

**ATTACHMENT B3**

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

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## ATTACHMENT B3

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

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## ATTACHMENT B3 QUALITY ASSURANCE OBJECTIVES FOR WASTE CHARACTERIZATION SAMPLING AND ANALYTICAL METHODS

### B3-1 Validation Methods

The Permittees shall require the generator/storage sites (**sites**) to perform validation of all data (qualitative as well as quantitative) so that data used for Waste Isolation Pilot Plant (**WIPP**) compliance programs will be of known and acceptable quality. Validation includes a quantitative determination of precision, accuracy, completeness, and method detection limits (as appropriate) for analytical data (headspace Volatile Organic Compounds (**VOC**), total VOCs, Semivolatile Organic Compounds (**SVOC**), and metals data). Quantitative data validations shall be performed according to the conventional methods outlined below (equations B3-1 through B3-8). These quantitative determinations will be compared to the Quality Assurance Objectives (**QAOs**) specified in Sections B3-2 through B3-9. A qualitative determination of comparability and representativeness will also be performed.

The qualitative data or descriptive information generated by radiography and visual examination is not amenable to statistical data quality analysis. However, radiography and visual examination are complementary techniques yielding similar data for determining the waste matrix code and waste material parameter weights of waste present in a waste container. Therefore, visual examination results shall be used to verify the waste matrix code and waste material parameter weights determined by radiography. The waste matrix code is determined and waste material parameter weights are estimated to verify that the container is properly included in the appropriate waste stream.

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

#### Precision

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

$$RPD = \frac{C_1 - C_2}{\frac{(C_1 + C_2)}{2}} \times 100 \quad (B3-1)$$

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

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

$$3 \quad \% RSD = \frac{s}{y_{mean}} \times 100 \quad (B3-2)$$

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:

$$6 \quad s = \sqrt{\frac{\sum_{j=1}^n (y_j - y_{mean})^2}{n - 1}} \quad (B3-3)$$

7 where  $y_j$  is the measured value of the  
8  $j$ th replicate sample analysis measurement, and  $n$  equals 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:

$$12 \quad \% D = \frac{|C_1 - C_2|}{C_1} \times 100 \quad (B3-4)$$

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:

$$19 \quad \% R = \frac{C_m}{C_{sm}} \times 100 \quad (B3-5)$$

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



1 
$$\% R = \frac{S + U}{C_{sc}} \times 100 \tag{B3-6}$$

2 where S is the measured concentration in the spiked aliquot, U is the measured concentration  
3 in the unspiked aliquot, and C<sub>sc</sub> is the actual concentration of the spike added.

4 Method Detection Limit

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

9 
$$MDL = t_{(n-1, 1-\alpha=0.99)} \times s \tag{B3-7}$$

10 where T<sub>(n-1, 1-α=0.99)</sub> is the t-distribution value appropriate to a 99 percent confidence level and a  
11 standard deviation estimate with n-1 degrees of freedom, n is the number of observations, and  
12 s is the standard deviation of replicate measurements.

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

14 
$$MDL = 3s \tag{B3-8}$$

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

19 Completeness

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

25 
$$\% C = \frac{V}{n} \times 100 \tag{B3-9}$$

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

## Comparability

Comparability is the degree to which one data set can be compared to another. Comparability of data generated at different sites will be assured through the use of standardized, approved testing, sampling, preservation, and analytical techniques and by meeting the QAOs specified in Sections B3-2 through B3-9.

The comparability of waste characterization data shall be ensured through the use of generator/storage site data usability criteria. The Permittees shall ensure that data usability criteria are consistently established and used by the generator/storage sites to assess the usability of analytical and testing data. The criteria shall address, as appropriate, the following:

- Definition or reference of criteria used to define and assign data qualifier flags based on Quality Assurance Objective results,
- Criteria for assessing the useability of data impacted by matrix interferences,
- Criteria for assessing the useability of data based upon positive and negative bias as indicated by quality control data, of data qualifiers, and qualifier flags,
- Criteria for assessing the useability of data due to
  - Severe matrix effects,
  - Misidentification of compounds,
  - Gross exceedance of holding times,
  - Failure to meet calibration or tune criteria
- Criteria for assessing the useability of data that does not meet minimum detection limit requirements.

The Permittees shall be responsible for evaluating generator/storage site data useability and shall assess implementation through the generator/storage site audit.

## Representativeness

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

Representativeness of waste containers from waste streams subjected to visual examination and homogeneous solids and soil/gravel sampling and analysis will be validated, through documentation, that a true random sample with an adequate population was collected. Since representativeness is a quality characteristic that expresses the degree to which a sample or group of samples represents the population being studied, the random selection of waste containers ensures representativeness on a Program level. The Permittees shall require the site Project Manager to document that the selected waste containers from within a waste stream were randomly selected. Sampling personnel shall verify that proper procedures are

1 followed to ensure that samples are representative of the waste contained in a particular waste  
2 container or a waste stream.

### 3 Nonconformance to Data Quality Objectives (DQOs)

4 For any non-administrative nonconformance related to applicable requirements specified in this  
5 Waste Analysis Plan (**WAP**) which are first identified at the site Project Manager signature  
6 release level (i.e., a failure to meet a data quality objective [**DQO**]), the Permittees shall receive  
7 written notification within five (5) calendar days of identification and shall also receive a  
8 nonconformance report within thirty (30) calendar days of identification of the incident. The  
9 Permittees shall require the generator/storage site to implement a corrective action which  
10 remedies the nonconformance prior to management, storage, or disposal of the waste at WIPP.  
11 The Permittees shall send NMED a monthly summary of nonconformances identified during the  
12 previous month, indicating the number of nonconformances received and the generator/storage  
13 sites responsible.

### 14 Identification of Tentatively Identified Compounds

15 In accordance with SW-846 convention, identification of compounds detected by gas  
16 chromatography/mass spectrometry methods that are not on the list of target analytes shall be  
17 reported. Both composited and individual container headspace gas, volatile analysis  
18 (TCLP/Totals), and semi-volatile (TCLP/Totals) shall be subject to tentatively identified  
19 compound (**TIC**) reporting. These TICs for GC/MS Methods are identified in accordance with  
20 the following SW-846 criteria:

- 21 ● Relative intensities of major ions in the reference spectrum (ions greater than 10% of  
22 the most abundant ion) should be present in the sample spectrum.
- 23 ● The relative intensities of the major ions should agree within  $\pm 20$  percent.
- 24 ● Molecular ions present in the reference spectrum should be present in the sample  
25 spectrum.
- 26 ● Ions present in the sample spectrum but not in the reference spectrum should be  
27 reviewed for possible background contamination or presence of coeluting compounds.
- 28 ● Ions present in the reference spectrum but not in the sample spectrum should be  
29 reviewed for possible subtraction from the sample spectrum because of background  
30 contamination or coeluting peaks.
- 31 ● The reference spectra used for identifying TICs shall include, at minimum, all of the  
32 available spectra for compounds that appear in the 20.4.1.200 NMAC (incorporating 40  
33 CFR Part 261) Appendix VIII list. The reference spectra may be limited to VOCs when  
34 analyzing headspace gas samples.
- 35 ● TICs for headspace gas analyses that are performed through FTIR analyses shall be  
36 identified in accordance with the specifications of SW-846 Method 8410.

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

- 3 ● a TIC in an individual container headspace gas or solids sample shall be  
4 reported in the analytical batch data report if the TIC meets the SW-846  
5 identification criteria listed above and is present with a minimum of 10% of the  
6 area of the nearest internal standard.
- 7 ● a TIC in a composited headspace gas sample that contains 2 to 5 individual  
8 container samples shall be reported in the analytical batch data report if the TIC  
9 meets the SW-846 identification criteria listed above and is present with a  
10 minimum of 2% of the area of the nearest internal standard.
- 11 ● a TIC in a composited headspace gas sample that contains 6 to 10 individual  
12 container samples shall be reported in the analytical batch data report if the TIC  
13 meets the SW-846 identification criteria listed above and is present with a  
14 minimum of 1% of the area of the nearest internal standard.
- 15 ● a TIC in a composited headspace gas sample that contains 11 to 20 individual  
16 container samples shall be reported in the analytical batch data report if the TIC  
17 meets the SW-846 identification criteria listed above and is present with a  
18 minimum of 0.5% of the area of the nearest internal standard.

19 TICs that meet the SW-846 identification criteria, are reported in 25 percent of all waste  
20 containers sampled from a given waste stream, and that appear in the 20.4.1.200 NMAC  
21 (incorporating 40 CFR §261) Appendix VIII list, will be compared to acceptable knowledge data  
22 to determine if the TIC is a listed waste in the waste stream. TICs identified through headspace  
23 gas analyses that meet the Appendix VIII list criteria and the 25 percent reporting criteria for a  
24 waste stream will be added to the headspace gas waste stream target list regardless of the  
25 hazardous waste listing associated with the waste stream. TICs reported from the Totals VOC  
26 or SVOC analyses may be excluded from the target analyte list for a waste stream if the TIC is  
27 a constituent in an F-listed waste whose presence is attributable to waste packaging materials  
28 or radiolytic degradation from acceptable knowledge documentation. If a listed waste  
29 constituent TIC cannot be attributed to waste packaging materials, radiolysis, or other origins,  
30 the constituent will be added to the target analyte list and new hazardous waste codes will be  
31 assigned, if appropriate. TICs subject to inclusion on the target analyte list that are toxicity  
32 characteristic parameters shall be added to the target analyte list regardless of origin because  
33 the hazardous waste designation for these codes is not based on source. However, for toxicity  
34 characteristic and non-toxic F003 constituents, the site may take concentration into account  
35 when assessing whether to add a hazardous waste code. If a target analyte list for a waste  
36 stream is expanded due to the presence of TICs, all samples collected from that waste stream  
37 will be analyzed for constituents on the expanded list.

1 B3-2 Headspace-Gas Sampling

2 Quality Assurance Objectives

3 Headspace-gas sampling will occur from the headspace within each drum of transuranic (TRU)  
4 mixed waste or randomly selected containers from waste streams that meet the conditions for  
5 reduced headspace gas sampling listed in Attachment B, Section B-3a(1).

6 The precision and accuracy of the drum headspace-gas sampling operations must be assessed  
7 by analyzing field QC headspace-gas samples. These samples must include equipment blanks,  
8 field reference standards, field blanks, and field duplicates. If the QAOs described below are  
9 not met, a nonconformance report must be prepared, submitted, and resolved (Section B3-13).

