In addition, the Site Project
29 Manager must document that random sampling of containers was performed for the purposes of
30 waste stream characterization.

31 For each waste stream characterized, the Permittees shall require each Site Project Manager to
32 determine if sufficient data have been collected to determine the following WAP-required waste
33 parameters, as applicable:

34 Waste matrix code

35 Waste material parameter weights

36 If each waste container of waste contains TRU radioactive waste

- 1 Mean concentrations, UCL_{90} for the mean concentrations, standard deviations, and the
2 number of samples collected for each VOC in the headspace gas of waste containers
3 in the waste stream
- 4 Mean concentrations, UCL_{90} for the mean concentrations, standard deviations, and
5 number of samples collected for VOCs, SVOCs, and metals in the waste stream
- 6 Whether the waste stream exhibits a toxicity characteristic (**TC**) under 40 CFR Part 261,
7 Subpart C
- 8 Whether the waste stream contains listed waste found in 20.4.1.200 NMAC incorporating
9 40 CFR Part 261, Subpart D
- 10 Whether the waste stream can be classified as hazardous or nonhazardous at the 90-
11 percent confidence level
- 12 Whether an appropriate packaging configuration and DAC were applied and documented
13 in the headspace gas sampling documentation, and whether the drum age was met
14 prior to sampling.
- 15 Whether all TICs were appropriately identified and reported in accordance with the
16 requirements of Section C3-1 prior to submittal of a WSPF for a waste stream or waste
17 stream lot.
- 18 Whether the overall completeness, comparability, and representativeness QAOs were met
19 for each of the analytical and testing procedures as specified in Sections C3-2 through
20 C3-9 prior to submittal of a WSPF for a waste stream or waste stream lot.
- 21 Whether the PRQLs for all analyses were met prior to submittal of a WSPF for a waste
22 stream or waste stream lot.
- 23 If the Site Project Manager determines that insufficient data have been collected to make the
24 determinations listed above, additional data collection efforts must be undertaken. The
25 reconciliation of a waste stream shall be performed, as described in Permit Attachment C4, prior
26 to submittal of WSPF and Characterization Information Summary to the Permittees for that
27 waste stream. The Permittees shall not manage, store, or dispose a TRU mixed waste stream
28 at WIPP unless the Site Project Manager determines that the WAP-required waste parameters
29 listed above have been met for that waste stream.
- 30 The statistical procedure presented in Permit Attachment C2 shall be used by participating Site
31 Project Managers to evaluate and report waste characterization data from the analysis of
32 homogeneous solids and soil/gravel. The procedure, which calculates UCL_{90} values, shall be
33 used to assess compliance with the DQOs in Attachment C, Section C-4a(1) as well as with
34 RCRA regulations. The procedure must be applied to all laboratory analytical data for total
35 VOCs, total SVOCs, and total metals. For RCRA regulatory compliance (40 CFR §261.24), data
36 from the analysis of the appropriate metals and organic compounds shall be expressed as
37 toxicity characteristic leaching procedure (**TCLP**) values or results may also be compared to the
38 TC levels expressed as total values. These total values will be considered the regulatory
39 threshold limit (**RTL**) values for the WAP. RTL values are obtained by calculating the

1 weight/weight concentration (in the solid) of a TC analyte that would give the regulatory
2 weight/volume concentration (in the TCLP extract), assuming 100-percent analyte dissolution.

3 C3-11b Reconciliation at the Permittee Level

4 The Permittees must also ensure that data of sufficient type, quality, and quantity are collected
5 to meet WAP DQOs. The Permittees will ensure sufficient data have been collected to
6 determine if the waste characterization information is adequate to demonstrate the Permittees'
7 compliance with Attachment C, Section C-4a(1). This is performed during the Permittees' review
8 of the WSPF and Characterization Information Summary and is documented by DOE's approval
9 of the WSPF.

10 C3-12 Data Reporting Requirements

11 Data reporting requirements define the type of information and the method of transmittal for data
12 transfer from the data generation level to the project level and from the project level to the
13 Permittees.

14 C3-12a Data Generation Level

15 Data shall be transmitted by hard copy or electronically (provided a hard copy is available on
16 demand) from the data generation level to the project level. Transmitted data shall include all
17 Batch Data Reports and data review checklists. The Batch Data Reports and checklists used
18 must contain all of the information required by the testing, sampling, and analytical techniques
19 described in Permit Attachments C1 through C6, as well as the signature releases to document
20 the review, validation, and verification as described in Section C3-10. All Batch Data Reports
21 and checklists shall be in approved formats, as provided in site-specific documentation.

22 Batch Data Reports shall be forwarded to the Site Project Manager. All Batch Data Reports
23 shall be assigned serial numbers, and each page shall be numbered. The serial number used
24 for Batch Data Reports can be the same as the testing, sampling, or analytical batch number.

25 QA documentation, including raw data, shall be maintained in either testing, sampling, and
26 analytical facility files, or site project files for those facilities located on site in accordance with
27 the document storage requirements of site approved site QAPjPs. DOE approved laboratories
28 shall forward testing, sampling, and analytical QA documentation along with Batch Data Reports
29 to the site project office for inclusion in site project files.

30 C3-12b Project Level

31 The site project office shall prepare a WSPF for each waste stream certified for shipment to
32 WIPP based on information obtained from acceptable knowledge and Batch Data Reports, if
33 applicable. In addition, the site project office must ensure that the Characterization Information
34 Summary and the Waste Stream Characterization Package (when requested by the Permittees)
35 are prepared as appropriate. The Site Project Manager must also verify these reports are
36 consistent with information found in analytical batch reports. Summarized testing, sampling, and
37 analytical data are included in the Characterization Information Summary. The contents of the
38 WSPF, Characterization Information Summary, and Waste Stream Characterization Package
39 are discussed in the following sections.

