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**RENEWAL APPLICATION
APPENDIX B3**

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

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TABLE OF CONTENTS

8	List of Tables	iii
9	List of Figures	iii
10	B3-1 Validation Methods.....	B3-1
11	B3-2 Headspace Gas Sampling.....	B3-7
12	B3-3 Sampling of Homogeneous Solids and Soils/Gravel.....	B3-9
13	B3-4 Non Destructive Examination Methods.....	B3-11
14	B3-4a Radiography	B3-12
15	B3-4b Visual Examination.....	B3-12
16	B3-5 Gas Volatile Organic Compound Analysis.....	B3-13
17	B3-6 Total Volatile Organic Compound Analysis.....	B3-15
18	B3-7 Total Semivolatile Organic Compound Analysis	B3-16
19	B3-8 Total Metal Analysis.....	B3-18
20	B3-9 Acceptable Knowledge	B3-21
21	B3-10 Data Review, Validation, and Verification Requirements.....	B3-22
22	B3-10a Data Generation Level	B3-23
23	B3-10a(1) Independent Technical Review	B3-24
24	B3-10b Project Level	B3-26
25	B3-10b(1) <u>Certified Characterization Program</u> Site Project Manager Review	B3-26
26	B3-10b(2) Prepare <u>Certified Characterization Program</u> Site Project Manager Summary and	
27	Data Validation Summary.....	B3-27
28	B3-10b(3) Prepare Waste Stream Characterization Package.....	B3-28
29	B3-10c Permittee Level	B3-28
30	B3-11 Reconciliation with Data Quality Objectives	B3-29
31	B3-11a Reconciliation at the Project Level	B3-29
32	B3-11b Reconciliation at the Permittee Level.....	B3-31
33	B3-12 Data Reporting Requirements.....	B3-31
34	B3-12a Data Generation Level	B3-31
35	B3-12b Project Level	B3-31
36	B3-12b(1) Waste Stream Profile Form.....	B3-32
37	B3-12b(2) Characterization Information Summary.....	B3-33
38	B3-12b(3) Waste Stream Characterization Package.....	B3-34
39	B3-12b(4) WIPP Waste Information System (WWIS) Data Reporting	B3-34

1	B3-13 Nonconformances	B3-34
2	B3-14 Special Training Requirements and Certifications	B3-37
3	B3-15 Changes to <u>Waste Analysis Plan</u> WAP -Related Plans or Procedures	B3-37
4	B3-16 List of References	B3-39

List of Tables

Table	Title
B3-1	Waste Material Parameters and Descriptions
B3-2	Gas Volatile Organic Compounds Target Analyte List and Quality Assurance Objectives
B3-3	Summary of Laboratory Quality Control Samples and Frequencies for Gas Volatile Organic Compound Analysis
B3-4	Volatile Organic Compounds Target Analyte List and Quality Assurance Objectives <u>for Solids Analysis</u>
B3-5	Summary of Laboratory Quality Control Samples and Frequencies for Volatile Organic Compound Analysis
B3-6	Semi-volatile Organic Compound Target Analyte List and Quality Assurance Objectives <u>for Solids Analysis</u>
B3-7	Summary of Laboratory Quality Control Samples and Frequencies for Semi-Volatile Organic Compounds Analysis
B3-8	Metals Target Analyte List and Quality Assurance Objectives
B3-9	Summary of Laboratory Quality Control Samples and Frequencies for Metals Analysis
B3-10	Minimum Training and Qualifications Requirements
B3-11	Testing Batch Data Report Contents
B3-12	Sampling Batch Data Report Contents
B3-13	Analytical Batch Data Report Contents
B3-14	Data Reporting Flags

List of Figures

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

Waste Isolation Pilot Plant
Hazardous Waste Facility Permit
Draft Renewal Application
May 2009

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APPENDIX B3**

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B3-1 Validation Methods

The Permittees shall require the generator/storage sites (~~sites~~) certified characterization programs or Permittee approved laboratories 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 Organics Compounds (**VOC**), total VOCs, Semi-volatile 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 (VE) is not amenable to statistical data quality analysis. However, radiography and ~~visual examination~~ VE are complementary techniques yielding similar data for determining the waste matrix code. The waste matrix code is determined to ensure 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:

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

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

3
4 For three or more replicate measurements, the precision expressed as the %RSD is calculated as
5 follows:

$$6 \quad \%RSD = \frac{s}{y_{mean}} \times 100 \quad (B3-2)$$

7
8
9
10 where s is the standard deviation and y_{mean} is the mean of the replicate sample analyses.

11
12 The standard deviation, s , is calculated as follows:

$$13 \quad s = \sqrt{\frac{\sum_{i=1}^n (y_i - y_{mean})^2}{n - 1}} \quad (B3-3)$$

14
15
16
17
18 where y_i is the measured value of the i th replicate sample analysis measurement, and n equals the
19 number of replicate analyses.

20
21 Another aspect of precision is associated with analytical equipment calibration. In these
22 instances, the percent difference (%D) between multiple measurements of an equipment
23 calibration standard shall be calculated as follows:

$$24 \quad \%D = \frac{|C_1 - C_2|}{C_1} \times 100 \quad (B3-4)$$

25
26
27
28 where C_1 is the initial measurement and C_2 is the second or other additional measurement.

29 Accuracy

30
31 Accuracy is the degree of agreement between a measured analyte concentration (or the average
32 of replicate measurements of a single analyte concentration) and the true or known
33 concentration. Accuracy is determined as the percent recovery (%R).

34
35 For situations where a standard reference material is used, the %R is calculated as follows:

1
$$\%R = \frac{C_m}{C_{sm}} \times 100 \quad (B3-5)$$

2
3 where C_m is the measured concentration value obtained by analyzing the sample and C_{sm} is the
4 “true” or certified concentration of the analyte in the sample.

5
6 For measurements where matrix spikes are used, the %R is calculated as follows:

7
8
9
$$\%R = \frac{S - U}{C_{sc}} \times 100 \quad (B3-6)$$

10
11
12 where S is the measured concentration in the spiked aliquot, U is the measured concentration in
13 the unspiked aliquot, and C_{sc} is the actual concentration of the spike added.

14
15 Method Detection Limit

16
17 The method detection limit (**MDL**) is the minimum concentration of an analyte that can be
18 measured and reported with 99 percent confidence that the analyte concentration is greater than
19 zero. The MDL for all quantitative measurements (except for those using Fourier Transform
20 Infrared Spectroscopy (**FTIRS**)) is defined as follows:

21
22
23
$$MDL = t_{(n-1, 1-\alpha=0.99)} \times s \quad (B3-7)$$

24
25
26 where $t_{(n-1, 1-\alpha=0.99)}$ is the t-distribution value corresponding to a 99 percent confidence level with n-
27 1 degrees of freedom, n is the number of observations, and s is the standard deviation of replicate
28 measurements.

29
30 For headspace gas (**HSG**) analysis using FTIRS, MDL is defined as follows:

31
32
33
$$MDL = 3s \quad (B3-8)$$

34
35
36 where s is the standard deviation. Initially, a minimum of seven samples spiked at a level of
37 three to five times the estimated MDL and analyzed on non-consecutive days must be used to
38 establish the MDLs. **The** MDLs should be updated using the results of the laboratory control
39 sample or on-line control samples.