10 Precision

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

16 Accuracy

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

21 Field blanks must also be collected at a frequency of 1 field blank for every 20 drums or  
22 sampling batch sampled to assess possible contamination in the headspace gas sampling  
23 method. Equipment blanks must also be collected at a frequency of 1 equipment blank for each  
24 equipment cleaning batch to assess possible contamination in the equipment cleaning method.  
25 Corrective actions must be taken if the blank exceeds three times the MDLs listed for any of the  
26 compounds listed in Table B3-2.

27 Completeness

28 Sampling completeness shall be expressed as the number of valid samples collected as a  
29 percent of the total number of samples collected for each waste stream. The completeness can  
30 also be expressed as the number of valid samples collected as a percent of the total number of  
31 drums for each waste stream. A valid sample is defined as a sample collected in accordance  
32 with approved sampling methods and the drum was properly prepared for sampling (e.g., the  
33 polyliner was vented to the drum headspace). The Permittees shall require participating  
34 sampling facilities to achieve a minimum 90 percent completeness. The amount and type of  
35 data that may be lost during the headspace-gas sampling operation cannot be predicted in  
36 advance. The Permittees shall require the Site Project Quality Assurance (QA) Officer to  
37 evaluate the importance of any lost or contaminated headspace-gas samples and take  
38 corrective action as appropriate.

1 Comparability

2 Consistent use and application of uniform procedures and equipment, as specified in Permit  
3 Attachment B1 and application of data useability criteria, should ensure that headspace gas  
4 sampling operations are comparable when sampling headspace at the different sampling  
5 facilities. The Permittees shall require each site to take corrective actions if uniform procedures,  
6 equipment, or operations are not followed without approved and justified deviations. In addition,  
7 laboratories analyzing samples must successfully participate in the Performance Demonstration  
8 Program (**PDP**).

9 Representativeness

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

- 11 ● Selection of the correct DAC Scenario and waste packaging configuration and  
12 meeting DAC equilibrium times.
- 13 ● A sample canister cleaning and leak check after assembly
- 14 ● Sampling equipment cleaning or disposal after use
- 15 ● Sampling equipment leak check after sample collection
- 16 ● Use of sample canisters with passivated internal surfaces
- 17 ● Use of low-internal-volume sampling equipment
- 18 ● Collection of samples with a low-sample volume to available headspace volume  
19 ratio (less than 10 percent of the headspace when the headspace can be  
20 determined)
- 21 ● Careful and documented pressure regulation of all activities specified in  
22 Attachment B1, Section B1-1
- 23 ● Performance audits
- 24 ● Collection of equipment blanks, field reference standard, field blanks, and field  
25 duplicates at the specified frequencies.
- 26 ● Manifold pressure sensors and temperature sensors calibrated before initial use  
27 and annually using NIST, or equivalent standards.
- 28 ● OVA calibrated daily, prior to first use, or as necessary according to  
29 manufacturers specifications.

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

1 B3-3 Sampling of Homogenous Solids and Soils/Gravel

2 Quality Assurance Objectives

3 To ensure that sampling is conducted in a representative manner on a waste-stream basis for  
4 waste containers containing homogenous solids and soil/gravel, samples must be collected  
5 randomly in both the horizontal and vertical planes of each container's waste. For waste  
6 containers that contain homogenous solids and soil/gravel in smaller containers (e.g., 1 gal  
7 [4.0 L] poly bottles) within the waste container, one randomly chosen smaller container must be  
8 sampled from each drum.

9 Precision

10 Sampling precision must be determined by collecting and sampling field duplicates (e.g.,  
11 co-located cores or co-located samples as described in Permit Attachment B1-2b(1)) once per  
12 sampling batch or once per week during sampling operations, whichever is more frequent. A  
13 sampling batch is a suite of homogenous solids and soil/gravel samples collected consecutively  
14 using the same sampling equipment within a specific time period. A sampling batch can be up  
15 to 20 samples (excluding field QC samples), all of which must be collected within 14 days of the  
16 first sample in the batch. The Permittees shall require the site Project QA Officer to calculate  
17 and report the RPD between co-located core/samples.

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

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

34 Accuracy

35 Sampling accuracy through the use of standard reference materials shall not be measured.  
36 Because waste containers containing homogenous solids and soil/gravel with known quantities  
37 of analytes are not available, sampling accuracy cannot be determined. However, sampling  
38 methods and requirements described are designed to minimize sample degradation and hence  
39 maximize sampling accuracy.

1 Sampling accuracy as a function of sampling cross-contamination will be measured. Equipment  
2 blanks will be collected at a frequency of once per equipment cleaning batch. Corrective actions  
3 must be taken if the blank exceeds three times the MDLs (PRDLs for metals) listed for any of  
4 the compounds or analytes listed in Tables B3-4, B3-6, and B3-8. Equipment blanks will be  
5 collected from the following equipment types:

- 6 ● Fully assembled coring tools
- 7 ● Liners cleaned separately from coring tools
- 8 ● Miscellaneous sampling equipment that is reused (bowls, spoons, chisels)

### 9 Completeness

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

### 15 Comparability

16 Consistent use and application of uniform procedures, sampling equipment, and measurement  
17 units must ensure that sampling operations are comparable. Consistent application of data  
18 useability criteria will also ensure comparability. In addition, the Permittees shall require  
19 laboratories analyzing samples to successfully participate in the PDP.

### 20 Representativeness

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

- 23 ● Coring tools and sampling equipment must be clean prior to sampling.
- 24 ● The entire depth of the waste minus a site defined approved safety factor must  
25 be cored, and the core collected must have a length greater than or equal to 50  
26 percent of the depth of the waste. This is called the core recovery and is  
27 calculated as follows:

$$28 \text{ Core recovery (percent) } = \frac{y}{x} \times 100 \quad (\text{B3-10})$$

29 where

30 x = the depth of the waste in the container  
31 y = the length of the core collected from the waste.

- 32 ● Coring operations and tool selection should be designed to minimize alteration of  
33 the in-place waste characteristics. Minimal waste disturbance must be verified by



1 visually examining the core and describing the observation (e.g., undisturbed,  
2 cracked, or pulverized) in the field logbook.

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

6 One randomly selected container within a drum will be chosen if the drum contains  
7 individual waste containers.

#### 8 B3-4 Radiography

##### 9 Quality Assurance Objectives

10 The QAOs for radiography are detailed in this section. If the QAOs described below are not  
11 met, then corrective action shall be taken. It should be noted that radiography does not have a  
12 specific MDL because it is primarily a qualitative determination. The objective of radiography for  
13 the program is to verify the waste matrix code and identify prohibited items for each waste  
14 container and to estimate each waste material parameter weight (Table B3-1). The Permittees  
15 shall require each site to describe all activities required to achieve these objectives in the site  
16 quality assurance project plan (**QAPJP**) and standard operating procedures (**SOP**).

17 Data to meet these objectives must be obtained from an audio/videotaped (or equivalent media)  
18 scan provided by trained radiography operators at the sites. Results must also be recorded on a  
19 radiography data form. The precision, accuracy, completeness, and comparability objectives for  
20 radiography data are presented below.

##### 21 Precision

22 ~~The quantitative determination of the vent hole diameter is verified through confirmatory visual~~  
23 ~~examination and through replicate scan measurements. Because of the criticality of the vent~~  
24 ~~diameter in establishing DAC equilibrium times, the precision limit for a measurement is 0%~~  
25 ~~RPD as defined in Section B3-1.~~

26 The qualitative determinations, such as verifying the waste matrix code, made during  
27 radiography do not lend themselves to statistical evaluation of precision because of the  
28 qualitative nature of the inspection. However, comparison of data derived from radiography and  
29 visual examination on the same waste containers at the Rocky Flats Environmental Technology  
30 Site and the Idaho National Engineering Laboratory indicates that radiography operators can  
31 provide estimated inventories and weights of waste items in a waste container. As a measure of  
32 precision, the Permittees shall require each Site Project QA Officer to calculate and report the  
33 RPD between the estimated waste material parameter weights as determined by radiography  
34 and these same parameters as determined by visual examination. Additionally, the precision of  
35 radiography is verified prior to use by tuning precisely enough to demonstrate compliance with  
36 QAOs through viewing an image test pattern.

1 Accuracy

2 The programmatic accuracy at which the waste matrix code and waste material parameter  
3 weights can be determined must be documented through visual examination of a randomly  
4 selected statistical portion of waste containers. The Permittees shall require the Site Project QA  
5 Officer to calculate and report the miscertification rate of waste containers that require  
6 assignment to a different waste matrix code or are found to contain prohibited items after visual  
7 examination as a measure of radiography accuracy. The miscertification rate shall be used to  
8 determine the number of drums subject to confirmatory visual examination.

9 Completeness

10 An audio/videotape (or equivalent media) of the radiography examination and a validated  
11 radiography data form will be obtained for 100 percent of the retrievably stored waste  
12 containers in the program for all waste containers subject to radiography. All audio/videotapes  
13 (or equivalent media) and radiography data forms will be subject to validation as indicated in  
14 Section B3-10.

15 Comparability

16 The comparability of radiography data from different sites shall be enhanced by using  
17 standardized radiography procedures and operator qualifications.

18 B3-5 Gas Volatile Organic Compound Analysis

19 Quality Assurance Objectives

20 The development of DQOs specifically for this program has resulted in the QAOs listed in Table  
21 B3-2. The specified QAOs represent the required quality of data necessary to draw valid  
22 conclusions regarding program objectives. WAP-required limits, such as the program required  
23 quantitation limits (**PRQL**) associated with VOC analysis, are specified to ensure that the  
24 analytical data collected satisfy the requirements of all data users. A summary of the Quality  
25 Control Samples and the associated acceptance criteria is included in Table B3-3. Key data-  
26 quality indicators for laboratory measurements are defined below.

27 Precision

28 Precision shall be assessed by analyzing laboratory duplicates and replicate analyses of  
29 laboratory-control samples and PDP blind-audit samples. Results from measurements on these  
30 samples must be compared to the criteria listed in Table B3-2. These QC measurements will be  
31 used to demonstrate acceptable method performance and to trigger corrective action when  
32 control limits are exceeded.