1 After approval of a WSPF and the associated Characterization Information Summary by DOE,
2 the generator/storage site are required to maintain a cross reference of container identification
3 numbers to each Batch Data Report.

4 A Waste Stream Characterization Package shall be transmitted by hard copy or electronically
5 from the Site Project Manager to the Permittees when requested.

6 C3-12b(1) Waste Stream Profile Form

7 The Waste Stream Profile Form (WSPF, Figure C-1) shall include the following information:

8 Generator/storage site name

9 Generator/storage site EPA ID

10 Date of audit report approval by NMED (if obtained)

11 Original generator of waste stream

12 Whether waste is Contact-Handled or Remote-Handled

13 The Waste Stream WIPP Identification Number

14 Summary Category Group

15 Waste Matrix Code Group

16 Waste Material Parameter Weight Estimates per unit of waste

17 Waste stream name

18 A description of the waste stream

19 Applicable EPA hazardous waste numbers

20 Applicable TRUCON codes

21 A listing of acceptable knowledge documentation used to identify the waste stream

22 The waste characterization procedures used and the reference and date of the procedure

23 Certification signature of Site Project Manager, name, title, and date signed

24 C3-12b(2) Characterization Information Summary

25 The Characterization Information Summary shall include the following elements, if applicable:

26 Data reconciliation with DQOs

1 Headspace gas summary data listing the identification numbers of samples used in the
2 statistical reduction, the maximum, mean, standard deviation, UCL₉₀, RTL, and
3 associated EPA hazardous waste numbers that must be applied to the waste stream.

4 Total metal, VOC, and SVOC analytical results for homogeneous solids and soil/gravel (if
5 applicable).

6 TIC listing and evaluation.

- 7 • Radiography and VE summary to document that all prohibited items are absent in the
8 waste (if applicable).

9 A justification for the selection of radiography and/or/VE as an appropriate method for
10 characterizing the waste.

11 A complete listing of all container identification numbers used to generate the WSPF,
12 cross-referenced to each Batch Data Report

13 Complete AK summary, including stream name and number, point of generation, waste
14 stream volume (current and projected), generation dates, TRUCON codes, Summary
15 Category Group, Waste Matrix Code(s) and Waste Matrix Code Group, other TWBIR
16 information, waste stream description, areas of operation, generating processes,
17 RCRA determinations, radionuclide information, all references used to generate the
18 AK summary, and any other information required by Permit Attachment C4, Section
19 C4-2b.

20 Method for determining Waste Material Parameter Weights per unit of waste.

21 List of any AK Sufficiency Determinations requested for the waste stream.

22 Certification through acceptable knowledge or testing and/or analysis that any waste
23 assigned the hazardous waste number of U134 (hydrofluoric acid) no longer exhibits
24 the characteristic of corrosivity. This is verified by ensuring that no liquid is present in
25 U134 waste.

26 C3-12b(3) Waste Stream Characterization Package

27 The Waste Stream Characterization Package includes the following information:

28 Waste Stream Profile Form (WSPF, Section C3-12b(1))

29 Accompanying Characterization Information Summary (Section C3-12b(2))

30 Complete AK summary (Section C3-12b(2))

31 Batch Data Reports supporting the characterization of the waste stream and any others
32 requested by the Permittees

33 Raw analytical data requested by the Permittees

1 C3-12b(4) WIPP Waste Information System (WWIS) Data Reporting

2 The WWIS Data Dictionary includes all of the data fields, the field format and the limits
3 associated with the data as established by this WAP. These data will be subjected to edit and
4 limit checks that are performed automatically by the database, as defined in the *Waste Data*
5 *System User's Manual* (DOE, 2009). If a container was part of a composite headspace gas
6 sample, the analytical results from the composite sample must be assigned as the container
7 headspace gas data results, including associated TICs, for every waste container associated
8 with the composite sample.

9 C3-13 Nonconformances

10 The Permittees shall require the status of work and the WAP activities at participating
11 generator/storage sites to be monitored and controlled by the Site Project Manager. This
12 monitoring and control shall include nonconformance identification, documentation, and
13 reporting.

14 The nonconformances and corrective action processes specified in this section describe
15 procedures between the Permittees and the generator/storage sites.

16 Nonconformances

17 Nonconformances are uncontrolled and unapproved deviations from an approved plan or
18 procedure. Nonconforming items and activities are those that do not meet the WAP
19 requirements, procurement document criteria, or approved work procedures. Nonconforming
20 items shall be identified by marking, tagging, or segregating, and the affected generator/storage
21 site(s) notified. Any waste container for which a nonconformance report (**NCR**) has been written
22 will not be shipped to the WIPP facility unless the condition that led to the NCR for that
23 container has been dispositioned in accordance with DOE's Quality Assurance Program
24 Description (**QAPD**). Disposition of nonconforming items shall be identified and documented.
25 The QAPjPs shall identify the person(s) responsible for evaluating and dispositioning
26 nonconforming items and shall include referenced procedures for handling them. For each
27 container selected for confirmation pursuant to Permit Attachment C7, the Permittees will
28 examine the respective NCR documentation to verify NCRs have been dispositioned for the
29 selected container.