1 Completeness
2

3 Completeness is a measure of the amount of valid data obtained from the overall measurement
4 system compared to the amount of data collected and submitted for analysis. Completeness must
5 be expressed as the number of samples analyzed with valid results as a percent of the total
6 number of samples submitted for analysis. Completeness, expressed as the percent complete
7 (%C), is calculated as follows:
8
9

10
$$\% C = \frac{V}{n} \times 100 \quad (B3-9)$$

11

12
13 where V is the number of valid sampling or analytical results obtained and n is the number of
14 samples submitted for analysis.
15

16 Comparability
17

18 Comparability is the degree to which one data set can be compared to another. Comparability of
19 data generated at different transuranic (TRU) waste sites will be ensured through the use of
20 standardized, approved testing, sampling, preservation, and analytical techniques and by meeting
21 the QAOs specified in Sections B3-2 through B3-9.
22

23 The comparability of waste characterization data shall be ensured through the use of
24 generator/storage site certified characterization programs or Permittee approved laboratories data
25 usability criteria. The Permittees shall ensure that data usability criteria are consistently
26 established and used by the generator/storage sites certified characterization programs or
27 Permittee approved laboratories to assess the usability of analytical and testing data. The criteria
28 shall address, as appropriate, the following:
29

- 30
- 31 • Definition or reference of criteria used to define and assign data qualifier flags based on
~~Quality Assurance Objective~~ QAO results;
 - 32 • Criteria for assessing the usability ~~useability~~ of data impacted by matrix interferences;
 - 33 • Criteria for assessing the usability ~~useability~~ of data based upon positive and negative
34 bias as indicated by quality control (QC) data, of data qualifiers, and qualifier flags;
 - 35 • Criteria for assessing the usability ~~useability~~ of data due to:
 - 36 • Severe matrix effects;
 - 37 • Misidentification of compounds;
 - 38 • Gross exceedance of holding times;
 - 39 • Failure to meet calibration or tune criteria
 - 40

- Criteria for assessing the usability useability of data that does not meet minimum detection limit requirements-

The Permittees shall be responsible for evaluating generator/storage site certified characterization program data useability usability and shall assess implementation through the generator/storage site certified characterization program 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 HSG headspace gas, homogeneous solids, and soil/gravel sampling and analysis will be validated, through documentation, that a true random sample with an adequate population was identified and collected consistent with Renewal Application Appendix B2 (Statistical Methods Used in Sampling and Analysis), Section B2-1. 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 P program level. The Permittees shall require the certified characterization program 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 followed to ensure that samples are representative of the waste contained in a particular waste container or a waste stream.

Identification of Tentatively Identified Compounds

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

- ~~• Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.~~
- ~~• The relative intensities of the major ions should agree within ± 20 percent.~~
- ~~• Molecular ions present in the reference spectrum should be present in the sample spectrum.~~
- ~~• Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.~~

- 1 • Ions present in the reference spectrum but not in the sample spectrum should be reviewed
2 for possible subtraction from the sample spectrum because of background contamination
3 or coeluting peaks.

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

- 8 • TICs for headspace gas analyses that are performed through FTIR analyses shall be
9 identified in accordance with the specifications of SW-846 Method 8410.

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

- 12
- 13 • a TIC in an individual container headspace gas or solids sample shall be reported in the
14 analytical batch data report if the TIC meets the SW-846 identification criteria listed
15 above and is present with a minimum of 10% of the area of the nearest internal standard.

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

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

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

28 TICs that meet the SW-846 identification criteria, are reported in 25 percent of all waste
29 containers sampled from a given waste stream, and that appear in the 20.4.1.200 NMAC
30 (incorporating 40 CFR §261) Appendix VIII list, will be compared to acceptable knowledge
31 data to determine if the TIC is a listed waste in the waste stream. TICs identified through
32 headspace gas analyses that meet the Appendix VIII list criteria and the 25 percent reporting
33 criteria for a waste stream will be added to the headspace gas waste stream target list regardless
34 of the hazardous waste listing associated with the waste stream. TICs reported from the Totals
35 VOC or SVOC analyses may be excluded from the target analyte list for a waste stream if the
36 TIC is a constituent in an F-listed waste whose presence is attributable to waste packaging
37 materials or radiolytic degradation from acceptable knowledge documentation. If a listed waste
38 constituent TIC cannot be attributed to waste packaging materials, radiolysis, or other origins,

1 the constituent will be added to the target analyte list and new hazardous waste numbers will be
2 assigned, if appropriate. TICs subject to inclusion on the target analyte list that are toxicity
3 characteristic parameters shall be added to the target analyte list regardless of origin because the
4 hazardous waste designation for these numbers is not based on source. However, for toxicity
5 characteristic and non-toxic F003 constituents, the site may take concentration into account when
6 assessing whether to add a hazardous waste number. If a target analyte list for a waste stream is
7 expanded due to the presence of TICs, all subsequent samples collected from that waste stream
8 will be analyzed for constituents on the expanded list.

10 B3-2 Headspace Gas Sampling

11 Quality Assurance Objectives

12
13 The precision and accuracy of the container HSG headspace gas sampling operations must be
14 assessed by analyzing field QC HSG headspace gas samples. These samples must include
15 equipment blanks, field reference standards, field blanks, and field duplicates. If the QAOs
16 described below are not met, a nonconformance report (NCR) must be prepared, submitted, and
17 resolved (Section B3-13).

19 Precision

20
21 The precision of the HSG headspace gas sampling and analysis operation must be assessed by
22 sequential collection of field duplicates for manifold sampling operations or simultaneous
23 collection of field duplicates for direct canister sampling operations for VOCs determination.
24 Corrective actions must be taken if the RPD exceeds 25 percent for any analyte found greater
25 than the Program Required Quantitation Limits (PRQL) in both samples of the duplicate
26 samples.

28 Accuracy

29
30 A field reference standard must be collected using HSG headspace gas sampling equipment to
31 assess the accuracy of the HSG headspace gas sampling operation at a frequency of one field
32 reference standard for every 20 containers sampled or per sampling batch. Corrective action
33 must be taken if the %R of the field-reference standard is less than 70 or greater than 130.

34
35 Field blanks must also be collected at a frequency of ± one field blank for every 20 containers or
36 sampling batch sampled to assess possible contamination in the HSG headspace gas sampling
37 method. Equipment blanks must also be collected at a frequency of ± one equipment blank for
38 each equipment cleaning batch to assess possible contamination in the equipment cleaning
39 method. Corrective actions must be taken if the blank exceeds three times the MDLs listed for
40 any of the compounds listed in Table B3-2.

44 Completeness

1
2 Sampling completeness shall be expressed as the number of valid samples collected as a percent
3 of the total number of samples collected for each waste stream A valid sample is defined as a
4 sample collected in accordance with approved sampling methods ~~and the~~ from a container that
5 was properly prepared for sampling (e.g., the polyliner was vented to the container headspace).
6 The Permittees shall require participating sampling facilities to achieve a minimum 90 percent
7 completeness. The amount and type of data that may be lost during the HSG ~~headspace gas~~
8 sampling operation cannot be predicted in advance. The Permittees shall require the certified
9 characterization program Site Project Manager to evaluate the importance of any lost or
10 contaminated HSG ~~headspace gas~~ samples and take corrective action as appropriate.