33 Accuracy

34 Accuracy as %R shall be assessed for the laboratory operations by analyzing PDP blind-audit  
35 samples and laboratory-control samples. Results from these measurements must be compared  
36 to the criteria listed in Table B3-2. These QC measurements will be used to demonstrate

1 acceptable method performance and to trigger corrective action when control limits are  
2 exceeded.

### 3 Calibration

4 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated  
5 using the procedures and criteria specified in Table B3-3. These criteria will be used to  
6 demonstrate acceptable calibration and to trigger corrective action when control limits are  
7 exceeded.

### 8 Method Detection Limit

9 MDLs shall be expressed in nanograms for VOCs and must be less than or equal to those listed  
10 in Table B3-2. MDLs shall be determined based on the method described in Section B3-1. The  
11 detailed procedures for MDL determination shall be included in site SOPs.

### 12 Program Required Quantitation Limit

13 Laboratories must demonstrate the capability to quantitate analytes at or below the PRQLs  
14 given in Table B3-2. Laboratories shall set the concentration of at least one calibration standard  
15 below the PRQL. The detailed procedures for PRQL demonstration shall be included in  
16 laboratory SOPs.

### 17 Completeness

18 Laboratory completeness shall be expressed as the number of samples analyzed with valid  
19 results as a percent of the total number of samples submitted for analysis. A composited  
20 sample is treated as one sample for the purposes of completeness, because only one sample is  
21 run through the analytical instrument. Valid results are defined as results that meet the data  
22 useability criteria based on application of the Quality Control Criteria specified in Tables B3-2  
23 and B3-3; and meet the detection limit, calibration representativeness, and comparability criteria  
24 within this section. The Permittees shall require that participating laboratories meet the  
25 completeness criteria specified in Table B3-2.

### 26 Comparability

27 For VOC analysis, data generated through analysis of samples from different sites shall be  
28 comparable. The Permittees shall require each site to achieve comparability by using  
29 standardized methods and traceable standards and by requiring all sites to successfully  
30 participate in the PDP.

### 31 Representativeness

32 Representativeness for VOC analysis shall be achieved by collecting sufficient numbers of  
33 samples using clean sampling equipment that does not introduce sample bias. Samples must  
34 be collected as described in Permit Attachment B1.

1 B3-6 Total Volatile 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 B3-4. The specified QAOs represent the required quality of data necessary to draw valid  
5 conclusions regarding program objectives. WAP-required limits, such as the PRQL associated  
6 with VOC analysis, are specified to ensure that the analytical data collected satisfy the  
7 requirements of all data users. Key data-quality indicators for laboratory measurements are  
8 defined below.

9 Precision

10 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,  
11 replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from  
12 measurements on these samples must be compared to the criteria listed in Table B3-4. These  
13 QC measurements will be used to demonstrate acceptable method performance and to trigger  
14 corrective action when control limits are exceeded.

15 Accuracy

16 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control  
17 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from  
18 these measurements for matrix spikes samples must be compared to the %R criteria listed in  
19 Table B3-4. Results for surrogates and internal standards are evaluated as specified in the SW-  
20 846 method (EPA 1996) or Table B3-5. These QC measurements will be used to demonstrate  
21 acceptable method performance and to trigger corrective action when control limits are  
22 exceeded.

23 Laboratory blanks shall be assessed to determine possible laboratory contamination and are  
24 evaluated as specified in Table B3-5. These QC measurements will be used to demonstrate  
25 acceptable levels of laboratory contamination and to trigger corrective action when control limits  
26 are exceeded.

27 Calibration

28 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated  
29 using the procedures and criteria specified in Table B3-5 and the SW-846 method (EPA 1996).  
30 These criteria will be used to demonstrate acceptable calibration and to trigger corrective action  
31 when control limits are exceeded.

32 Method Detection Limit

33 MDLs shall be expressed in milligrams per kilogram (mg/kg) for VOCs and must be less than or  
34 equal to those listed in Table B3-4. The detailed procedures for MDL determination shall be  
35 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 B3-4. 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 useability criteria based upon application of the Quality  
10 Control Criteria specified in Tables B3-4 and B3-5 and meet the calibration, detection limit,  
11 representativeness, and comparability criteria within this section. Participating laboratories must  
12 meet the completeness criteria specified in Table B3-4.

13 Comparability

14 For VOC 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 B3-4 and  
17 B3-5, traceable standards, and by requiring all sites to successfully participate in the PDP.  
18 Generator/storage sites may use the most recent version of SW-846. Any changes to SW-846  
19 methodology that results in the elimination of sample preparation or analytical methods in use  
20 at generator/storage sites must be addressed as a corrective action to address the  
21 comparability of data before and after the SW-846 modification.

22 Representativeness

23 Representativeness for VOC analysis shall be achieved by collecting unbiased samples.  
24 Samples must be collected as described in Permit Attachment B1.

25 B3-7 Total Semivolatile Organic Compound Analysis

26 Quality Assurance Objectives

27 The development of DQOs specifically for this program has resulted in the QAOs listed in Table  
28 B3-6. The specified QAOs represent the required quality of data necessary to draw valid  
29 conclusions regarding program objectives. WAP-required limits, such as the PRQLs, are  
30 specified to ensure that the analytical data collected satisfy the requirements of all data users.  
31 A summary of Quality Control Samples and associated acceptance criteria for this analysis is  
32 included in Table B3-7. Key data-quality indicators for laboratory measurements are defined  
33 below.

34 Precision

35 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,  
36 replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from

1 measurements on these samples must be compared to the criteria listed in Table B3-6. These  
2 QC measurements will be used to demonstrate acceptable method performance and to trigger  
3 corrective action when control limits are exceeded.

#### 4 Accuracy

5 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control  
6 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from  
7 these measurements for matrix spikes samples must be compared to the %R criteria listed in  
8 Table B3-6. Results for surrogates and internal standards are evaluated as specified in the SW-  
9 846 method (EPA 1996) or Table B3-7. These QC measurements will be used to demonstrate  
10 acceptable method performance and to trigger corrective action when control limits are  
11 exceeded.

12 Laboratory blanks shall be assessed to determine possible laboratory contamination and are  
13 evaluated as specified in Table B3-7. These QC measurements will be used to demonstrate  
14 acceptable levels of laboratory contamination and to trigger corrective action when control limits  
15 are exceeded.

#### 16 Calibration

17 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated  
18 using the procedures and criteria specified in Table B3-7 and the SW-846 method (EPA 1996).  
19 These criteria will be used to demonstrate acceptable calibration and to trigger corrective action  
20 when control limits are exceeded.

#### 21 Method Detection Limit

22 MDLs shall be expressed in mg/kg for SVOCs and must be less than or equal to those listed in  
23 Table B3-6. The detailed procedures for MDL determination shall be included in site SOPs.

#### 24 Program Required Quantitation Limit

25 Laboratories must demonstrate the capability to quantitate analytes in samples at or below the  
26 PRQLs given in Table B3-6. Laboratories shall set the concentration of at least one calibration  
27 standard below the PRQL. The detailed procedures for PRQL demonstration shall be included  
28 in laboratory SOPs.

#### 29 Completeness

30 Laboratory completeness shall be expressed as the number of samples analyzed with valid  
31 results as a percent of the total number of samples submitted for analysis. Valid results are  
32 defined as results that meet the data useability criteria based on application of the Quality  
33 Control Criteria specified in Tables B3-6 and B3-7 and meet the detection limit, calibration,  
34 representativeness, and comparability criteria within this section. The Permittees shall require  
35 participating laboratories to meet the level of completeness specified in Table B3-6.

1 Comparability

2 For SVOC analysis, data generated through analysis of samples from different sites shall be  
3 comparable. The Permittees shall require sites to achieve comparability by using standardized  
4 SW-846 sample preparation and methods that meet the QAO requirements in Tables B3-6 and  
5 B3-7, traceable standards, and by requiring all sites to successfully participate in the PDP.  
6 Generator/storage sites may use the most current version of SW-846 if the methods are  
7 consistent with QAO requirements. Any changes to SW-846 methodology that results in the  
8 elimination of sample preparation or analytical methods in use at generator/storage sites must  
9 be addressed as a corrective action to address the comparability of data before and after the  
10 SW-846 modification.

11 Representativeness

12 Representativeness for SVOC analysis shall be achieved by collecting unbiased samples.  
13 Samples must be collected as described in Permit Attachment B1.

14 B3-8 Total Metal Analysis

15 Quality Assurance Objectives

16 The development of DQOs for the program has resulted in the QAOs listed in Table B3-8. The  
17 specified QAOs represent the required quality of data necessary to draw valid conclusions  
18 regarding program objectives. WAP-required limits, such as the PRQLs associated with metal  
19 analysis, are specified to ensure that the analytical data collected satisfy the requirements of all  
20 data users. A summary of Quality Control Samples and the associated acceptance criteria for  
21 this analysis is provided in Table B3-9. Key data-quality indicators for laboratory measurements  
22 are defined below.

23 Precision

24 Precision shall be assessed by analyzing laboratory sample duplicates or laboratory matrix  
25 spike duplicates, replicate analyses of laboratory-control samples, and PDP blind-audit  
26 samples. Results from measurements on these samples must be compared to the criteria listed  
27 in Table B3-8. These QC measurements will be used to demonstrate acceptable method  
28 performance and to trigger corrective action when control limits are exceeded.

29 Accuracy

30 Accuracy shall be assessed through the analysis of laboratory matrix spikes, PDP blind-audit  
31 samples, serial dilutions, interference check samples, and laboratory-control samples. Results  
32 from these measurements must be compared to the criterion listed in Table B3-8 and B3-9.  
33 These QC measurements will be used to demonstrate acceptable method performance and to  
34 trigger corrective action when control limits are exceeded.

35 Laboratory blanks and calibration blanks shall be assessed to determine possible laboratory  
36 contamination and are evaluated as specified in Table B3-9. These QC measurements will be

1 used to demonstrate acceptable levels of laboratory contamination and to trigger corrective  
2 action when control limits are exceeded.

### 3 Calibration

4 Mass Tunes (for ICP MS only), Standards Calibration, Initial Calibration verifications, and  
5 Continuing Calibrations will be performed and evaluated using the procedures and criteria  
6 specified in Table B3-9 and the SW-846 method (EPA 1996). These criteria will be used to  
7 demonstrate acceptable calibration and to trigger corrective action when control limits are  
8 exceeded.