30 Management at all levels shall foster a "no-fault" attitude to encourage the identification of
31 nonconforming items and processes. Nonconformances may be detected and identified by
32 anyone performing WAP activities, including

33 Project staff - during field operations, supervision of subcontractors, data validation and
34 verification, and self-assessment

35 Laboratory staff - during the preparation for and performance of laboratory testing;
36 calibration of equipment; QC activities; laboratory data review, validation, and
37 verification; and self-assessment

38 QA personnel - during oversight activities or audits

1 A NCR shall be prepared for each nonconformance identified. Each NCR shall be initiated by
2 the individual(s) identifying the nonconformance. The NCR shall then be processed by
3 knowledgeable and appropriate personnel. For this purpose, a NCR including, or referencing as
4 appropriate, results of laboratory analysis, QC tests, audit reports, internal memoranda, or
5 letters shall be prepared. The NCR must provide the following information:

- 6 • Identification of the individual(s) identifying or originating the nonconformance
- 7 • Description of the nonconformance
- 8 • Method(s) or suggestions for correcting the nonconformance (corrective action)
- 9 • Schedule for completing the corrective action
- 10 • An indication of the potential ramifications and overall usability of the data, if applicable
- 11 • Any approval signatures specified in the site nonconformance procedures

12 The Permittees shall require the Site Project Manager to oversee the NCR process and be
13 responsible for developing a plan to identify and track all nonconformances and report this
14 information to the Permittees. The Site Project Manager is also responsible for notifying project
15 personnel of the nonconformance and verifying completion of the corrective action for
16 nonconformances.

17 Nonconformance to DQOs

18 For any non-administrative nonconformance related to applicable requirements specified in this
19 WAP which are first identified at the Site Project Manager signature release level (i.e., a failure
20 to meet a DQO), the Permittees shall receive written notification within seven calendar days of
21 identification and shall also receive a NCR within 30 calendar days of identification of the
22 incident. DOE shall require the generator/storage site to implement a corrective action which
23 remedies the nonconformance prior to management, storage, or disposal of the waste at WIPP.
24 The Permittees shall send NMED a monthly summary of nonconformances identified during the
25 previous month, indicating the number of nonconformances received and the generator/storage
26 sites responsible.

27 DOE's Corrective Action Process

28 DOE shall initiate a corrective action process when internal nonconformances and
29 nonconformances at the generator/storage sites are identified. Activities and processes that do
30 not meet requirements are documented as deficiencies.

31 When a deficiency is identified by the Permittees, the following process action steps are
32 required:

33 The condition is documented on a Corrective Action Report (**CAR**) by the individual
34 identifying the problem.

35 DOE has designated the CAR Initiator and Assessment Team Leader to review the CAR,
36 determine validity of the finding (determine that a requirement has been violated),
37 classify the significance of the condition, assign a response due date, and issue the
38 CAR to the responsible party.

1 The responsible organization reviews the CAR, evaluates the extent and cause of the
2 deficiency and provides a response to DOE, indicating remedial actions and actions to
3 preclude recurrence that will be taken.

4 DOE reviews the response from the responsible organization and, if acceptable,
5 communicates the acceptance to the responsible organization.

6 The responsible organization completes remedial actions and actions to preclude
7 recurrence of the condition.

8 After all corrective actions have been completed, DOE schedules and performs a
9 verification to ensure that corrective actions have been completed and are effective.
10 When all actions have been completed and verified as being effective, the CAR is
11 closed by the CAR Initiator and Assessment Team Leader on behalf of DOE.

12 As part of the planning process for subsequent audits and surveillances, past deficiencies
13 are reviewed and the previous deficient activity or process is subject to reassessment.

14 C3-14 Special Training Requirements and Certifications

15 Before performing activities that affect WAP quality, all personnel are required to receive
16 indoctrination into the applicable scope, purpose, and objectives of the WAP and the specific
17 QAOs of the assigned task. Personnel assigned to perform activities for the WAP shall have the
18 education, experience, and training applicable to the functions associated with the work.
19 Evidence of personnel proficiency and demonstration of competence in the task(s) assigned
20 must be demonstrated and documented. All personnel designated to work on specific aspects of
21 the WAP shall maintain qualification (i.e., training and certification) throughout the duration of
22 the work as specified in this WAP and applicable QAPjPs/procedures. Job performance shall be
23 evaluated and documented at periodic intervals, as specified in the implementing procedures.

24 Personnel involved in WAP activities shall receive continuing training to ensure that job
25 proficiency is maintained. If not specified by this WAP, the due date for required continuing
26 training courses and requalification shall be the end of the month of the anniversary date when
27 the training was previously completed. Training includes both education in principles and
28 enhancement of skills. Each participating site shall include in its QAPjP a description of the
29 procedures for implementing personnel qualification and training. All training records that
30 specify the scope of the training, the date of completion, and documentation of job proficiency
31 shall be maintained as QA Records in the site project file.

32 Analytical laboratory line management must ensure that analytical personnel are qualified to
33 perform the analytical method(s) for which they are responsible. The minimum qualifications for
34 certain specified positions for the WAP are summarized in Table C3-10. QAPjPs, or their
35 implementing SOPs, shall specify the site-specific titles and minimum training and qualification
36 requirements for personnel performing WAP activities. QAPjPs/procedures shall also contain
37 the requirements for maintaining records of the qualification, training, and demonstrations of
38 proficiency by these personnel.

39 An evaluation of personnel qualifications shall include comparing and evaluating the
40 requirements specified in the job/position description and the skills, training, and experience
41 included in the current resume of the person. This evaluation also must be performed for

1 personnel who change positions because of a transfer or promotion as well as personnel
2 assigned to short-term or temporary work assignments that may affect the quality of the WAP.
3 QAPjPs/procedures shall identify the responsible person(s) for ensuring that all personnel
4 maintain proficiency in the work performed and identify any additional training that may be
5 required.

6 C3-15 Changes to WAP-Related Plans or Procedures

7 Controlled changes to WAP-related plans or procedures shall be managed through the
8 document control process described in the QAPD. The Site Project Manager shall review all
9 non-administrative changes and evaluate whether those changes could impact DQOs specified
10 in the Permit. After site certification, any changes to WAP-related plans or procedures that could
11 positively or negatively impact DQOs (i.e., those changes that require prior approval of DOE as
12 defined in Attachment C5, Section C5-2) shall be reported to DOE within five days of
13 identification by the project level review. The Permittees shall send NMED a monthly summary
14 briefly describing the changes to plans and procedures identified pursuant to this section during
15 the previous month.