11 Comparability

12
13 Consistent use and application of uniform procedures and equipment, as specified in Renewal
14 Application Appendix B1 (Waste Characterization Sampling Methods) and application of data
15 ~~usability~~ usability criteria, should ensure that HSG ~~headspace gas~~ sampling operations are
16 comparable when sampling headspace at the different sampling facilities. The Permittees shall
17 require each site certified characterization program to take corrective actions if uniform
18 procedures, equipment, or operations are not followed without approved and justified deviations.
19 In addition, laboratories analyzing samples must successfully participate in a written ~~the~~
20 ~~Performance Demonstration Program~~ performance demonstration program (PDP) (DOE, 2003).
21
22

23 Representativeness

24
25 Specific HSG ~~headspace gas~~ sampling steps to ensure samples are representative include:

- 26
27 • Selection of the correct Drum Age Criteria (DAC) Scenario and waste packaging
28 configuration and meeting DAC equilibrium times-
- 29 • A sample canister cleaning and leak check after assembly
- 30 • Sampling equipment cleaning or disposal after use
- 31 • Sampling equipment leak check after sample collection
- 32 • Use of sample canisters with passivated internal surfaces
- 33 • Use of ~~low internal volume~~ low internal volume sampling equipment
- 34 • Collection of samples with a ~~low sample~~ low sample volume to available headspace
35 volume ratio (less than 10 percent of the headspace when the headspace can be
36 determined)
- 37 • Careful and documented pressure regulation of all activities specified in Appendix B1,
38 Section B1-1

- 1 • Performance audits
- 2 • Collection of equipment blanks, field reference standards, field blanks, and field
3 duplicates at the specified frequencies-
- 4 • Manifold pressure sensors and temperature sensors calibrated before initial use and
5 annually using NIST, or equivalent standards-
- 6 • Organic Vapor Analyzer OVA calibrated daily, prior to first use, or as necessary
7 according to manufacturer's specifications-

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

9 10 B3-3 Sampling of Homogeneous Solids and Soils/Gravel

11 Quality Assurance Objectives

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

19 20 Precision

21
22 Sampling precision must be determined by collecting and sampling field duplicates (e.g., co-
23 located cores or co-located samples as described in Renewal Application Appendix B1,
24 Section B1-2b(1)) once per sampling batch or once per week during sampling operations,
25 whichever is more frequent. A sampling batch is a suite of homogeneous solids and soil/gravel
26 samples collected consecutively using the same sampling equipment within a specific time
27 period. A sampling batch can be up to 20 samples (excluding field QC samples), all of which
28 must be collected within 14 days of the first sample in the batch. The Permittees shall require
29 the certified characterization program Site Project Manager to calculate and report the RPD
30 between co-located cores/samples.

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

1
2 The statistical test will involve calculating the variance for co-located cores and samples by
3 pooling the variances computed for each pair of duplicate results. The variance for the waste
4 stream will be computed excluding any data from containers with co-located cores, because the
5 test requires the variance estimates to be independent. All data must be transformed to normality
6 prior to computing variances and performing the test. The test hypothesis is evaluated using the
7 F distribution and the method for testing the difference in variances.
8

9 Accuracy

10
11 Sampling accuracy through the use of standard reference materials shall not be measured.
12 Because waste containers containing homogeneous solids and soil/gravel with known quantities
13 of analytes are not available, sampling accuracy cannot be determined. However, sampling
14 methods and requirements described are designed to minimize sample degradation and hence
15 maximize sampling accuracy.
16

17 Sampling accuracy as a function of sampling cross-contamination will be measured. Equipment
18 blanks will be collected at a frequency of once per equipment cleaning batch. Corrective actions
19 must be taken if the blank exceeds three times the MDLs (Program Required Detection Limits
20 (PRDLs) for metals) listed for any of the compounds or analytes listed in Tables B3-4, B3-6, and
21 B3-8. Equipment blanks will be collected from the following equipment types:
22

- 23 • Fully assembled coring tools
 - 24 • Liners cleaned separately from coring tools
 - 25 • Miscellaneous sampling equipment that is reused (bowls, spoons, chisels)
- 26

27 Completeness

28
29 Sampling completeness shall be expressed as the number of valid samples collected as a percent
30 of the total number of samples collected for each waste stream. A valid sample is any sample
31 that is collected ~~from a randomly selected container using randomly selected horizontal and~~
32 ~~vertical planes~~ in accordance with approved sampling methods. The Permittees shall require the
33 certified characterization program or Permittee approved participating sampling facility facilities
34 to achieve a minimum 90 percent completeness.
35

36 Comparability

37
38 Consistent use and application of uniform procedures, sampling equipment, and measurement
39 units must ensure that sampling operations are comparable. Consistent application of data
40 usability ~~useability~~ criteria will also ensure comparability. ~~In addition, the Permittees shall~~
41 ~~require laboratories analyzing samples to successfully participate in the PDP (DOE, 2005).~~
42

43 Representativeness

44
45 Representativeness is assured by collecting random samples using an approved method.

1
2 Specific steps to ensure the representativeness of samples include the following for both waste
3 containers and smaller containers:

- 4
- 5 • ~~Coring tools and sampling equipment must be clean prior to sampling.~~
 - 6 • ~~The entire depth of the waste minus a site defined approved safety factor must be cored,~~
7 ~~and the core collected must have a length greater than or equal to 50 percent of the depth~~
8 ~~of the waste. This is called the core recovery and is calculated as follows:~~

9
$$\text{Core recovery (percent)} = \frac{y}{x} \times 100 \text{ (B3-10)}$$

10 where

11 x = the depth of the waste in the container

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

- 13
- 14 • ~~Coring operations and tool selection should be designed to minimize alteration of the~~
15 ~~in-place waste characteristics. Minimal waste disturbance must be verified by visually~~
16 ~~examining the core and describing the observation (e.g., undisturbed, cracked, or~~
17 ~~pulverized) in the field logbook.~~

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

21 ~~One randomly selected container within a container will be chosen if the container contains~~
22 ~~individual waste containers.~~

24 B3-4 Non Destructive Examination Methods

25 Quality Assurance Objectives

26
27 The QAOs for non-destructive examination (NDE) are detailed in this section. ~~The~~ NDE can be
28 either radiography or ~~visual examination (VE)~~ ~~VE~~. If the QAOs described below are not met,
29 then corrective action shall be taken. It should be noted that NDE does not have a specific MDL
30 because it is primarily a qualitative determination. The objective of NDE for the program is to
31 determine the physical waste form, the absence of prohibited items, and additional waste
32 characterization techniques that may be used based on the Summary Category Groups (i.e.,
33 S3000, S4000, S5000). The Permittees shall require each site certified characterization program
34 to describe all activities required to achieve these objectives in the site certified characterization
35 program quality assurance project plan (QAPjP) and standard operating procedures (SOP).
36

1 B3-4a Radiography

2 Data to meet these objectives must be obtained from a video and audio recorded scan provided
3 by trained radiography operators at the TRU waste sites. Results must also be recorded on a
4 radiography data form. The precision, accuracy, completeness, and comparability objectives for
5 radiography data are presented below.