### 9 Program Required Detection Limits

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

### 19 Program Required Quantitation Limit

20 The Permittees shall require participating laboratories to demonstrate the capability of analyte  
21 quantitation at or below the PRQLs in units of mg/kg wet weight (given in Table B3-8). The  
22 PRDLs are set an order of magnitude less than the PRQLs (assuming 100 percent solid sample  
23 diluted by a factor of 100 during preparation). The Permittees shall require participating  
24 laboratories to set the concentration of at least one QC or calibration standard at or below the  
25 solution concentration equivalent of the PRQL. Detailed calibration procedures shall be included  
26 in site 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. Valid results are  
30 defined as results that meet the data useability criteria based upon application of the Quality  
31 Control Criteria specified in Tables B3-8 and B3-9 and meet the detection limit, calibration,  
32 representativeness, and comparability criteria within this section. The Permittees shall require  
33 participating laboratories to meet the completeness specified in Table B3-8.

### 34 Comparability

35 For metals analysis, data generated through analysis of samples from different sites shall be  
36 comparable. Comparability will be achieved by using standardized SW-846 sample preparation  
37 and methods that meet QAO requirements in Tables B3-8 and B3-9, demonstrating successful  
38 participation in the PDP, and use of traceable standards. Generator/storage sites may use the



1 most recent SW-846 update. Any changes to SW-846 methodology that results in the  
2 elimination of sample preparation or analytical methods in use at generator/storage sites must  
3 be addressed as a corrective action to address the comparability of data before and after the  
4 SW-846 modification.

#### 5 Representativeness

6 Representativeness for metals analysis shall be achieved by the collection of unbiased samples  
7 and the preparation of samples in the laboratory using representative and unbiased methods.  
8 Samples must be collected as described in Permit Attachment B1.

#### 9 B3-9 Acceptable Knowledge

10 Acceptable knowledge documentation provides primarily qualitative information that cannot be  
11 assessed according to specific data quality goals that are used for analytical techniques. QAOs  
12 for analytical results are described in terms of precision, accuracy, completeness,  
13 comparability, and representativeness. Appropriate analytical and testing results will be used to  
14 confirm the characterization of wastes based on acceptable knowledge (Section B4-4 of  
15 Attachment B4). To ensure that the acceptable knowledge process is consistently applied, the  
16 Permittees shall require sites to comply with the following data quality requirements for  
17 acceptable knowledge documentation:

- 18 ● Precision - Precision is the agreement among a set of replicate measurements  
19 without assumption of the knowledge of a true value. The qualitative  
20 determinations, such as compiling and assessing acceptable knowledge  
21 documentation, do not lend themselves to statistical evaluations of precision.  
22 However, the acceptable knowledge information will be addressed by the  
23 independent review of acceptable knowledge information during internal and  
24 external audits.
- 25 ● Accuracy - Accuracy is the degree of agreement between an observed sample  
26 result and the true value. The percentage of waste containers which require  
27 reassignment to a new waste matrix code and/or designation of different  
28 hazardous waste codes based on the reevaluation of acceptable knowledge and  
29 sampling and analysis data will be reported as a measure of acceptable  
30 knowledge accuracy.
- 31 ● Completeness - Completeness is an assessment of the number of waste  
32 streams or number of samples collected to the number of samples determined to  
33 be useable through the data validation process. The acceptable knowledge  
34 record must contain 100 percent of the required information (Permit Attachment  
35 B4-3). The useability of the acceptable knowledge information will be assessed  
36 for completeness during audits.
- 37 ● Comparability - Data are considered comparable when one set of data can be  
38 compared to another set of data. Comparability is ensured through sites meeting  
39 the training requirements and complying with the minimum standards outlined for  
40 procedures that are used to implement the acceptable knowledge process. All

1 sites must assign hazardous waste codes in accordance with Permit Attachment  
2 B4-4 and provide this information regarding its waste to other sites who store or  
3 generate a similar waste stream.

- 4 ● Representativeness - Representativeness expresses the degree to which sample  
5 data accurately and precisely represent characteristics of a population.  
6 Representativeness is a qualitative parameter that will be satisfied by ensuring  
7 that the process of obtaining, evaluating, and documenting acceptable  
8 knowledge information is performed in accordance with the minimum standards  
9 established in Permit Attachment B4. Sites also must assess and document the  
10 limitations of the acceptable knowledge information used to assign hazardous  
11 waste codes (e.g., purpose and scope of information, date of publication, type  
12 and extent to which waste parameters are addressed).

13 The Permittees shall require each generator/storage site to comply with the nonconformance  
14 notification and reporting requirements of Section B3-1 if the results of confirmatory analytical  
15 techniques specified in Permit Attachment B are inconsistent with acceptable knowledge  
16 documentation.

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

#### 24 B3-10 Data Review, Validation, and Verification Requirements

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

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

- 36 ● A Testing Batch Data Report or equivalent includes all data pertaining to radiography or  
37 visual examination for up to 20 waste containers without regard to waste matrix. Table  
38 B3-11 lists all of the information required in Testing Batch Data Reports (identified with  
39 an "X") and other information that is necessary for data validation, but is optional in  
40 Testing Batch Data Reports (identified with an "O").

- 1 ● A Sampling Batch Data Report or equivalent includes all sample collection data  
2 pertaining to a group of no more than 20 headspace gas or homogeneous waste  
3 samples that were collected for chemical analysis. Table B3-12 lists all of the  
4 information required in Sampling Batch Data Reports (identified with an "X") and other  
5 information that is necessary for data validation, but is optional in Sampling Batch Data  
6 Reports (identified with an "O").
  
- 7 ● An Analytical Batch Data Report or equivalent includes analytical data from the analysis  
8 of TRU-mixed waste for up to 20 headspace gas or homogeneous waste samples.  
9 Analytical Batch Data Reports or equivalent that contain results for composited  
10 headspace gas samples must contain sufficient information to identify the containers  
11 that were composited for each composite sample and the sample volume that was taken  
12 from each waste container. Because Analytical Batch Data Reports are generated  
13 based on the number of samples analyzed, an Analytical Batch Data Report may  
14 contain results that are applicable to more than 20 containers depending on how many  
15 composite samples are part of the report, but may not exceed a total of 20 samples  
16 analyzed. Table B3-13 lists all of the information required in Analytical Batch Data  
17 Reports (identified with an "X") and other information that is necessary for data  
18 validation, but is optional in Analytical Batch Data Reports (identified with an "O").
  
- 19 Raw analytical data need not be included in Analytical Batch Data Reports, but must be  
20 maintained in the site project files and be readily available for review upon request. Raw  
21 data may include all analytical bench sheet and instrumentation readouts for all  
22 calibration standard results, sample data, QC samples, sample preparation conditions  
23 and logs, sample run logs, and all re-extraction, re-analysis, or dilution information  
24 pertaining to the individual samples. Raw data may also include calculation records and  
25 any qualitative or semi-quantitative data collected for a sample and that has been  
26 recorded on a bench sheet or in a log book.
  
- 27 ● An On-line Batch Data Report or equivalent contains the combined information from the  
28 Sampling Batch Data Report and Analytical Batch Data Report that is relevant to the on-  
29 line method used.

### 30 B3-10a Data Generation Level

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

- 33 ● All raw data shall be signed and dated in reproducible ink by the person  
34 generating it. Alternately, unalterable electronic signatures may be used.
  
- 35 ● All data must be recorded clearly, legibly, and accurately in field and laboratory  
36 records (bench sheets, logbooks), and include applicable sample identification  
37 numbers (for sampling and analytical labs).
  
- 38 ● All changes to original data must be lined out, initialed, and dated by the  
39 individual making the change. A justification for changing the original data may  
40 also be included. Original data must not be obliterated or otherwise disfigured so

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

- 3 ● All data must be transferred and reduced from field and laboratory records  
4 completely and accurately.
- 5 ● All field and laboratory records must be maintained as specified in Table B-7 of  
6 Attachment B.
- 7 ● Data must be organized into a standard format for reporting purposes (Batch  
8 Data Report), as outlined in specific sampling and analytical procedures.
- 9 ● All electronic and video data must be stored appropriately to ensure that waste  
10 container, sample, and associated QC data are readily retrievable.

11 Data review, validation, and verification at this level involves scrutiny and signature release from  
12 qualified independent technical reviewer(s)<sup>1</sup>, technical supervisors(s), and a QA representative,  
13 as specified below. Individuals conducting this data review, validation, and verification must use  
14 checklists that address all of the items included in this section. Checklists must contain or  
15 reference tables showing the results of sampling, analytical or on-line batch QC samples, if  
16 applicable. Checklists must reflect review of all QC samples and quality assurance objective  
17 categories in accordance with criteria established in Tables B3-2 through B3-9 (as applicable to  
18 the methods validated). Completed checklists must be forwarded Batch Data Reports to the  
19 project level. Analytical raw data must be available and reviewed by the data generation level  
20 reviewer. **The Site Project Manager or designee shall determine the validity of the drum age  
21 criteria (DAC) assignment made at the data generation level based upon an assessment of the  
22 data collection and evaluation necessary to make the assignment.**

### 23 B3-10a(1) Independent Technical Review

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

28 One hundred percent of the Batch Data Reports must receive an independent technical review.  
29 This review shall be performed by an individual other than the data generator who is qualified to  
30 have performed the initial work. The independent technical review must be performed as soon  
31 as practicably possible in order to determine and correct negative quality trends in the sampling  
32 or analytical process. However at a minimum, the independent technical review must be  
33 performed before any waste associated with the data reviewed is managed, stored, or disposed  
34 at WIPP. The reviewer(s) must release the data as evidenced by signature, and as a  
35 consequence ensure the following:

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<sup>1</sup>Independent technical review is performed by a competent individual who is not directly responsible for performing the work.

- 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  
3 in the proper units and correct number of significant figures.
  
- 4 ● Calculations have been verified by a valid calculation program, a spot check of  
5 verified calculation programs, and/or 100 percent check of all hand calculations.  
6 Values that are not verifiable to within rounding or significant difference  
7 discrepancies must be rectified prior to completion of independent technical  
8 review.
  
- 9 ● The data have been reviewed for transcription errors.
  
- 10 ● The testing, sampling, or analytical data QA documentation for Batch Data  
11 Reports is complete and includes, as applicable, raw data, DAC and equilibrium  
12 calculations and times, calculation records, chain-of-custody (COC) forms,  
13 calibration records (or references to an available calibration package), QC  
14 sample results, and copies or originals of gas canister sample tags. Corrective  
15 action will be taken to ensure that all Batch Data Reports are complete and  
16 include all necessary raw data prior to completion of the independent technical  
17 review.
  