16 C3-16 List of References

17 DOE, 2009. Waste Data System User's Manual. DOE/WIPP 09-3427, Current Revision,
18 Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

19 DOE. 2003. Performance Demonstration Program Plan for the Analysis of Simulated
20 Headspace Gases. DOE/CAO-95-1076, Current Revision, Carlsbad, New Mexico, Carlsbad
21 Area Office, U.S. Department of Energy.

22 DOE. 2005. Performance Demonstration Program Plan for RCRA Constituent Analysis of
23 Solidified Wastes. DOE/CBFO-95-1077, Current Revision, Carlsbad, New Mexico, Carlsbad
24 Area Office, U.S. Department of Energy.

25 EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. SW-846,
26 Fourth Edition, Washington, D.C., Office of Solid Waste and Emergency Response, U.S.
27 Environmental Protection Agency.

28 Fisenne, I. M., et al. 1973. "Least Squares Analysis and Minimum Detection Levels Applied to
29 Multi-Component Alpha Emitting Samples." *Radiochem. Radioanal. Letters*, 16, No. 1: pp. 5-16.

30 Pasternack B. S. and N. H. Harley. 1971. "Detection Limits for Radionuclides in the Analysis of
31 Multi-Component Gamma-Spectrometric Data." *Nucl. Instr. and Meth*, No. 91: pp. 533-40.

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TABLES

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**Table C3-1
 Waste Material Parameters and Descriptions**

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

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**Table C3-2
Gas Volatile Organic Compounds Target Analyte List and Quality Assurance Objectives**

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

^a Criteria apply to PRQL concentrations.

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

^c These xylene isomers cannot be resolved by GC/MS.

^d The ethyl benzene PRQL for FTIRS is 20 ppm

CAS = Chemical Abstract Service

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

MDL = Method detection limit (maximum permissible value), for GC/MS and GC/FID; total number of nanograms delivered to the analytical system per sample (nanograms); for FTIRS based on 1 m sample cell

PRQL = Program required quantitation limit (parts per million/volume basis)

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Table C3-3
Summary of Laboratory Quality Control Samples and Frequencies for
Gas Volatile Organic Compound Analysis

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet method QAOs	Repeat until acceptable
Laboratory duplicates or on-line duplicates	One (1) per analytical batch or on-line batch	RPD \leq 25 ^b	Nonconformance if RPD >25
Laboratory blanks or on-line blanks	Daily prior to sample analysis for GC/MS and GC/FID. Otherwise, daily prior to sample analysis and one (1) per analytical batch or on-line	Analyte amounts \leq 3 \times MDLs for GC/MS and GC/FID; \leq PRQL for FTIRS	Flag Data if analyte amounts > 3 \times MDLs for GC/MS and GC/FID; > PRQL for FTIRS
Laboratory control samples or on-line control samples	One (1) per analytical batch or on-line batch	70-130 %R	Nonconformance if %R <70 or >130
GC/MS comparison sample (for FTIRS only)	One (1) per analytical or on-line batch	RPD \leq 25 ^b	Nonconformance if RPD > 25
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan
GC/MS	BFB Tune Every 12 hours	Abundance criteria for key ions are met	Repeat Until Acceptable
GC/MS	Minimum 5-point initial calibration (minimum of 5 standards) Initially and as needed	%RSD of response factor for each target analyte <35	Repeat Until Acceptable
GC/MS	Continuing calibration Every 12 hours	%D for all target analytes \leq 30 of initial calibration	Repeat Until Acceptable
GC/FID	Minimum 3-point initial calibration (minimum 3 standards) Initially and as needed	Correlation coefficient \geq 0.99 or %RSD <20 for each target analyte and the retention time of each target analyte within an acceptance criteria defined in the method	Repeat Until Acceptable
GC/FID	Continuing calibration Every 12 hours	%RSD \leq 15%	Repeat Until Acceptable

^a Corrective action per Section C3-13 when final reported QC samples do not meet the acceptance criteria.

^b Applies only to concentrations greater than the PRQLs listed in Table C3-2.

- MDL = Method Detection Limit
- QAO = Quality Assurance Objective
- PDP = Performance Demonstration Program
- PRQL = Program Required Quantitation Limit
- %R = Percent Recovery
- RPD = Relative Percent Difference
- BFB = 4-Bromofluorobenzene
- %D = Percent difference
- %RSD = Percent relative standard deviation

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**Table C3-4
Volatile Organic Compounds Target Analyte List and Quality Assurance Objectives**

Compound	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL ^b (mg/kg)	PRQL ^b (mg/kg)	Completeness (%)
Benzene	71-43-2	≤45	37-151	1	10	90
Bromoform	75-25-2	≤47	45-169	1	10	90
Carbon disulfide	75-15-0	≤50	60-150	1	10	90
Carbon tetrachloride	56-23-5	≤30	70-140	1	10	90
Chlorobenzene	108-90-7	≤38	37-160	1	10	90
Chloroform	67-66-3	≤44	51-138	1	10	90
1,4-Dichlorobenzene ^c	106-46-7	≤60	18-190	1	10	90
ortho-Dichlorobenzene ^c	95-50-1	≤60	18-190	1	10	90
1,2-Dichloroethane	107-06-2	≤42	49-155	1	10	90
1,1-Dichloroethylene	75-35-4	≤250	D-234 ^d	1	10	90
trans-1,2-Dichloroethylene	156-60-5	≤50	60-150	1	10	90
Ethyl benzene	100-41-4	≤43	37-162	1	10	90
Methylene chloride	75-09-2	≤50	D-221 ^d	1	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤55	46-157	1	10	90
Tetrachloroethylene	127-18-4	≤29	64-148	1	10	90
Toluene	108-88-3	≤29	47-150	1	10	90
1,1,1-Trichloroethane	71-55-6	≤33	52-162	1	10	90
1,1,2-Trichloroethane	79-00-5	≤38	52-150	1	10	90
Trichloroethylene	79-01-6	≤36	71-157	1	10	90
Trichlorofluoromethane	75-69-4	≤110	17-181	1	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤50	60-150	1	10	90
Vinyl chloride	75-01-4	≤200	D-251 ^d	1	4	90
m-xylene	108-38-3	≤50	60-150	1	10	90
o-xylene	95-47-6	≤50	60-150	1	10	90
p-xylene	106-42-3	≤50	60-150	1	10	90
Acetone	67-64-1	≤50	60-150	10 ^e	100	90
Butanol	71-36-3	≤50	60-150	10 ^e	100	90
Ethyl ether	60-29-7	≤50	60-150	10 ^e	100	90
Formaldehyde ^f	50-00-0	≤50	60-150	10 ^e	100	90
Hydrazine ^g	302-01-2	≤50	60-150	10 ^e	100	90
Isobutanol	78-83-1	≤50	60-150	10 ^e	100	90
Methanol	67-56-1	≤50	60-150	10 ^e	100	90
Methyl ethyl ketone	78-93-3	≤50	60-150	10 ^e	100	90
Pyridine ^c	110-86-1	≤50	60-150	10 ^e	100	90