6
7 Precision

8
9 Precision is maintained by reconciling any discrepancies between two radiography operators
10 with regard to identification of the waste matrix code, liquids in excess of Treatment, Storage
11 and Disposal Facility, Waste Acceptance Criteria (TSDF-WAC) limits, and compressed gases
12 through independent replicate scans and independent observations. Additionally, the precision
13 of radiography is verified prior to use by tuning precisely enough to demonstrate compliance
14 with QAOs through viewing an image test pattern.

15
16 Accuracy

17
18 Accuracy is obtained by using a target to tune the image for maximum sharpness and by
19 requiring operators to successfully identify 100 percent of the required items in a training
20 container during their initial qualification and subsequent requalification.

21
22 Completeness

23
24 A video and audio media recording of the radiography examination and a validated radiography
25 data form will be obtained for 100 percent of the waste containers subject to radiography. All
26 video and audio media recordings and radiography data forms will be subject to validation as
27 indicated in Section B3-10.

28
29 Comparability

30
31 The comparability of radiography data from different operators shall be enhanced by using
32 standardized radiography procedures and operator qualifications.

33
34 B3-4b Visual Examination

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

37
38 Precision

39
40 Precision is maintained by reconciling any discrepancies between the operator and the
41 independent technical reviewer with regard to identification of waste matrix code, liquids in
42 excess of TSDF-WAC limits, and compressed gases.

43
44

1 Accuracy

2
3 Accuracy is maintained by requiring operators to pass a comprehensive examination and
4 demonstrate satisfactory performance in the presence of the VE expert during their initial
5 qualification and subsequent requalification.

6
7 Completeness

8
9 A validated VE data form will be obtained for 100 percent of the waste containers subject to VE.

10
11 Comparability

12
13 The comparability of VE data from different operators shall be enhanced by using standardized
14 VE procedures and operator qualifications.

15
16 B3-5 Gas Volatile Organic Compound Analysis

17 Quality Assurance Objectives

18
19 The development of data quality objective (DQOs) specifically for this program has resulted in
20 the QAOs listed in Table B3-2. The specified QAOs represent the required quality of data
21 necessary to draw valid conclusions regarding program objectives. Waste Analysis Plan (WAP)
22 ~~WAP~~-required limits, such as the ~~program required quantitation limits (PRQL)~~ PRQL associated
23 with VOC analysis, are specified to ensure that the analytical data collected satisfy the
24 requirements of all data users. A summary of the ~~Quality Control QC~~ QC Samples and the
25 associated acceptance criteria is included in Table B3-3. ~~Key data quality indicators~~ Quality
26 Assurance Objectives for laboratory measurements are defined below.

27
28 Precision

29
30 Precision shall be assessed by analyzing laboratory duplicates and replicate analyses of
31 laboratory control ~~laboratory control~~ samples and PDP (DOE, 2005) blind audit ~~blind audit~~
32 samples. Results from measurements on these samples must be compared to the criteria listed in
33 Table B3-2. These QC measurements will be used to demonstrate acceptable method
34 performance and to trigger corrective action when control limits are exceeded.

35
36 Accuracy

37
38 Accuracy as %R shall be assessed for the laboratory operations by analyzing PDP (DOE, 2005)
39 blind audit ~~blind audit~~ samples and laboratory control ~~laboratory control~~ samples. Results from
40 these measurements must be compared to the criteria listed in Table B3-2. These QC
41 measurements will be used to demonstrate acceptable method performance and to trigger
42 corrective action when control limits are exceeded.

1 Calibration

2
3 The GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and
4 evaluated using the procedures and criteria specified in Table B3-3. These criteria will be used
5 to demonstrate acceptable calibration and to trigger corrective action when control limits are
6 exceeded.

7
8 Method Detection Limit

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

14
15 Program Required Quantitation Limit

16
17 Laboratories must demonstrate the capability to quantitate analytes at or below the PRQLs given
18 in Table B3-2. Laboratories shall set the concentration of at least one calibration standard below
19 the PRQL. The detailed procedures for PRQL demonstration shall be included in certified
20 characterization program or Permittee approved laboratory SOPs.

21
22 Completeness

23
24 Laboratory completeness shall be expressed as the number of samples analyzed with valid results
25 as a percent of the total number of samples submitted for analysis. A composited sample is
26 treated as one sample for the purposes of completeness, because only one sample is run through
27 the analytical instrument. Valid results are defined as results that meet the data usability
28 usability criteria based on application of the ~~Quality Control~~ QC Criteria specified in Tables
29 B3-2 and B3-3; and that meet the detection limit, calibration, representativeness, and
30 comparability criteria within this section. The Permittees shall require that the certified
31 characterization program or Permittee approved laboratory participating laboratories meet the
32 completeness criteria specified in Table B3-2.

33
34 Comparability

35
36 For VOC analysis, data generated through analysis of samples from different TRU waste sites
37 shall be comparable. The Permittees shall require each site certified characterization program or
38 Permittee approved laboratory to achieve comparability by using standardized methods and
39 traceable standards and by requiring all sites certified characterization programs or Permittee
40 approved laboratories to successfully participate in a written the PDP (~~DOE, 2003~~).

41
42 Representativeness

43
44 Representativeness for VOC analysis shall be achieved by collecting sufficient numbers of
45 samples using clean sampling equipment that does not introduce sample bias. Samples must be
46 collected as described in Renewal Application Appendix B1.

1
2 B3-6 Total Volatile Organic Compound Analysis

3 Quality Assurance Objectives

4
5 The development of DQOs specifically for this program has resulted in the QAOs listed in Table
6 B3-4. The specified QAOs represent the required quality of data necessary to draw valid
7 conclusions regarding program objectives. ~~The WAP required~~ ~~WAP required~~ limits, such as the
8 PRQL associated with VOC analysis, are specified to ensure that the analytical data collected
9 satisfy the requirements of all data users. ~~Key data quality indicators~~ The QAOs for laboratory
10 measurements are defined below.

11
12 Precision

13
14 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,
15 replicate analyses of laboratory control samples, and PDP ~~(DOE, 2005) blind audit~~ ~~blind-audit~~
16 samples. Results from measurements on these samples must be compared to the criteria listed in
17 Table B3-4. These QC measurements will be used to demonstrate acceptable method
18 performance and to trigger corrective action when control limits are exceeded.

19
20 Accuracy

21
22 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control
23 samples, matrix spikes, surrogate compounds, and PDP ~~(DOE, 2005) blind audit~~ ~~blind-audit~~
24 samples. Results from these measurements for matrix spikes ~~spike~~ samples must be compared to
25 the %R criteria listed in Table B3-4. Results for surrogates and internal standards are evaluated
26 as specified in the SW-846 ~~(EPA, 1996)~~ ~~(EPA 1996)~~ method or Table B3-5. These QC
27 measurements will be used to demonstrate acceptable method performance and to trigger
28 corrective action when control limits are exceeded.

29
30 Laboratory blanks shall be assessed to determine possible laboratory contamination and are
31 evaluated as specified in Table B3-5. These QC measurements will be used to demonstrate
32 acceptable levels of laboratory contamination and to trigger corrective action when control limits
33 are exceeded.