- 18 ● QC sample results are within established control limits, and if not, the data have  
19 been appropriately qualified in accordance with data useability criteria. Data  
20 outside of established control limits will be qualified as appropriate, assigned an  
21 appropriate qualifier flag, discussed in the case narrative, and included as  
22 appropriate in calculations for completeness .
  
- 23 ● Reporting flags (Table B3-14) were assigned correctly.
  
- 24 ● Sample holding time and preservation requirements were met, or exceptions  
25 documented.
  
- 26 ● Radiography tapes have been reviewed (independent observation) on a waste  
27 container basis at a minimum of once per testing batch or once per day of  
28 operation, whichever is less frequent (Attachment B1, Section B1-3b(2)). The  
29 radiography tape will be reviewed against the data reported on the radiography  
30 form to ensure that the data are correct and complete.
  
- 31 ● Field sampling records are complete. Incomplete or incorrect field sampling  
32 records will be subject to resubmittal prior to completion of the independent  
33 technical review.

34 B3-10a(2) Technical Supervisor Review

35 The technical supervisor review ensures that the independent technical review was performed  
36 completely, that the Batch Data Report is complete, and verifies that the results are technically  
37 reasonable. This review validates and verifies that the characterization performed in this area is  
38 ready for QA office review.

1 One hundred percent of the batch data reports must receive technical supervisory signature  
2 release for each testing batch, sampling batch, analytical batch and on-line batch. The technical  
3 supervisory signature release must occur as soon as practicably possible after the independent  
4 technical review in order to determine and correct negative quality trends in the sampling or  
5 analytical process. However at a minimum, the technical supervisory signature release must be  
6 performed before any waste associated with the data reviewed is managed, stored, or disposed  
7 at WIPP. This release must ensure the following:

- 8 ● The data are technically reasonable based on the technique used.
- 9 ● All data have received independent technical review with the exception of  
10 radiography tapes, which shall receive periodic technical review as specified in  
11 Attachment B1, Section B1-3b(2).
- 12 ● The testing, sampling, or analytical data QA documentation for Batch Data  
13 Reports is complete and includes, as applicable, raw data, **DAC and equilibrium**  
14 **calculations and times**, calculation records, COC forms, calibration records, QC  
15 sample results, and original or copies of gas sample canister tags.
- 16 ● Sample holding time requirements were met, or exceptions documented.
- 17 ● Field sampling records are complete.

18 **B3-10a(3) QA Officer Review**

19 The data generation level QA review ensures that the Batch Data Report is complete, that QC  
20 checks meet the acceptance criteria, and that the appropriate QAOs have been met. This  
21 review verifies and validates that the characterization results meet the program QA/QC, that  
22 instrument performance criteria have been met, and that QAOs for the subject characterization  
23 area have been met.

24 The Permittees shall require for each site that one hundred percent of the Batch Data Reports  
25 receive QA officer (or designee) signature release. The QA Officer signature release must  
26 occur as soon as practicably possible after the technical supervisory signature release in order  
27 to determine and correct negative quality trends in the sampling or analytical process. However  
28 at a minimum, the QA Officer signature release must be performed before any waste  
29 associated with the data reviewed is managed, stored, or disposed at WIPP. This release must  
30 ensure the following:

- 31 ● Independent technical and technical supervisory reviews have been performed  
32 as evidenced by the appropriate signature releases.
- 33 ● The QA documentation for Batch Data Reports is complete as appropriate for  
34 the point of data generation.
- 35 ● Sampling and analytical QC checks have been properly performed. QC criteria  
36 that were not met are documented.

- QAOs have been met according to the methods outlined in Section B3-11.

#### B3-10b Project Level

Data validation and verification at this level involves scrutiny and signature release from the Site Project Manager (or designee) and the Site Project QA Officer (or designee). The Permittees shall require each site to meet the following minimum requirements for each waste container. Any nonconformance identified during this process shall be documented on a nonconformance report (Section B3-13).

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

#### B3-10b(1) Site Project QA Officer

The Site Project QA Officer review ensures that the Batch Data Reports received from the data generation level is complete, validates and verifies that the QC checks were done properly and meet program criteria, and ensures that the QAOs have been met.

One hundred percent of the Batch Data Reports must receive Site Project QA Officer signature release. The Site Project QA Officer signature release must occur as soon as practicably possible in order to determine and correct negative quality trends in the sampling or analytical process. However at a minimum, the Site Project QA Officer signature release must be performed before any waste associated with the data reviewed is managed, stored, or disposed at WIPP. This signature release must ensure the following:

- Batch Data Reports are complete and data are properly reported (i.e., data are reported in correct units, with correct significant figures, and with correct qualifying flags).
- Sampling batch QC checks (e.g., equipment blanks, field duplicates, field reference standards) were properly performed, and meet the established QAOs and are within established data useability criteria.
- Testing batch QC checks (e.g., replicate scans, measurement system checks) were properly performed. Radiography data are complete and acceptable based on evidence of videotape review of one waste container per day or once per testing batch, whichever is less frequent, as specified in B1-3b(2).
- Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix spikes, matrix spike duplicates, laboratory control samples) were properly performed and meet the established QAOs and are within established data useability criteria.

- 1           ●       On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates,  
2                   on-line control samples) were properly performed and meet the established  
3                   QAOs and are within established data useability criteria.
  
- 4           ●       Proper procedures were followed to ensure representative samples of  
5                   headspace gas and homogenous solids and soil/gravel were taken.

6       B3-10b(2) Site Project Manager

7       The Site Project Manager Review is the final validation that all of the data contained in Batch  
8       Data Reports have been properly reviewed as evidenced by signature release and completed  
9       checklists.

10       One hundred percent of the Batch Data Reports must have Site Project Manager signature  
11       release. The Site Project Manager signature release must occur as soon as practicably  
12       possible after the Site Project QA officer signature release in order to determine and correct  
13       negative quality trends in the sampling or analytical process. However at a minimum, the Site  
14       Project Manager signature release must be performed before any waste associated with the  
15       data reviewed is managed, stored, or disposed at WIPP. This signature release must ensure  
16       the following:

- 17           ●       Data generation level independent technical, technical supervisory, and QA  
18                   officer (or designee) review, validation, and verification have been performed as  
19                   evidenced by the completed review checklists and appropriate signature  
20                   releases.
  
- 21           ●       Batch data review checklists are complete.
  
- 22           ●       Batch Data Reports are complete and data are properly reported (e.g., data are  
23                   reported in the correct units, with the correct number of significant figures, and  
24                   with qualifying flags).
  
- 25           ●       Verify that data are within established data assessment criteria and meet all  
26                   applicable QAOs (Section B3-11).

27       B3-10b(3) Prepare Site Project QA Officer Summary and Data Validation Summary

28       To document the project-level validation and verification described above, the Permittees shall  
29       require each Site Project QA Officer (or designee) to prepare a Site Project QA Officer  
30       Summary and the Site Project Manager (or designee) to prepare a Data Validation Summary.  
31       These reports may be combined to eliminate redundancy, and may be included with the Site  
32       Project QA Officer and Site Project Manager checklists. The Site Project QA Officer Summary  
33       includes a validation checklist for each Batch Data Report. Checklists for the Site Project QA  
34       Officer Summary must be sufficiently detailed to validate all aspects of a Batch Data Report that  
35       affect data quality. The Data Validation Summary provides confirmation that, on a per waste  
36       container basis as evidenced by Batch Data Report reviews, all data have been validated in  
37       accordance with the site QAPjP. The Data Validation Summary must identify each Batch Data  
38       Report reviewed (including all waste container numbers), describe how the validation was



1 performed and whether or not problems were detected (e.g., nonconformance reports), and  
2 include a statement indicating that all data are acceptable. Summaries must include release  
3 signatures.

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

### 13 B3-10b(4) Prepare Waste Stream Characterization Package

14 In the event the Permittees request detailed information on a waste stream, the site will provide  
15 a Waste Stream Characterization Package. The Site Project Manager can require each  
16 characterization area, data generation level technical supervisor, and QA officer to assist in  
17 preparation and review of the Waste Stream Characterization Package (Section B3-12b(2)) as  
18 necessary to ensure the package will support the Site Project Manager's waste characterization  
19 determinations.

### 20 B3-10c Permittee Level

21 The final level of data verification occurs at the Permittee level and must, at a minimum, consist  
22 of an inventory check of the Batch Data Reports to verify completeness. The Permittees are  
23 responsible for the verification that Batch Data Reports include the following:

- 24 ● Project-level signature releases
- 25 ● Listing of all waste containers being presented in the report
- 26 ● Listing of all testing, sampling, and analytical batch numbers associated with  
27 each waste container being reported in the package
- 28 ● Analytical Batch Data Report case narratives
- 29 ● Site Project QA Officer Summary
- 30 ● Data Validation Summary
- 31 ● Complete summarized qualitative and quantitative data for all waste containers  
32 with data flags and qualifiers.

33 For each Waste Stream Profile Form (**WSPF**) submitted for approval, the Permittees must  
34 verify that each submittal (i.e., WSPF and Characterization Information Summary) is complete  
35 and notify the originating site in writing of the WSPF approval. The Permittees will maintain the

1 data as appropriate for use in the regulatory compliance programs. At a minimum, the  
2 verification must:

- 3 ● Ensure the correct assignment of the waste stream description, Waste Matrix  
4 Code Group, Summary Category Groups, and EPA hazardous waste codes
- 5 ● Reconcile data
- 6 ● Contain summarized results of characterization
- 7 ● Contain acceptable knowledge summary documentation
- 8 ● List the methods used for characterization

9 For subsequent shipments made after the initial WSPF approval, the verification will also  
10 include WWIS internal limit checks (Attachment B, Section B-4b(1)(i)).

### 11 B3-11 Reconciliation with Data Quality Objectives

12 Reconciling the results of waste testing and analysis with the DQOs provides a way to ensure  
13 that data will be of adequate quality to support the regulatory compliance programs.  
14 Reconciliation with the DQOs will take place at both the project level and the Permittees' level.  
15 At the project level, reconciliation will be performed by the Site Project Manager; at the  
16 Permittees' level, reconciliation will be performed as described below.