^a Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

^b TCLP MDL and PRQL values are reported in units of mg/l and limits are reduced by a factor of 20.

^c Can also be analyzed as a semi-volatile organic compound. If analyzed as a semi-volatile compound, the QAOs of Table C3-6 apply.

^d Detected; result must be greater than zero.

- ^e Estimate, to be determined.
 - ^f Required only for homogeneous solids and soil/gravel waste from Savannah River Site, if analysis is required to resolve assignment of EPA hazardous waste numbers.
 - ^g Required only for homogeneous solids and soil/gravel waste from Oak Ridge National Laboratory and Savannah River Site, if analysis is required to resolve assignment of EPA hazardous waste numbers.
- CAS = Chemical Abstract Service
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
MD = Method detection limit (maximum permissible value) (milligrams per kilogram)
PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for benzene assuming a 0.9 oz (25-gram [g]) sample, 0.1 gal (0.5 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilogram)

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**Table C3-5
 Summary of Laboratory Quality Control Samples and
 Frequencies for Volatile Organic Compound Analysis**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table C3-4 QAOs	Repeat until acceptable
Laboratory duplicates ^b	One (1) per analytical batch	Meet Table C3-4 precision QAOs	Nonconformance if RPDs > values in Table C3-4
Laboratory blanks	One (1) per analytical batch	Analyte concentrations $\leq 3 \times$ MDLs	Nonconformance if analyte concentrations > $3 \times$ MDLs
Matrix spikes ^b	One (1) per analytical batch	Meet Table C3-4 accuracy QAOs	Nonconformance if %Rs are outside the range specified in Table C3-4
Matrix spike duplicates	One (1) per analytical batch	Meet Table C3-4 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table C3-4
Laboratory control samples	One (1) per analytical batch	Meet Table C3-4 accuracy QAOs	Nonconformance if %R < 80 or > 120
GC/MS Calibration	BFB Tune every 12 hours 5-pt. Initial Calibration initially, and as needed	Abundance criteria met as per method Calibrate according to SW-846 Method requirements: %RSD for CCC ≤ 30 , %RSD for all other compounds $\leq 15\%$ Average response factor (RRF) used if %RSD ≤ 15 , use linear regression if %RSD > 15; R or R ² ≥ 0.990 if using alternative curve System Performance Check Compound (SPCC) minimum RRF as per SW-846 Method; RRF for all other compounds ≥ 0.01	Repeat until acceptable
GC/MS Calibration (continued)	Continuing Calibration every 12 hours	%D ≤ 20 for CCC; SPCC minimum RRF as per SW-846 Method; RRF for all other compounds ≥ 0.01 RT for internal standard must be ± 30 seconds from last daily calibration, internal standard area count must be >50% and <200% of last daily calibration	Repeat until acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
GC/FID Calibration	3-pt. Initial Calibration initially and as needed Continuing Calibration every 12 hours	Correlation Coefficient \geq 0.990 or %RSD \leq 20 for all analytes %D or %Drift for all analytes \leq 15 of expected values, RT \pm 3 standard deviations from initial RT calibration per applicable SW-846 Method	Repeat until acceptable.
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples for a given matrix \pm 3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviation) or > (average %R + 3 standard deviation)
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^a Corrective Action per Section C3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

^b May be satisfied using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table C3-4.

MDL = Method detection limit

QAO = Quality assurance objective

PDP = Performance Demonstration Program

%R = Percent recovery

RPD = Relative percent difference

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**Table C3-6
Semi-Volatile Organic Compound Target Analyte List and Quality Assurance Objectives**

Compound	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL ^b (mg/kg)	PRQL ^b (mg/kg)	Completeness (%)
Cresols	1319-77-3	≤50	25-115	5	40	90
1,4-Dichlorobenzene ^{bc}	106-46-7	≤86	20-124	5	40	90
ortho-Dichlorobenzene ^c	95-50-1	≤64	32-129	5	40	90
2,4-Dinitrophenol	51-28-5	≤119	D-172 ^d	5	40	90
2,4-Dinitrotoluene	121-14-2	≤46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤319	D-152 ^d	0.3	2.6	90
Hexachloroethane	67-72-1	≤44	40-113	5	40	90
Nitrobenzene	98-95-3	≤72	35-180	5	40	90
Pentachlorophenol	87-86-5	≤128	14-176	5	40	90
Pyridine ^c	110-86-1	≤50	25-115	5	40	90

CAS = Chemical Abstract Service

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)

PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for nitrobenzene assuming a 100-gram (g) sample, 0.5 gal (2 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilograms)

^a Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

^b TCLP MDL and PRQL values are reported in units of mg/l and limits are reduced by a factor of 20.