34
35 Calibration

36
37 ~~The~~ GC/MS Tunes, Initial Calibrations, and Continuing Calibrations will be performed and
38 evaluated using the procedures and criteria specified in Table B3-5 and the SW-846 ~~(EPA, 1996)~~
39 method ~~(EPA 1996)~~. These criteria will be used to demonstrate acceptable calibration and to
40 trigger corrective action when control limits are exceeded.

41
42 Method Detection Limit

43
44 ~~The~~ MDLs shall be expressed in milligrams per kilogram (mg/kg) for VOCs and must be less
45 than or equal to those listed in Table B3-4. The detailed procedures for MDL determination

1 shall be included in site certified characterization program or Permittee approved laboratory
2 SOPs.

3 4 Program Required Quantitation Limit

5
6 Laboratories must demonstrate the capability to quantitate analytes in samples at or below the
7 PRQLs given in Table B3-4. Laboratories shall set the concentration of at least one calibration
8 standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in
9 certified characterization program or Permittee approved laboratory SOPs.

10 11 Completeness

12
13 Laboratory completeness shall be expressed as the number of samples analyzed with valid results
14 as a percent of the total number of samples submitted for analysis. Valid results are defined as
15 results that meet the data usability ~~useability~~ criteria based upon application of the Quality
16 ~~Control~~ QC Criteria specified in Tables B3-4 and B3-5 and meet the calibration, detection limit,
17 representativeness, and comparability criteria within this section. Participating laboratories must
18 meet the completeness criteria specified in Table B3-4.

19 20 Comparability

21
22 For VOC analysis, data generated through analysis of samples from different sites certified
23 characterization programs or Permittee approved laboratories shall be comparable. The
24 Permittees shall require sites to achieve comparability by using standardized SW-846 (EPA,
25 1996) sample preparation and analytical methods that meet the QAO requirements in Tables B3-
26 4 and B3-5, traceable standards, and by requiring all sites certified characterization programs or
27 Permittee approved laboratories to successfully participate in a written ~~the~~ PDP (DOE, 2003).
28 ~~Generator/storage sites~~ Certified characterization programs or Permittee approved laboratories
29 may use the most recent version of SW-846 (EPA, 1996). Any changes to SW-846 (EPA, 1996)
30 methodology that ~~results~~ result in the elimination of sample preparation or analytical methods in
31 use ~~at generator/storage sites~~ by certified characterization programs or Permittee approved
32 laboratories must be addressed as a corrective action to address the comparability of data before
33 and after the SW-846 (EPA, 1996) modification.

34 35 Representativeness

36
37 Representativeness for VOC analysis shall be achieved by collecting unbiased samples. Samples
38 must be collected as described in Renewal Application Appendix B1.

39 40 B3-7 Total Semivolatile Organic Compound Analysis

41 Quality Assurance Objectives

42
43 The development of DQOs specifically for this program has resulted in the QAOs listed in Table
44 B3-6. The specified QAOs represent the required quality of data necessary to draw valid

1 conclusions regarding program objectives. ~~The WAP required~~ WAP-required limits, such as the
2 PRQLs, are specified to ensure that the analytical data collected satisfy the requirements of all
3 data users. A summary of ~~Quality Control~~ QC Samples and associated acceptance criteria for
4 this analysis is included in Table B3-7. ~~Key data quality indicators~~ The QAOs for laboratory
5 measurements are defined below.

6 7 Precision 8

9 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,
10 replicate analyses of laboratory control samples, and PDP (DOE, 2005) blind audit blind-audit
11 samples. Results from measurements on these samples must be compared to the criteria listed in
12 Table B3-6. These QC measurements will be used to demonstrate acceptable method
13 performance and to trigger corrective action when control limits are exceeded.
14

15 Accuracy 16

17 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control
18 samples, matrix spikes, surrogate compounds, and PDP (DOE, 2005) blind audit blind-audit
19 samples. Results from these measurements for matrix spikes samples must be compared to the
20 %R criteria listed in Table B3-6. Results for surrogates and internal standards are evaluated as
21 specified in the SW-846 (EPA, 1996) method (EPA-1996) or Table B3-7. These QC
22 measurements will be used to demonstrate acceptable method performance and to trigger
23 corrective action when control limits are exceeded.
24

25 Laboratory blanks shall be assessed to determine possible laboratory contamination and are
26 evaluated as specified in Table B3-7. These QC measurements will be used to demonstrate
27 acceptable levels of laboratory contamination and to trigger corrective action when control limits
28 are exceeded.
29

30 Calibration 31

32 Gas chromatography/mass spectroscopy GC/MS Tunes, Initial Calibrations, and Continuing
33 Calibrations will be performed and evaluated using the procedures and criteria specified in Table
34 B3-7 and the SW-846 (EPA, 1996) method (EPA-1996). These criteria will be used to
35 demonstrate acceptable calibration and to trigger corrective action when control limits are
36 exceeded.
37

38 Method Detection Limit 39

40 The MDLs shall be expressed in mg/kg for SVOCs and must be less than or equal to those listed
41 in Table B3-6. The detailed procedures for MDL determination shall be included in site certified
42 characterization program or Permittee approved laboratory SOPs.
43
44
45

1 Program Required Quantitation Limit

2
3 Laboratories must demonstrate the capability to quantitate analytes in samples at or below the
4 PRQLs given in Table B3-6. Laboratories shall set the concentration of at least one calibration
5 standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in
6 certified characterization program or Permittee approved laboratory SOPs.

7
8 Completeness

9
10 Laboratory completeness shall be expressed as the number of samples analyzed with valid results
11 as a percent of the total number of samples submitted for analysis. Valid results are defined as
12 results that meet the data ~~useability~~ usability criteria based on application of the ~~Quality Control~~
13 QC Criteria specified in Tables B3-6 and B3-7 and that meet the detection limit, calibration,
14 representativeness, and comparability criteria within this section. The Permittees shall require
15 participating laboratories to meet the level of completeness specified in Table B3-6.

16
17 Comparability

18
19 For SVOC analysis, data generated through analysis of samples from different TRU waste sites
20 shall be comparable. The Permittees shall require sites certified characterization programs or
21 Permittee approved laboratories to achieve comparability by using standardized SW-846 (EPA,
22 1996) sample preparation and analytical methods that meet the QAO requirements in Tables
23 B3-6 and B3-7, traceable standards, and by requiring all sites certified characterization programs
24 or Permittee approved laboratories to successfully participate in a written the PDP (DOE, 2003).
25 ~~Generator/storage sites~~ Certified characterization programs or Permittee approved laboratories
26 may use the most current version of SW-846 (EPA, 1996) if the methods are consistent with
27 QAO requirements. Any changes to SW-846 (EPA, 1996) methodology that results in the
28 elimination of sample preparation or analytical methods in use ~~at generator/storage sites~~ by
29 certified characterization programs or Permittee approved laboratories must be addressed as a
30 corrective action to address the comparability of data before and after the SW-846 (EPA, 1996)
31 modification.