#### 17 B3-11a Reconciliation at the Project Level

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

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

- 31 ● Waste matrix code
- 32 ● Waste material parameter weights
- 33 ● If each waste container of waste contains TRU radioactive waste

- 1           ●       Mean concentrations,  $UCL_{90}$  for the mean concentrations, standard deviations,  
2                    and the number of samples collected for each VOC in the headspace gas of  
3                    waste containers in the waste stream
  
- 4           ●       The potential flammability of TRU waste headspace gases
  
- 5           ●       Mean concentrations,  $UCL_{90}$  for the mean concentrations, standard deviations,  
6                    and number of samples collected for VOCs, SVOCs, and metals in the waste  
7                    stream
  
- 8           ●       Whether the waste stream exhibits a toxicity characteristic (**TC**) under 40 CFR  
9                    Part 261, Subpart C
  
- 10          ●       Whether the waste stream can be classified as hazardous or nonhazardous at  
11                    the 90-percent confidence level
  
- 12          ●       Whether a sufficient number of waste containers have been visually examined  
13                    (as a QC check on radiography) to determine with a reasonable level of certainty  
14                    that the  $UCL_{90}$  for the miscertification rate is less than 14 percent
  
- 15          ●       Whether an appropriate packaging configuration and Drum Age Criteria (**DAC**)  
16                    were applied and documented in the headspace gas sampling documentation,  
17                    and whether the drum age was met prior to sampling.
  
- 18          ●       Whether all TICs were appropriately identified and reported in accordance with  
19                    the requirements of Section B3-1 prior to submittal of a WSPF for a waste  
20                    stream or waste stream lot.
  
- 21          ●       Whether the overall completeness, comparability, and representativeness QAOs  
22                    were met for each of the analytical and testing procedures as specified in  
23                    Sections B3-2 through B3-9 prior to submittal of a WSPF for a waste stream or  
24                    waste stream lot.
  
- 25          ●       Whether the PRQLs for all analyses were met prior to submittal of a WSPF for a  
26                    waste stream or waste stream lot.

27       If the Site Project Manager determines that insufficient data have been collected to make the  
28       determinations listed above, additional data collection efforts must be undertaken. The  
29       reconciliation of a waste stream shall be performed prior to submittal of WSPF for that waste  
30       stream. For subsequent shipments, data reconciliation is done on all containers or samples  
31       prior to shipment to WIPP. The Permittees shall not manage, store, or dispose TRU mixed  
32       waste at WIPP unless the Site Project Manager determines that the WAP-required waste  
33       parameters listed above have been met.

34       The statistical procedure presented in Permit Attachment B2 shall be used by participating Site  
35       Project Managers to evaluate and report waste characterization data from the analysis of  
36       homogeneous solids and soil/gravel. The procedure, which calculates  $UCL_{90}$  values, shall be  
37       used to assess compliance with the DQOs in Attachment B, Section B-4a(1) as well as with

1 RCRA regulations. The procedure must be applied to all laboratory analytical data for total  
2 VOCs, total SVOCs, and total metals. For RCRA regulatory compliance (40 CFR § 261.24),  
3 data from the analysis of the appropriate metals and organic compounds shall be expressed as  
4 toxicity characteristic leaching procedure (**TCLP**) values or results may also be compared to the  
5 TC levels expressed as total values. These total values will be considered the regulatory  
6 threshold limit (**RTL**) values for the WAP. RTL values are obtained by calculating the  
7 weight/weight concentration (in the solid) of a TC analyte that would give the regulatory  
8 weight/volume concentration (in the TCLP extract), assuming 100-percent analyte dissolution.

### 9 B3-11b Reconciliation at the Permittee Level

10 The Permittees must also ensure that data of sufficient type, quality, and quantity are collected  
11 to meet WAP DQOs. The Permittees will ensure sufficient data have been collected in  
12 accordance with Attachment B, Section B-4a(1) to determine the following:

- 13 ● The concentration of VOC constituents in the headspace in the total waste  
14 inventory has not exceeded the environment performance standards of  
15 20.4.1.500 NMAC (incorporating 40 CFR §264.601(c)) as specified in Module IV;
- 16 ● Whether waste streams proposed for disposal in WIPP have been adequately  
17 characterized; and
- 18 ● Whether data supports the information contained in the WIPP RCRA permit  
19 application

### 20 B3-12 Data Reporting Requirements

21 Data reporting requirements define the type of information and the method of transmittal for  
22 data transfer from the data generation level to the project level and from the project level to the  
23 Permittees.

#### 24 B3-12a Data Generation Level

25 Data shall be transmitted by hard copy or electronically (provided a hard copy is available on  
26 demand) from the data generation level to the project level. Transmitted data shall include all  
27 Batch Data Reports and data review checklists. The Batch Data Reports and checklists used  
28 must contain all of the information required by the testing, sampling, and analytical techniques  
29 described in Permit Attachments B1 through B6 , as well as the signature releases to document  
30 the review, validation, and verification as described in Section B3-10. All Batch Data Reports  
31 and checklists shall be in approved formats, as provided in site-specific documentation.

32 Batch Data Reports shall be forwarded to the site project office. Site QAPjPs shall specify the  
33 individual at the site project office who will receive these reports. After review by the Site Project  
34 QA Officer, all Batch Data Reports will be forwarded to the Site Project Manager. All Batch Data  
35 Reports shall be assigned serial numbers, and each page shall be numbered. The serial  
36 number used for Batch Data Reports can be the same as the testing, sampling, or analytical  
37 batch number.

1 QA documentation, including raw data, shall be maintained in either testing, sampling, and  
2 analytical facility files, or site project files for those facilities located on site in accordance with  
3 the document storage requirements of site approved site QAPjPs. Contract waste  
4 characterization facilities shall forward testing, sampling, and analytical QA documentation  
5 along with Batch Data Reports to the site project office for inclusion in site project files.

6 **B3-12b Project Level**

7 The site project office shall prepare a WSPF for each waste stream certified for shipment to  
8 WIPP based on information obtained from Batch Data Reports. In addition, the site project  
9 office must ensure that the Characterization Information Summary and the Waste Stream  
10 Characterization Package (when requested by the Permittees) are prepared as appropriate.  
11 The Site Project QA Officer must also verify these reports are consistent with information found  
12 in analytical batch reports. Summarized testing, sampling, and analytical data are included in  
13 the Characterization Information Summary. The contents of the WSPF, Characterization  
14 Information Summary, and Waste Stream Characterization Package are discussed in the  
15 following sections.

16 After approval of a WSPF and the associated Characterization Information Summary by the  
17 Permittees, the generator/storage site are required to maintain a cross reference of container  
18 identification numbers to each Batch Data Report.

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

21 **B3-12b(1) Waste Stream Profile Form**

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

- 23 ● Generator/storage site name
- 24 ● Generator/storage site EPA ID
- 25 ● Date of audit report approval by NMED (if obtained)
- 26 ● Original generator of waste stream
- 27 ● The Waste Stream WIPP Identification Number
- 28 ● Summary Category Group
- 29 ● Waste Matrix Code Group
- 30 ● Waste stream name
- 31 ● A description of the waste stream
- 32 ● Applicable EPA hazardous waste codes

- 1           ●       Applicable TRUCON codes
- 2           ●       A listing of acceptable knowledge documentation used to identify the waste
- 3           stream
- 4           ●       The waste characterization procedures used and the reference and date of the
- 5           procedure
- 6           ●       Certification signature of Site Project Manager, name, title, and date signed

7   B3-12b(2) Characterization Information Summary

8   The Characterization Information Summary shall include the following elements:

- 9           ●       Data reconciliation with DQOs
- 10          ●       Headspace gas summary data listing the identification numbers of samples used
- 11          in the statistical reduction, the maximum, mean, standard deviation, UCL<sub>90</sub>, RTL,
- 12          and associated EPA hazardous waste codes that must be applied to the waste
- 13          stream.
- 14          ●       Total metal, VOC, and SVOC analytical results for homogeneous solids and
- 15          soil/gravel (if applicable)
- 16          ●       TIC listing and evaluation, and verification that acceptable knowledge (**AK**) was
- 17          confirmed.
- 18          ●       Radiography and visual examination summary to document that all prohibited
- 19          items are absent in the waste and to confirm AK.
- 20          ●       A complete listing of all container identification numbers used to generate the
- 21          WSPF, cross-referenced to each Batch Data Report
- 22          ●       Complete AK summary, including stream name and number, point of generation,
- 23          waste stream volume (current and projected), generation dates, TRUCON
- 24          codes, Summary Category Group, Waste Matrix Code(s) and Waste Matrix
- 25          Code Group, other TWBIR information, waste stream description, areas of
- 26          operation, generating processes, RCRA determinations, radionuclide
- 27          information, all references used to generate the AK summary, and any other
- 28          information required by Permit Attachment B4, Section B4-2b.

29   B3-12b(3) Waste Stream Characterization Package

30   The Waste Stream Characterization Package includes the following information:

- 31          ●       Waste Stream Profile Form (WSPF, Section B3-12b(1))
- 32          ●       Accompanying Characterization Information Summary (Section B3-12b(2))

- 1           ●       Complete AK summary (Section B3-12b(2))
- 2           ●       Batch Data Reports supporting the confirmation of AK and any others requested
- 3                    by the Permittees
- 4           ●       Raw analytical data requested by the Permittees

#### 5   B3-12b(4) WIPP Waste Information System (WWIS) Data Reporting

6   The WWIS Data Dictionary includes all of the data fields, the field format and the limits  
7   associated with the data as established by this WAP. These data will be subjected to edit and  
8   limit checks that are performed automatically by the database, as defined in the *WIPP Waste*  
9   *Information System User's Manual for Use by Shippers/Generators* (DOE, 2001). If a container  
10   was part of a composite headspace gas sample, the analytical results from the composite  
11   sample must be assigned as the container headspace gas data results, including associated  
12   TICs, for every waste container associated with the composite sample.

13   The Permittees will coordinate the data transmission with each generator/storage site. Actual  
14   data transmission will use appropriate technology to ensure the integrity of the data  
15   transmissions. The Permittees will require sites with large waste inventories and large  
16   databases to populate a data structure provided by the Permittees that contains the required  
17   data dictionary fields that are appropriate for the waste stream (or waste streams) at that site.  
18   For example, totals analysis data will not be requested from sites that do not have  
19   homogeneous solids or soil/gravel waste. The Permittees will access this data via the Internet  
20   to ensure an efficient transfer of this data. Small quantity sites will be given a similar data  
21   structure by the Permittees that is tailored to their types of waste. Sites with very small  
22   quantities of waste will be provided with the ability to assemble the data interactively to this data  
23   structure on the WWIS.