^c Can also be analyzed as a volatile organic compound

^d Detected; result must be greater than zero

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**Table C3-7
 Summary of Laboratory Quality Control Samples and
 Frequencies for Semi-Volatile Organic Compounds Analysis**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table C3-6 QAOs	Repeat until acceptable
Laboratory duplicates ^b	One (1) per analytical batch	Meet Table C3-6 precision QAOs	Nonconformance if RPDs > values in Table C3-6
Laboratory blanks	One (1) per analytical batch	Analyte concentrations ≤ 3 × MDLs	Nonconformance if analyte concentrations > 3 × MDLs
Matrix spikes	One (1) per analytical batch	Meet Table C3-6 accuracy QAOs	Nonconformance if RPDs > values and %Rs outside range in Table C3-6
GC/MS Calibration	DFTPP Tune every 12 hours 5-pt. Initial Calibration initially, and as needed Continuing Calibration every 12 hours	Abundance criteria met as per method Calibrate according to SW-846 Method requirements: %RSD for CCC ≤ 30, %RSD for all other compounds ≤ 15% Average response factor (RRF) used if %RSD ≤ 15, use linear regression if >15; R or R ² ≥ 0.990 if using alternative curve System Performance Check Compound (SPCC) minimum RRF as per SW-846 Method; RRF for all other compounds ≥ 0.01 %D ≤ 20 for CCC, SPCC minimum RRF as per SW-846 Method; RRF for all other compounds ≥ 0.01 RT for internal standard must be ± 30 seconds from last daily calibration, internal standard area count must be >50% and <200% of last daily calibration	Repeat until acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
GC/ECD Calibration	5-pt. Calibration initially and as needed Continuing Calibration every 12 hours	Correlation Coefficient \geq 0.990 or %RSD < 20 for all analytes %D or %Drift for all analytes \leq 15 of expected values, RT \pm 3 standard deviations of initial RT calibration per applicable SW-846 Method	Repeat until acceptable
Matrix spike duplicates	One (1) per analytical batch	Meet Table C3-6 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table C3-6
Laboratory control samples	One (1) per analytical batch	Meet Table C3-6 accuracy QAOs	Nonconformance if %R < 80 or > 120
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples from a given matrix \pm 3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviations) or > (average %R + 3 standard deviations)
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^a Corrective action per Section C3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

^b May be satisfied by using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table C3-6.

- MDL = Method Detection Limit
- QAO = Quality Assurance Objective
- PDP = Performance Demonstration Program
- %R = Percent Recovery
- RPD = Relative Percent Difference

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**Table C3-8
Metals Target Analyte List and Quality Assurance Objectives**

Analyte	CAS Number	Precision (%RSD or RPD) ^a	Accuracy (%R) ^b	PRDL ^d (µg/L)	PRQL ^c (mg/kg)	Completeness (%)
Antimony	7440-36-0	≤30	80-120	100	100	90
Arsenic	7440-38-2	≤30	80-120	100	100	90
Barium	7440-39-3	≤30	80-120	2000	2000	90
Beryllium	7440-41-7	≤30	80-120	100	100	90
Cadmium	7440-43-9	≤30	80-120	20	20	90
Chromium	7440-47-3	≤30	80-120	100	100	90
Lead	7439-92-1	≤30	80-120	100	100	90
Mercury	7439-97-6	≤30	80-120	4.0	4.0	90
Nickel	7440-02-0	≤30	80-120	100	100	90
Selenium	7782-49-2	≤30	80-120	20	20	90
Silver	7440-22-4	≤30	80-120	100	100	90
Thallium	7440-28-0	≤30	80-120	100	100	90
Vanadium	7440-62-2	≤30	80-120	100	100	90
Zinc	7440-66-6	≤30	80-120	100	100	90

^a ≤ 30 percent control limits apply when sample and duplicate concentrations are ≥ 10 × IDL for ICP-AES and AA techniques, and ≥ 100 × IDL for Inductively Coupled Plasma—Mass Spectrometry (ICP-MS) techniques. If less than these limits, the absolute difference between the two values shall be less than or equal to the PRQL.

^b Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

^c TCLP PRQL values are reported in units of mg/l and limits are reduced by a factor of 20.

^d PRDL set such that it is a factor of 10 below the PRQL for 100 percent solid samples, assuming a 100× dilution during digestion.

CAS = Chemical Abstract Service

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

PRDL = Program required detection limit (i.e., maximum permissible value for IDL) (micrograms per liter)

PRQL = Program required quantitation limit (milligrams per kilogram)

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**Table C3-9
 Summary of Laboratory Quality Control Samples and Frequencies for Metals Analysis**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table C3-8 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	$\leq 3 \times \text{IDL}$ ($\leq 5 \times \text{IDL}$ for ICP-MS) ^b	Redigest and reanalyze any samples with analyte concentrations which are $\leq 10 \times$ blank value and $\geq 0.5 \times$ PRQL
Matrix spikes	One (1) per analytical batch	Meet Table C3-8 accuracy QAOs	Nonconformance if %R outside the range specified in Table C3-8
Matrix spike duplicates	One (1) per analytical batch	Meet Table C3-8 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table C3-8
ICP-MS Tune (ICP-MS Only)	Daily	4 Replicate %RSD ≤ 5 ; mass calibration within 0.9 amu; resolution < 1.0 amu full width at 10% peak height	Nonconformance if %RSD > 5; mass calibration > 0.9 amu; resolution > 1.0 amu
Initial Calibration 1 blank, 1 standard (ICP, ICP-MS) 3 standard, 1 blank (GFAA, FLAA) 5 standard, 1 blank (CVAA, HAA)	Daily	90-110 %R (80-120% for CVAA, GFAA, HAA, FLAA) for initial calibration verification solution. Regression coefficient ≥ 0.995 for FLAA, CVAA, GFAA, MAA	Correct problem and recalibrate; repeat initial calibration
Continuing Calibration	Every 10 samples and beginning and end of run	90-110% for continuing calibration verification solution. (80-120% for CVAA, GFAA, HAA, FLAA)	Correct problem and recalibrate; rerun last 10 samples
Internal Standard Area Verification (ICP-MS)	Every Sample	Meet SW-846 Method 6020 criteria	Nonconformance if not reanalyzed at 5 \times dilution until criteria are met
Serial Dilution (ICP, ICP-MS)	One (1) per analytical batch	5 \times dilution must be $\leq 10\%$ D of initial value for sample > 50 \times IDL	Flag Data if >10% and > 50 \times IDL
Interference Correction Verification (ICP, ICP-MS)	Beginning and end of run or every 12 hours (8 for ICP) whichever is more frequent	80-120% recovery for analytes Note: Acceptance Criteria and Corrective Action apply only if interferences found in samples at levels greater than ICS A Solution	Correct problem and recalibrate, nonconformance if not corrected