32
33 Representativeness

34
35 Representativeness for SVOC analysis shall be achieved by collecting unbiased samples.
36 Samples must be collected as described in Renewal Application Appendix B1.
37

38 B3-8 Total Metal Analysis

39 Quality Assurance Objectives

40
41 The development of DQOs for the program has resulted in the QAOs listed in Table B3-8. The
42 specified QAOs represent the required quality of data necessary to draw valid conclusions
43 regarding program objectives. The WAP required ~~WAP required~~ limits, such as the PRQLs
44 associated with metal analysis, are specified to ensure that the analytical data collected satisfy

1 the requirements of all data users. A summary of ~~Quality Control~~ **QC** Samples and the
2 associated acceptance criteria for this analysis is provided in Table B3-9. ~~Key data quality~~
3 ~~indicators~~ **The QAOs** for laboratory measurements are defined below.

4 5 Precision

6
7 Precision shall be assessed by analyzing laboratory sample duplicates or laboratory matrix spike
8 duplicates, replicate analyses of **laboratory control** ~~laboratory control~~ samples, and PDP **(DOE,**
9 **2005) WAP required** ~~WAP required~~ samples. Results from measurements on these samples
10 must be compared to the criteria listed in Table B3-8. These QC measurements will be used to
11 demonstrate acceptable method performance and to trigger corrective action when control limits
12 are exceeded.

13 14 Accuracy

15
16 Accuracy shall be assessed through the analysis of laboratory matrix spikes, PDP **(DOE, 2005)**
17 **blind audit** ~~blind audit~~ samples, serial dilutions, interference check samples, and **laboratory**
18 **control** ~~laboratory control~~ samples. Results from these measurements must be compared to the
19 criterion listed in Table B3-8 and B3-9. These QC measurements will be used to demonstrate
20 acceptable method performance and to trigger corrective action when control limits are
21 exceeded.

22
23 Laboratory blanks and calibration blanks shall be assessed to determine possible laboratory
24 contamination and are evaluated as specified in Table B3-9. These QC measurements will be
25 used to demonstrate acceptable levels of laboratory contamination and to trigger corrective
26 action when control limits are exceeded.

27 28 Calibration

29
30 Mass Tunes (for **Inductively Coupled Plasma-Mass Spectroscopy ICP-MS** only), Standards
31 Calibration, Initial Calibration verifications, and Continuing Calibrations will be performed and
32 evaluated using the procedures and criteria specified in Table B3-9 and the SW-846 method
33 (EPA, 1996). These criteria will be used to demonstrate acceptable calibration and to trigger
34 corrective action when control limits are exceeded.

35 36 37 38 Program Required Detection Limits

39
40 **The** PRDLs, expressed in units of micrograms per L ($\mu\text{g/L}$), are the maximum values for
41 instrument detection limits (**IDLs**) permissible for program support under the WAP. **The** IDLs
42 must be less than or equal to the **program required detection limit (PRDL)** ~~PRDL~~ for the method
43 used to quantitate a specific analyte. Any method listed in Table B-5 of the Renewal Application
44 Chapter B (Waste Analysis Plan) ~~(Permit Chapter B)~~ may be used if the IDL meets this **criterion**
45 **criteria**. For high concentration samples, an exception to the above requirements may be made in

1 cases where the sample concentration exceeds five times the IDL of the instrument being used.
2 In this case, the analyte concentration may be reported even though the IDL may exceed the
3 PRDL. The IDLs shall be determined semiannually (i.e., every six months). Detailed
4 procedures for IDL determination shall be included in laboratory SOPs.
5

6 Program Required Quantitation Limit

7
8 The Permittees shall require participating laboratories to demonstrate the capability of analyte
9 quantitation at or below the PRQLs in units of mg/kg wet weight (given in Table B3-8). The
10 PRDLs are set an order of magnitude less than the PRQLs (assuming 100 percent solid sample
11 diluted by a factor of 100 during preparation). The Permittees shall require participating
12 laboratories to set the concentration of at least one QC or calibration standard at or below the
13 solution concentration equivalent of the PRQL. Detailed calibration procedures shall be
14 included in site certified characterization program or Permittee approved laboratory SOPs.
15

16 Completeness

17
18 Laboratory completeness shall be expressed as the number of samples analyzed with valid results
19 as a percent of the total number of samples submitted for analysis. Valid results are defined as
20 results that meet the data usability ~~useability~~ criteria based upon application of the Quality
21 ~~Control~~ QC Criteria specified in Tables B3-8 and B3-9 and meet the detection limit, calibration,
22 representativeness, and comparability criteria within this section. The Permittees shall require
23 participating laboratories to meet the completeness specified in Table B3-8.
24

25 Comparability

26
27 For metals analysis, data generated through analysis of samples from different TRU waste sites
28 shall be comparable. Comparability will be achieved by using standardized SW-846 (EPA,
29 1996) sample preparation and analytical methods that meet QAO requirements in Tables B3-8
30 and B3-9, demonstrating successful participation in a written the PDP (~~DOE, 2003~~), and use of
31 traceable standards. ~~Generator/storage sites~~ Certified characterization programs or Permittee
32 approved laboratories may use the most recent SW-846 (EPA, 1996) update. Any changes to
33 SW-846 (EPA, 1996) methodology that results in the elimination of sample preparation or
34 analytical methods in use ~~at generator/storage sites~~ by certified characterization programs or
35 Permittee approved laboratories must be addressed as a corrective action to address the
36 comparability of data before and after the SW-846 (EPA, 1996) modification.
37

38 Representativeness

39
40 Representativeness for metals analysis shall be achieved by the collection of unbiased samples
41 and the preparation of samples in the laboratory using representative and unbiased methods.
42 Samples must be collected as described in Renewal Application Appendix B1.
43

1 B3-9 Acceptable Knowledge

2 Acceptable knowledge documentation provides primarily qualitative information that cannot be
3 assessed according to specific data quality goals that are used for analytical techniques. The
4 QAOs for analytical results are described in terms of precision, accuracy, completeness,
5 comparability, and representativeness. Appropriate analytical and testing results may be used to
6 augment the characterization of wastes based on acceptable knowledge AK. To ensure that the
7 acceptable knowledge AK process is consistently applied, the Permittees shall require sites
8 certified characterization programs to comply with the following data quality requirements for
9 acceptable knowledge AK documentation:

- 11 • Precision - Precision is the agreement among a set of replicate measurements without
12 assumption of the knowledge of a true value. The qualitative determinations, such as
13 compiling and assessing acceptable knowledge AK documentation, do not lend
14 themselves to statistical evaluations of precision. However, the acceptable knowledge
15 AK information will be addressed by the independent review of acceptable knowledge
16 AK information during internal and external audits.
- 17 • Accuracy - Accuracy is the degree of agreement between an observed sample result and
18 the true value. The percentage of waste containers which require reassignment to a new
19 waste matrix code and/or designation of different HWNs hazardous waste numbers based
20 on sampling and analysis data and discrepancies identified by the Permittees during waste
21 confirmation will be reported as a measure of acceptable knowledge AK accuracy.
- 22 • Completeness - Completeness is an assessment of the number of waste streams or
23 number of samples collected to the number of samples determined to be useable through
24 the data validation process. The acceptable knowledge AK record must contain 100
25 percent of the required information in (Renewal Application Appendix B4 (TRU Mixed
26 Waste Characterization Using Acceptable Knowledge), Section B4-3). The usability
27 useability of the acceptable knowledge AK information will be assessed for completeness
28 during audits.
- 29 • Comparability - Data are considered comparable when one set of data can be compared to
30 another set of data. Comparability is ensured through sites certified characterization
31 programs meeting the training requirements and complying with the minimum standards
32 outlined for procedures that are used to implement the acceptable knowledge AK process.
33 All sites certified characterization programs must assign HWNs hazardous waste
34 numbers in accordance with Renewal Application Appendix B4, Section B4-3b and
35 provide this information regarding its waste to other sites certified characterization
36 programs ~~who~~ that store or generate a similar waste stream.
- 37 • Representativeness - Representativeness expresses the degree to which sample data
38 accurately and precisely represent characteristics of a population. Representativeness is a
39 qualitative parameter that will be satisfied by ensuring that the process of obtaining,
40 evaluating, and documenting acceptable knowledge AK information is performed in