#### 24   B3-13 Nonconformances

25   The Permittees shall require the status of work and the WAP activities at participating  
26   generator/storage sites to be monitored and controlled by the Site Project Manager and Site  
27   Project QA Officer. This monitoring and control shall include nonconformance identification,  
28   documentation, and reporting.

29   The nonconformances and corrective action processes specified in this section describe  
30   procedures between the Permittees and the generator/storage sites. The Permittees shall  
31   comply with the nonconformance requirements specified in Section B3-1 of this Permit  
32   Attachment.

#### 33   Nonconformances

34   Nonconformances are uncontrolled and unapproved deviations from an approved plan or  
35   procedure. Nonconforming items and activities are those that do not meet the WAP  
36   requirements, procurement document criteria, or approved work procedures. Nonconforming  
37   items shall be identified by marking, tagging, or segregating, and the affected  
38   generator/storage site(s) notified. The Permittees shall require participating sites reconcile and

1 correct nonconforming items as appropriate in accordance with the Permittees' Quality  
2 Assurance Program Description (**QAPD**). Disposition of nonconforming items shall be identified  
3 and documented. The QAPjPs shall identify the person(s) responsible for evaluating and  
4 dispositioning nonconforming items and shall include referenced procedures for handling them.

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

- 8 ● Project staff - during field operations, supervision of subcontractors, data  
9 validation and verification, and self-assessment
- 10 ● Laboratory staff - during the preparation for and performance of laboratory  
11 testing; calibration of equipment; QC activities; laboratory data review, validation,  
12 and verification; and self-assessment
- 13 ● QA personnel - during oversight activities or audits

14 A nonconformance report shall be prepared for each nonconformance identified. Each  
15 nonconformance report shall be initiated by the individual(s) identifying the nonconformance.  
16 The nonconformance report shall then be processed by knowledgeable and appropriate  
17 personnel. For this purpose, a nonconformance report including, or referencing as appropriate,  
18 results of laboratory analysis, QC tests, audit reports, internal memoranda, or letters shall be  
19 prepared. The nonconformance report must provide the following information:

- 20 ● Identification of the individual(s) identifying or originating the nonconformance
- 21 ● Description of the nonconformance
- 22 ● Method(s) or suggestions for correcting the nonconformance (corrective action)
- 23 ● Schedule for completing the corrective action
- 24 ● An indication of the potential ramifications and overall useability the data, if  
25 applicable
- 26 ● Any approval signatures specified in the site nonconformance procedures

27 The Permittees shall require the Site Project QA Officer to oversee the nonconformance report  
28 process and be responsible for developing a plan to identify and track all nonconformances and  
29 report this information to the Permittees. Documentation of nonconformances shall be made  
30 available to the Site Project Manager, who in turn is responsible for notifying project personnel  
31 of the nonconformance. Completion of the corrective action for nonconformances must be  
32 verified by the Site Project QA Officer.

33 The Permittees will receive written notification of all non-administrative nonconformances (i.e., a  
34 failure to meet a DQO) first identified during the Site Project Manager Review within five (5)  
35 days of identification. The Permittees will also receive a nonconformance report within thirty



1 (30) days of identification. The generator/storage site will implement a corrective action process  
2 and resolve the identified nonconformance prior to the Permittees management, storage, or  
3 disposal of TRU mixed waste at WIPP.

#### 4 Permittees' Corrective Action Process

5 The Permittees shall initiate a corrective action process when internal nonconformances and  
6 nonconformances at the generator/storage sites are identified. Activities and processes that do  
7 not meet requirements are documented as deficiencies.

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

- 10 ● The condition is documented on a Corrective Action Report (**CAR**) by the  
11 individual identifying the problem.
- 12 ● The Permittees have designated the CAR Initiator and Assessment Team  
13 Leader to review the CAR, determine validity of the finding (determine that a  
14 requirement has been violated), classify the significance of the condition, assign  
15 a response due date, and issue the CAR to the responsible party.
- 16 ● The responsible organization reviews the CAR, evaluates the extent and cause  
17 of the deficiency and provides a response to the Permittees, indicating remedial  
18 actions and actions to preclude recurrence that will be taken.
- 19 ● The Permittees review the response from the responsible organization and, if  
20 acceptable, communicate the acceptance to the responsible organization.
- 21 ● The responsible organization completes remedial actions and actions to preclude  
22 recurrence of the condition.
- 23 ● After all corrective actions have been completed, the Permittees schedule and  
24 perform a verification to assure that corrective actions have been completed and  
25 are effective. When all actions have been completed and verified as being  
26 effective, the CAR is closed by the CAR Initiator and Assessment Team Leader  
27 on behalf of the Permittees.
- 28 ● As part of the planning process for subsequent audits and surveillances, past  
29 deficiencies are reviewed and the previous deficient activity or process is subject  
30 to reassessment.

#### 31 B3-14 Special Training Requirements and Certifications

32 Before performing activities that affect WAP quality, all personnel are required to receive  
33 indoctrination into the applicable scope, purpose, and objectives of the WAP and the specific  
34 QAOs of the assigned task. Personnel assigned to perform activities for the WAP shall have  
35 the education, experience, and training applicable to the functions associated with the work.  
36 Evidence of personnel proficiency and demonstration of competence in the task(s) assigned

1 must be demonstrated and documented. All personnel designated to work on specific aspects  
2 of the WAP shall maintain qualification (i.e., training and certification) throughout the duration of  
3 the work as specified in this WAP and applicable QAPjPs/procedures. Job performance shall  
4 be evaluated and documented at periodic intervals, as specified in the implementing  
5 procedures.

6 Personnel involved in WAP activities shall receive continuing training to ensure that job  
7 proficiency is maintained. Training includes both education in principles and enhancement of  
8 skills. Each participating site shall include in its QAPjP a description of the procedures for  
9 implementing personnel qualification and training. All training records that specify the scope of  
10 the training, the date of completion, and documentation of job proficiency shall be maintained  
11 as QA Records in the site project file.

12 Analytical laboratory line management must ensure that analytical personnel are qualified to  
13 perform the analytical method(s) for which they are responsible. The minimum qualifications for  
14 certain specified positions for the WAP are summarized in Table B3-10. QAPjPs, or their  
15 implementing SOPs, shall specify the site-specific titles and minimum training and qualification  
16 requirements for personnel performing WAP activities. QAPjPs/procedures shall also contain  
17 the requirements for maintaining records of the qualification, training, and demonstrations of  
18 proficiency by these personnel.

19 An evaluation of personnel qualifications shall include comparing and evaluating the  
20 requirements specified in the job/position description and the skills, training, and experience  
21 included in the current resume of the person. This evaluation also must be performed for  
22 personnel who change positions because of a transfer or promotion as well as personnel  
23 assigned to short-term or temporary work assignments that may affect the quality of the WAP.  
24 QAPjPs/procedures shall identify the responsible person(s) for ensuring that all personnel  
25 maintain proficiency in the work performed and identify any additional training that may be  
26 required.

### 27 B3-15 Changes to WAP-Related Plans or Procedures

28 Controlled changes to WAP-related plans or procedures shall be managed through the  
29 document control process described in the QAPD. The Site Project Manager and the Site  
30 Project QA Officer shall review all non-administrative changes and evaluate whether those  
31 changes could impact DQOs specified in the Permit. After site certification, any changes to  
32 WAP-related plans or procedures that could positively or negatively impact DQOs (i.e., those  
33 changes that require prior approval of the Permittees as defined in Attachment B5, Section B5-  
34 2) shall be reported to the Permittees within five (5) days of identification by the project level  
35 review. The Permittees shall send NMED a monthly summary briefly describing the changes to  
36 plans and procedures identified pursuant to this section during the previous month.

### 37 B3-16 List of References

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## **TABLES**

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**TABLE B3-1  
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

**TABLE B3-2  
GAS VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST  
AND QUALITY ASSURANCE OBJECTIVES**

Compound	CAS Number	Precision <sup>a</sup> (%RSD or RPD)	Accuracy <sup>a</sup> (%R)	MDL <sup>b,f</sup> (ng)	FTIRS MDL <sup>b</sup> (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
cis-1,2-Dichloroethylene	156-59-2	#25	70-130	10	5	10	90
trans-1,2-Dichloroethylene	156-60-5	#25	70-130	10	5	10	90
Ethyl benzene <sup>f</sup>	100-41-4	#25	70-130	10	10	10	90
Ethyl ether	60-29-7	#25	70-130	10	5	10	90
Formaldehyde <sup>c</sup>	50-00-0	#25	70-130	10	10	10	90
Hydrazine <sup>d</sup>	302-01-2	#25	70-130	10	10	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 <sup>e</sup>	108-38-3	#25	70-130	10	5	10	90
o-Xylene	95-47-6	#25	70-130	10	5	10	90
p-Xylene <sup>e</sup>	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

<sup>a</sup> Criteria apply to PRQL concentrations.

<sup>b</sup> Values based on delivering 10 mL to the analytical system.

<sup>c</sup> Required only for homogenous solids and soil/gravel waste from Los Alamos National Laboratory and Savannah River Site.

<sup>d</sup> Required only for homogenous solids and soil/gravel waste from Oak Ridge National Laboratory and Savannah River Site.

<sup>e</sup> These xylene isomers cannot be resolved by GC/MS.

<sup>f</sup> 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)



**TABLE B3-3  
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND  
FREQUENCIES FOR  
GAS VOLATILE ORGANIC COMPOUND ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
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 # 25 <sup>b</sup>	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 # 3 x MDLs for GC/MS and GC/FID; # PRQL for FTIRS	Flag Data if analyte amounts > 3 x 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 # 25 <sup>b</sup>	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

<sup>a</sup> Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.

<sup>b</sup> Applies only to concentrations greater than the PRQLs listed in Table B3-2.