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Laboratory Control Samples	One (1) per analytical batch	Table C3-8 accuracy QAOs	Redigest and reanalyze for affected analytes; non conformance if not reanalyzed
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^a Corrective action per Section C3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

^b Applies only to concentrations greater than the PRQLs listed in Table C3-8.

- IDL = Instrument Detection Limit
- PDP = Performance Demonstration Program
- PRQL = Program Required Quantitation Limit
- %R = Percent Recovery
- RPD = Relative Percent Difference

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**Table C3-10
 Minimum Training and Qualifications Requirements ^a**

Personnel	Requirements ^a
Radiography Operators ^c	Site-specific training based on waste matrix codes and waste material parameters; requalification every 2 years
FTIRS Technical Supervisors ^b FTIRS Operators ^c	Site-specific and on-the-job training based on the site-specific FTIRS system; requalification every 2 years
Gas Chromatography Technical Supervisors ^b Gas Chromatography Operators ^c	B.S. or equivalent experience and 6 months previous applicable experience
Gas Chromatography/Mass Spectrometry Operators ^c Mass Spectrometry Operators ^c	B.S. or equivalent experience and 1 year independent spectral interpretation or demonstrated expertise
Gas Chromatography/Mass Spectrometry Technical Supervisors ^b Mass Spectrometry Technical Supervisors ^b Atomic Absorption Spectroscopy Technical Supervisors ^b Atomic Absorption Spectroscopy Operators ^c Atomic Mass Spectrometry Operators ^c Atomic Emission Spectroscopy Operators ^c	B.S. or equivalent experience and 1 year applicable experience
Atomic Mass Spectrometry Technical Supervisors ^b	B.S. and specialized training in Atomic Mass Spectrometry and 2 years applicable experience
Atomic Emission Spectroscopy Technical Supervisors ^b	B.S. and specialized training in Atomic Emission Spectroscopy and 2 years applicable experience.

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

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

^c Operators are those persons responsible for the actual operation of analytical equipment. QAPjPs shall include the site-specific title for this position.

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**Table C3-11
Testing Batch Data Report Contents**

Required Information	Radiography	Visual Examination	Comment
Batch Data Report Date	X	X	
Batch number	X	X	
Waste container number	X	X	
Waste stream name and/or number	O	O	
Waste Matrix Code	X	X	Summary Category Group included in waste matrix code
Implementing procedure (specific version used)	X	X	If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.
Container type	O	O	Drums, Standard Waste Box, Ten Drum Overpack, etc.
Video media reference	X	X	Reference to Video media applicable to each container. For visual examination of newly generated waste, video media not required if two trained operators review the contents of the waste container to ensure correct reporting.
Imaging check	O		
Camera check		O	
Audio check	O	O	
QC documentation	X	X	
Verification that the physical form matches the waste stream description and Waste Matrix Code.	X	X	Summary Category Group included in waste matrix code
Comments	X	X	
Reference to or copy of associated NCRs, if any	X	X	Copies of associated NCRs must be available.
Verify absence of prohibited items	X	X	
Operator signature and date of test	X	X	Signatures of both operators required for Visual Verification of Acceptable Knowledge
Data review checklists	X	X	All data review checklists will be identified

LEGEND:

X - Required in batch data report.

O - Information must be documented and traceable; inclusion in batch data report is optional.

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**Table C3-12
Sampling Batch Data Report Contents**

Required Information	Headspace Gas	Solid Sampling	Comment
Batch Data Report Date	X	X	
Batch number	X	X	
Waste stream name and/or number	O	O	
Waste Matrix Code		X	Summary Category Group included in Waste Matrix Code
Procedure (specific version used)	X	X	If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.
Container number	X	X	
Container type	O	O	Drums, Standard Waste Box, Ten Drum Overpack, etc.
Sample matrix and type	X	X	
Analyses requested and laboratory	X	X	
Point of origin for sampling	X	X	Location where sample was taken (e.g., building number, room)
Sample number	X	X	
Sample size	X	X	
Sample location	X	X	Location within container where sample is taken. (For HSG, specify what layer of confinement was sampled. For solids, physical location within container.)
Sample preservation	X	X	
Person collecting sample	X	X	
Person attaching custody seal	O	O	May or may not be the same as the person collecting the sample
Chain of custody record	X	X	Original or copy is allowed
Sampling equipment numbers	X	X	For disposable equipment, a reference to the lot