1 accordance with the minimum standards established in Renewal Application Appendix
2 B4. Sites Certified characterization programs also must assess and document the
3 limitations of the ~~acceptable knowledge~~ AK information used to assign HWNs ~~hazardous~~
4 ~~waste numbers~~ (e.g., purpose and scope of information, date of publication, type and
5 extent to which waste parameters are addressed).

6 The Permittees shall require each ~~generator/storage site~~ certified characterization program to
7 comply with the nonconformance notification and reporting requirements of Section B3-13 if the
8 results of sampling and analysis specified in Renewal Application Chapter B are inconsistent
9 with ~~acceptable knowledge~~ AK documentation.

10
11 The Permittees shall require each site certified characterization program to address ~~quality~~
12 ~~control~~ QC by tracking its performance with regard to the use of ~~acceptable knowledge~~ AK by:
13 1) assessing the frequency of inconsistencies among information, and 2) documenting ~~acceptable~~
14 ~~knowledge~~ AK inconsistencies identified through radiography, ~~visual examination~~ VE, HSG
15 ~~headspace gas~~ analyses, and solidified waste analyses. In addition, the ~~acceptable knowledge~~
16 AK process and waste stream documentation must be evaluated through internal assessments by
17 ~~generator/storage site~~ certified characterization program quality assurance organizations and
18 assessments by auditors external to the organization (i.e., the Permittees).
19

20 B3-10 Data Review, Validation, and Verification Requirements

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

30 The Permittees shall require the sites certified characterization programs to generate the
31 following Batch Data Reports (BDRs) for data validation, verification, and quality assurance
32 activities:
33

- 34 • A Testing BDR ~~Batch Data Report~~ or equivalent includes all data pertaining to
35 radiography or ~~visual examination~~ VE for up to 20 waste containers without regard to
36 waste matrix. Table B3-11 lists all of the information required in Testing BDRs ~~Batch~~
37 ~~Data Reports~~ (identified with an "X") and other information that is necessary for data
38 validation, but is optional in Testing BDRs ~~Batch Data Reports~~ (identified with an "O").
- 39 • A Sampling BDR ~~Batch Data Report~~ or equivalent includes all sample collection data
40 pertaining to a group of no more than 20 HSG ~~headspace gas~~ or homogeneous waste
41 samples that were collected for chemical analysis. Table B3-12 lists all of the
42 information required in Sampling BDRs ~~Batch Data Reports~~ (identified with an "X") and

1 other information that is necessary for data validation, but is optional in Sampling BDRs
2 Batch Data Reports (identified with an “O”).

- 3 • An Analytical BDR ~~Batch Data Report~~ or equivalent includes analytical data from the
4 analysis of ~~TRU-mixed~~ TRU mixed waste for up to 20 HSG ~~headspace-gas~~ or
5 homogeneous waste samples. Analytical BDRs ~~Batch Data Reports~~ or equivalent that
6 contain results for composited HSG ~~headspace-gas~~ samples must contain sufficient
7 information to identify the containers that were composited for each composite sample
8 and the sample volume that was taken from each waste container. Because Analytical
9 BDRs ~~Batch Data Reports~~ are generated based on the number of samples analyzed, an
10 Analytical BDR ~~Batch Data Report~~ may contain results that are applicable to more than
11 20 containers depending on how many composite samples are part of the report, but may
12 not exceed a total of 20 samples analyzed. Table B3-13 lists all of the information
13 required in Analytical BDRs ~~Batch Data Reports~~ (identified with an “X”) and other
14 information that is necessary for data validation, but is optional in Analytical BDRs
15 Batch Data Reports (identified with an “O”).

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

- 25 • An On-line BDR ~~Batch Data Report~~ or equivalent contains the combined information
26 from the Sampling BDR ~~Batch Data Report~~ and Analytical BDR ~~Batch Data Report~~ that
27 is relevant to the on-line method used.

28 B3-10a Data Generation Level

29 The following are minimum requirements for raw data collection and management which the
30 Permittees shall require for each site certified characterization program or Permittee approved
31 laboratory:

- 32 • All raw data shall be signed and dated in reproducible ink by the person generating it.
33 Alternately, unalterable electronic signatures may be used.
- 34 • All data must be recorded clearly, legibly, and accurately in field and laboratory records
35 (bench sheets, logbooks), and include applicable sample identification numbers (for
36 Permittee approved sampling laboratories ~~sampling and analytical labs~~).

- 1 • All changes to original data must be lined out, initialed, and dated by the individual
2 making the change. A justification for changing the original data may also be included.
3 Original data must not be obliterated or otherwise disfigured so as not to be readable.
4 Data changes shall only be made by the individual who originally collected the data or an
5 individual authorized to change the data.

- 6 • All data must be transferred and reduced from field and laboratory records completely
7 and accurately.

- 8 • All field and laboratory records must be maintained as specified in Table B-6 of
9 Chapter B.

- 10 • Data must be organized into a standard format for reporting purposes (e.g., BDR ~~Batch~~
11 Data Report), as outlined in specific sampling and analytical procedures.

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

18 Data review, validation, and verification at this level involves scrutiny and signature release from
19 qualified independent technical reviewer(s)¹ as specified below. Individuals conducting this data
20 review, validation, and verification must use checklists that address all of the items included in
21 this section. Checklists must contain or reference tables showing the results of sampling,
22 analytical or on-line batch QC samples, if applicable. Checklists must reflect review of all QC
23 samples and ~~quality assurance objective~~ QAO categories in accordance with criteria established
24 in Tables B3-2 through B3-9 (as applicable to the methods validated). Completed checklists
25 must be forwarded with BDRs ~~Batch Data Reports~~ to the project level. Analytical raw data must
26 be available and reviewed by the data generation level reviewer.

27 28 B3-10a(1) Independent Technical Review

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

34
35 One hundred percent of the BDRs ~~Batch Data Reports~~ must receive an independent technical
36 review. This review shall be performed by an individual other than the data generator who is

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

1 qualified to have performed the initial work. The independent technical review must be
2 performed as soon as practicably possible in order to determine and correct negative quality
3 trends in the sampling or analytical process. However at a minimum, the independent technical
4 review must be performed before any waste associated with the data reviewed is managed,
5 stored, or disposed at WIPP, unless the data are being obtained from waste sampling and analysis
6 as containers are being retrieved or generated after initial Waste Stream Profile Form (WSPF)
7 approval as described in Renewal Application Appendix B2, Section B2-1. The reviewer(s)
8 must release the data as evidenced by signature, and as a consequence ensure the following:
9

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

- 1 • Field sampling records are complete. Incomplete or incorrect field sampling records will
2 be subject to resubmittal prior to completion of the independent technical review.
- 3 • The QAOs have been met according to the methods outlined in Sections B3-2 through
4 B3-9.