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

**TABLE B3-4  
VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST  
AND QUALITY ASSURANCE OBJECTIVES**

Compound	CAS Number	Precision <sup>a</sup> (%RSD or RPD)	Accuracy <sup>a</sup> (%R)	MDL <sup>b</sup> (mg/kg)	PRQL <sup>b</sup> (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 <sup>c</sup>	106-46-7	#60	18-190	1	10	90
ortho-Dichlorobenzene <sup>c</sup>	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 <sup>d</sup>	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 <sup>d</sup>	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 <sup>d</sup>	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 <sup>e</sup>	100	90
Butanol	71-36-3	#50	60-150	10 <sup>e</sup>	100	90
Ethyl ether	60-29-7	#50	60-150	10 <sup>e</sup>	100	90
Formaldehyde <sup>f</sup>	50-00-0	#50	60-150	10 <sup>e</sup>	100	90
Hydrazine <sup>g</sup>	302-01-2	#50	60-150	10 <sup>e</sup>	100	90
Isobutanol	78-83-1	#50	60-150	10 <sup>e</sup>	100	90
Methanol	67-56-1	#50	60-150	10 <sup>e</sup>	100	90
Methyl ethyl ketone	78-93-3	#50	60-150	10 <sup>e</sup>	100	90
Pyridine <sup>c</sup>	110-86-1	#50	60-150	10 <sup>e</sup>	100	90

<sup>a</sup> 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.

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

<sup>c</sup> Can also be analyzed as a semi-volatile organic compound. If analyzed as a semi-volatile compound, the QAOs of Table B3-6 apply.

<sup>d</sup> Detected; result must be greater than zero.

<sup>e</sup> Estimate, to be determined.

<sup>f</sup> Required only for homogenous solids and soil/gravel waste from Los Alamos National Laboratory and Savannah River Site.

<sup>g</sup> Required only for homogenous solids and soil/gravel waste from Oak Ridge National Laboratory and Savannah River Site.

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 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)

**TABLE B3-5  
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND  
FREQUENCIES FOR VOLATILE ORGANIC COMPOUND ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-4 QAOs	Repeat until acceptable
Laboratory duplicates <sup>b</sup>	One (1) per analytical batch	Meet Table B3-4 precision QAOs	Nonconformance if RPDs > values in Table B3-4
Laboratory blanks	One (1) per analytical batch	Analyte concentrations # 3 x MDLs	Nonconformance if analyte concentrations > 3 x MDLs
Matrix spikes <sup>b</sup>	One (1) per analytical batch	Meet Table B3-4 accuracy QAOs	Nonconformance if %Rs are outside the range specified in Table B3-4
Matrix spike duplicates	One (1) per analytical batch	Meet Table B3-4 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table B3-4
Laboratory control samples	One (1) per analytical batch	Meet Table B3-4 accuracy QAO's	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 # 15%  Average response factor (RRF) used if %RSD # 15, use linear regression if %RSD >15; R or R <sup>2</sup> \$ 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

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
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 $\pm 30$ seconds from last daily calibration, internal standard area count must be >50% and <200% of last daily calibration	Repeat until acceptable
GC/FID Calibration	3-pt. Initial Calibration initially and as needed  Continuing Calibration every 12 hours	Correlation Coefficient \$ 0.990 or %RSD # 20 for all analytes  %D or %Drift for all analytes # 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

<sup>a</sup> Corrective Action per section B3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

<sup>b</sup> May be satisfied using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table B3-4.

MDL = Method detection limit  
QAO = Quality assurance objective  
PDP = Performance Demonstration Program  
%R = Percent recovery  
RPD = Relative percent difference

**TABLE B3-6  
SEMI-VOLATILE ORGANIC COMPOUND TARGET ANALYTE LIST  
AND QUALITY ASSURANCE OBJECTIVES**

Compound	CAS Number	Precision <sup>a</sup> (%RSD or RPD)	Accuracy <sup>a</sup> (%R)	MDL <sup>b</sup> (mg/kg)	PRQL <sup>b</sup> (mg/kg)	Completeness (%)
Cresols	1319-77-3	#50	25-115	5	40	90
1,4-Dichlorobenzene <sup>bc</sup>	106-46-7	#86	20-124	5	40	90
ortho-Dichlorobenzene <sup>c</sup>	95-50-1	#64	32-129	5	40	90
2,4-Dinitrophenol	51-28-5	#119	D-172 <sup>e</sup>	5	40	90
2,4-Dinitrotoluene	121-14-2	#46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	#319	D-152 <sup>e</sup>	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
Polychlorinated Biphenyls				5	40	90
Aroclor 1016 <sup>d</sup>	12674-11-2	#33	50-114	5	40	90
Aroclor 1221 <sup>d</sup>	11104-28-2	#110	15-178	5	40	90
Aroclor 1232 <sup>d</sup>	11141-16-5	#128	10-215	5	40	90
Aroclor 1242 <sup>d</sup>	53469-21-9	#49	39-150	5	40	90
Aroclor 1248 <sup>d</sup>	12672-29-6	#55	38-158	5	40	90
Aroclor 1254 <sup>d</sup>	11097-69-1	#62	29-131	5	40	90
Aroclor 1260 <sup>d</sup>	11096-82-5	#56	8-127	5	40	90
Pentachlorophenol	87-86-5	#128	14-176	5	40	90
Pyridine <sup>c</sup>	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)

<sup>a</sup> 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.

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

<sup>c</sup> Can also be analyzed as a volatile organic compound

<sup>d</sup> Required only for waste matrix code S3220 (organic sludges)

<sup>e</sup> Detected; result must be greater than zero

**TABLE B3-7  
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND  
FREQUENCIES FOR SEMI-VOLATILE ORGANIC COMPOUNDS ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-6 QAOs	Repeat until acceptable
Laboratory duplicates <sup>b</sup>	One (1) per analytical batch	Meet Table B3-6 precision QAOs	Nonconformance if RPDs > values in Table B3-6
Laboratory blanks	One (1) per analytical batch	Analyte concentrations # 3 x MDLs	Nonconformance if analyte concentrations > 3 x MDLs
Matrix spikes	One (1) per analytical batch	Meet Table B3-6 accuracy QAOs	Nonconformance if RPDs > values and %Rs outside range in Table B3-6
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 <sup>2</sup> \$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 <sup>a</sup>
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 # 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 B3-6 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table B3-6
Laboratory control samples	One (1) per analytical batch	Meet Table B3-6 accuracy QAO's	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

<sup>a</sup> Corrective action per section B3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

<sup>b</sup> May be satisfied by using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table B3-6.

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

**TABLE B3-8  
METALS TARGET ANALYTE LIST  
AND QUALITY ASSURANCE OBJECTIVES**

Analyte	CAS Number	Precision (%RSD or RPD) <sup>a</sup>	Accuracy (%R) <sup>b</sup>	PRDL <sup>d</sup> (Fg/L)	PRQL <sup>c</sup> (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

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

<sup>b</sup> 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.

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

<sup>d</sup> PRDL set such that it is a factor of 10 below the PRQL for 100 percent solid samples, assuming a 100x 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)



**TABLE B3-9  
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND  
FREQUENCIES FOR METALS ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-8 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	# 3 x IDL (# 5 x IDL for ICP-MS) <sup>b</sup>	Redigest and reanalyze any samples with analyte concentrations which are #10 x blank value and \$ 0.5 x PRQL
Matrix spikes	One (1) per analytical batch	Meet Table B3-8 accuracy QAOs	Nonconformance if %R outside the range specified in Table B3-8
Matrix spike duplicates	One (1) per analytical batch	Meet Table B3-8 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table B3-8
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, CVA, 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 X dilution until criteria are met

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Serial Dilution (ICP, ICP-MS)	One (1) per analytical batch	5 X dilution must be #10% D of initial value for sample > 50xIDL	Flag Data if >10% and > 50xIDL
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
Laboratory Control Samples	One (1) per analytical batch	Table B3-8 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

<sup>a</sup> Corrective action per section B3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

<sup>b</sup> Applies only to concentrations greater than the PRQLs listed in Table B3-8.

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

**TABLE B3-10  
MINIMUM TRAINING AND QUALIFICATIONS REQUIREMENTS <sup>a</sup>**

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

<sup>a</sup> 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).

<sup>b</sup> 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.

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

**TABLE B3-11  
TESTING BATCH DATA REPORT CONTENTS**

Required Information	Radiography	Visual Examination as QC Check on Radiography	Visual Verification of Acceptable Knowledge	Comment
Batch Data Report Date	X	X	X	
Batch number	X	X	X	
Waste container number	X	X	X	
Waste stream name and/or number	O	O	O	
Waste Matrix Code	X	X	X	Summary Category Group included in waste matrix code
Implementing procedure (specific version used)	X	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	O	Drums, Standard Waste Box, Ten Drum Overpack, etc.
Videotape reference	X	X		Reference to Videotape(s) applicable to each container. For visual examination (for characterization) of newly generated waste, videotape 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 check of scales		O	O	Available documented evidence calibrated scale(s) were used. Only applicable if items are weighed during the visual examination.
QC documentation	X	X	X	
Description of liners and layers of confinement (if possible)	X	X	X	
Indication of vented rigid liners	X	X	X	Only required for containers with rigid liners. If radiography is used to verify, then include in Testing Batch Data Report.
Description of container contents	X	X	X	Provide enough detail to identify all discernible waste items, etc., and to verify estimated weights for the 12 waste matrix material parameters.

Required Information	Radiography	Visual Examination as QC Check on Radiography	Visual Verification of Acceptable Knowledge	Comment
Verification that the physical form matches the waste stream description and Waste Matrix Code.	X	X	X	Summary Category Group included in waste matrix code
Indication of sealed containers > 4L	X	X	X	
Amount of free liquids	X	X	X	
Estimated weights for the 12 waste matrix material parameters	X	X	X	Table B3-1 lists waste matrix material parameters.
Container gross weight	X	X	X	
Container empty weight	O	O	O	Established, documented empty container weights can be used.
Comments	X	X	X	
Reference to or copy of associated NCRs, if any	X	X	X	Copies of associated NCRs must be available.
Visual examination expert decisions		X		Only applicable if visual examination expert is consulted during visual examination.
Verify absence of prohibited items	X	X	X	
Operator signature and date of test	X	X	X	Signatures of both operators required for Visual Verification of Acceptable Knowledge
Signature of visual examination expert and date		X		
Data review checklists	X	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.

**TABLE B3-12  
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
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
<del>Packaging Configuration</del>	<del>X</del>		<del>If Scenario 3 is used, the packaging configuration used in determining the DAG must be documented in the headspace gas sampling documentation.</del>

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.
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
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.



**TABLE B3-13  
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	
Confirmation 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 B3-14 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.

### TABLE B3-14 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

## **FIGURES**

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Figure B3-1  
Overall Headspace-Gas Sampling Scheme Illustrating Manifold Sampling