Required Information	Headspace Gas	Solid Sampling	Comment
Drum age	X		Must include all supporting determinative information, including but not limited to packaging date, equilibrium start time, storage temperature, and sampling date/time. If Scenario 3 is used, the packaging configuration, filter diffusivity, liner presence/absence, and rigid liner vent hole diameter used in determining the DAC must be documented. If Scenario 1 and 2 are used together, the filter diffusivity and rigid liner vent hole diameter used in determining the DAC must be documented. If default values are used for retrievably stored waste, these values must clearly be identified as such.
Cross-reference of sampling equipment numbers with associated cleaning batch numbers	O	X	As applicable to the equipment used for the sampling. For disposable equipment, a reference to the lot and procurement records to support cleanliness is sufficient
Drum age	X		
Equilibration time	X		
Verification of rigid liner venting	X		Only applicable to containers with rigid liners
Verification that sample volume taken is small in comparison to the available volume	X		Must include headspace gas volume when it can be estimated
Scale Calibration		O	
Depth of waste		X	For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a representative sample has been taken.
Calculation of core recovery		X	For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a representative sample has been taken.
Co-located core description		X	For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a QC sample has been taken.
Time between coring and subsampling		X	Only applicable to coring.
OVA calibration and reading	O		Only applicable to manifold systems. Must be done in accordance with manufacturer's specifications

Required Information	Headspace Gas	Solid Sampling	Comment
Field Records	X	X	Must contain the following as applicable to the sampling method used: Collection problems, Sequence of sampling collection, Inspection of the solids sampling area, Inspection of the solids sampling equipment, Coring tool test, random location of sub-sample, canister pressure, and ambient temperature and pressure.
Reference to or copy of associated NCRs, if any	X	X	Copies of associated NCRs must be available.
Operator Signature and date and time of sampling	X	X	
Data review checklists	X	X	All data review checklists will be identified

LEGEND:

X - Required in batch data report.

O - Information must be documented and traceable; inclusion in batch data report is optional.

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Table C3-13
Analytical Batch Data Report Contents

Required Information	Headspace Gas	Solid Sampling	Comment
Batch Data Report Date	X	X	
Batch number	X	X	
Sample numbers	X	X	
QC designation for sample	X	X	
Implementing procedure (specific version used)	X	X	If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.
QC sample results	X	X	
Sample data forms	X	X	Form should contain reduced data for target analytes and TICs
Chain of custody	X	X	Original or copy
Gas canister tags	X		Original or copy
Sample preservation	X	X	
Holding time		X	
Cross-reference of field numbers to laboratory sample numbers	X	X	
Date and time analyzed	X	X	
Verification of spectra used for results	O	O	Analyst must qualitatively evaluate the validity of the results based on the spectra, can be implemented as a check box for each sample
TIC evaluation	X	X	
Reporting flags, if any	X	X	Table C3-14 lists applicable flags
Case narrative	X	X	
Reference to or copy of associated NCRs, if any	X	X	Copies of associated NCRs must be available.
Operator signature and analysis date	X	X	
Data review checklists	X	X	All data review checklists will be identified

LEGEND:

X - Required in batch data report.

O - Information must be documented and traceable; inclusion in batch data report is optional.

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**Table C3-14
Data Reporting Flags**

Data Flag	Indicator
B	Analyte detected in blank (Organics/ Headspace gases)
B	Analyte blank concentration greater than or equal to 20 percent of sample concentration prior to dilution corrections (Metals)
E	Analyte exceeds calibration curve (Organics/ Headspace gases)
J	Analyte less than PRQL but greater than or equal to MDL (Organics/ Headspace gases)
J	Analyte greater than or equal to IDL but less than 5 times the IDL before dilution correction (Metals)
U	Analyte was not detected and value is reported as the MDL (IDL for Metals)
D	Analyte was quantitated from a secondary dilution, or reduced sample aliquot (Organics/ Headspace gases)
Z	One or more QC samples do not meet acceptance criteria
H	Holding time exceeded

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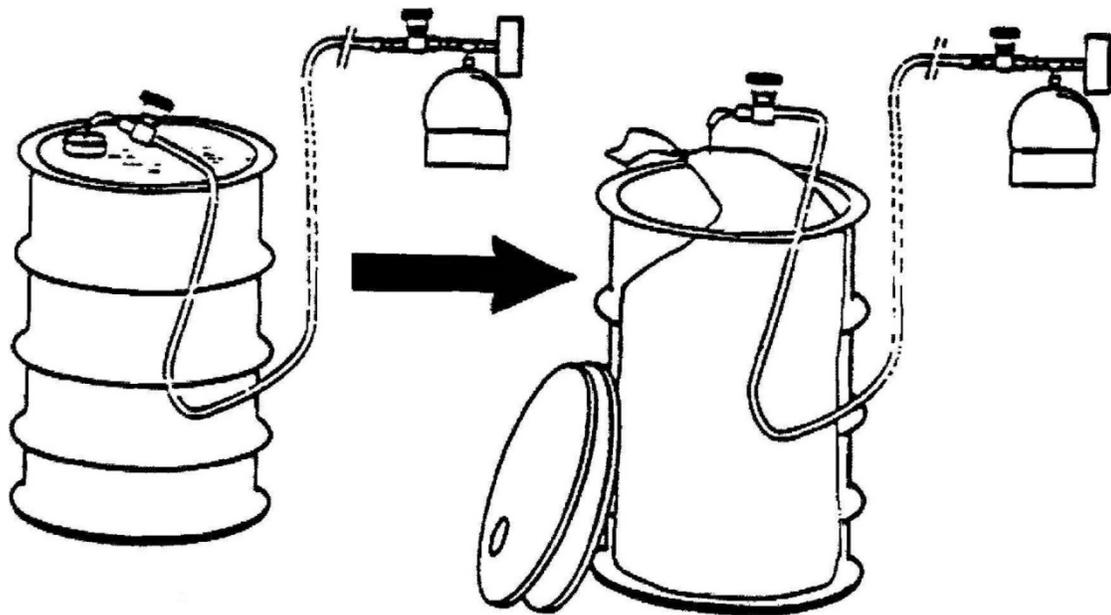


Figure C3-1
Overall Headspace-Gas Sampling Scheme Illustrating Manifold Sampling