5 B3-10b Project Level

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

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

16 B3-10b(1) Certified Characterization Program Site Project Manager Review

17 The certified characterization programs Site Project Manager Review is the final validation that
18 all of the data contained in BDRs ~~Batch Data Reports~~ from the data generation level are
19 complete and have been properly reviewed as evidenced by signature release and completed
20 checklists.

21 One hundred percent of the BDRs ~~Batch Data Reports~~ must have a certified characterization
22 program Site Project Manager signature release. At a minimum, the certified characterization
23 program Site Project Manager signature release must be performed before any waste associated
24 with the data reviewed is managed, stored, or disposed at WIPP, unless the data are being
25 obtained from waste sampling and analysis as containers are being retrieved or generated as
26 described in Renewal Application Appendix B2, Section B2-1. This signature release must
27 ensure the following:

- 28 • The validity of the DAC assignment made at the data generation level based upon an
29 assessment of the data collection and evaluation necessary to make the assignment.
- 30 • Testing batch QC checks (e.g., replicate scans, measurement system checks) were
31 properly performed. Radiography data are complete and acceptable based on evidence of
32 videotape review of one waste container per day or once per testing batch, whichever is
33 less frequent, as specified in Renewal Application Appendix B1, Section B1-3.
- 34 • Sampling batch QC checks (e.g., equipment blanks, field duplicates, field reference
35 standards) were properly performed, ~~and~~ meet the established QAOs and are within
36 established data usability ~~usability~~ criteria.

- 1 • Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix spikes,
2 matrix spike duplicates, laboratory control samples) were properly performed, and meet
3 the established QAOs and are within established data usability ~~useability~~ criteria.

- 4 • On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates, on-line
5 control samples) were properly performed, and meet the established QAOs and are within
6 established data usability ~~useability~~ criteria.

- 7 • Proper procedures were followed to ensure representative samples of HSG ~~headspace gas~~
8 and homogeneous solids and soil/gravel were taken.

- 9 • Data generation level independent technical review, validation, and verification have
10 been performed as evidenced by the completed review checklists and appropriate
11 signature releases.

- 12 • Batch data review checklists are complete.

- 13 • Batch Data Reports are complete and data are properly reported (e.g., data are reported in
14 the correct units, with the correct number of significant figures, and with qualifying
15 flags).

- 16 • Verify that data are within established data assessment criteria and meet all applicable
17 QAOs (Sections B3-2 through B3-9).

18 B3-10b(2) Prepare Certified Characterization Program Site Project Manager Summary and Data
19 Validation Summary

20 To document the ~~project-level~~ project level validation and verification described above, the
21 Permittees shall require each certified characterization program Site Project Manager (or
22 designee) to prepare a certified characterization program Site Project Manager Summary and a
23 Data Validation Summary. These reports may be combined to eliminate redundancy. The
24 certified characterization program Site Project Manager Summary includes a validation checklist
25 for each BDR ~~Batch Data Report~~. Checklists for the certified characterization program Site
26 Project Manager Summary must be sufficiently detailed to validate all aspects of a BDR ~~Batch~~
27 ~~Data Report~~ that affect data quality. The Data Validation Summary provides verification that, on
28 a per waste container or sample basis as evidenced by BDR ~~Batch Data Report~~ reviews, all data
29 have been validated in accordance with the site certified characterization program QAPjP. The
30 Data Validation Summary must identify each BDR ~~Batch Data Report~~ reviewed (including all
31 waste container numbers), describe how the validation was performed and whether or not
32 problems were detected (e.g., NCRs ~~nonconformance reports~~), and include a statement indicating
33 that all data are acceptable. Summaries must include release signatures.

34
35 ~~Once the data have received project-level validation and verification or when the Site Project~~
36 ~~Manager decides the sample no longer needs to be retained, the Site Project Manager must~~
37 ~~ensure that the laboratory is notified. Samples must be retained by the laboratory until this~~

1 notification is received. ~~Gas sample canisters may then be released from storage for cleaning,~~
2 ~~recertification, and subsequent reuse.~~ Sample tags must be removed and retained in the project
3 files before recycling the canisters. If the certified characterization program Site Project
4 Manager requests that samples or canisters be retained for future use (e.g., an experimental
5 holding time study), the same sample identification and COC forms shall be used and cross-
6 referenced to a document which specifies the purpose for sample or canister retention.

7
8 B3-10b(3) Prepare Waste Stream Characterization Package

9 In the event the Permittees request detailed information on a waste stream, the certified
10 characterization program Site Project Manager will provide a Waste Stream Characterization
11 Package. The certified characterization program Site Project Manager must ensure that the
12 Waste Stream Characterization Package (Section B3-12b(3)) will support waste characterization
13 determinations.

14
15 B3-10c Permittee Level

16 The final level of data verification occurs at the Permittee level and must, at a minimum, consist
17 of reviewing a sample of the BDRs ~~Batch Data Reports~~ during audits of ~~generator/storage sites~~
18 certified characterization programs and Permittee approved laboratories to verify completeness.
19 During such audits, the Permittees are responsible for the verification that BDRs ~~Batch Data~~
20 ~~Reports~~ include the following:

- 21 • Project-level Project level signature releases
- 22
- 23 • Listing of all waste containers being presented in the report
- 24 • Listing of all testing, sampling, and analytical batch numbers associated with each waste
25 container being reported in the package
- 26 • Analytical BDR ~~Batch Data Report~~ case narratives
- 27 • Certified Characterization Program Site Project Manager Summary
- 28 • Data Validation Summary
- 29 • Complete summarized qualitative and quantitative data for all waste containers with data
30 flags and qualifiers-

31 For each ~~Waste Stream Profile Form (WSPF)~~ WSPF submitted for approval, the Permittees must
32 verify that each submittal (i.e., WSPF and Characterization Information Summary) is complete
33 and notify the originating site certified characterization program in writing of the WSPF
34 approval. The Permittees will maintain the data as appropriate for use in the regulatory
35 compliance programs. For subsequent shipments made after the initial WSPF approval, the
36 verification will also include WIPP Waste Information System (WWIS) ~~WWIS~~ internal limit
37 checks (Chapter B, Section B-5a(1)).

1
2 B3-11 Reconciliation with Data Quality Objectives

3 Reconciling the results of waste testing and analysis with the DQOs provides a way to ensure
4 that data will be of adequate quality to support the regulatory compliance programs.
5 Reconciliation with the DQOs will take place at both the project level and the Permittees'
6 Permittee level. At the project level, reconciliation will be performed by the certified
7 characterization program Site Project Manager as described in Section B3-11a, while at the
8 Permittees' Permittee level, reconciliation will be performed as described in Section B3-11b
9 below.

10
11 B3-11a Reconciliation at the Project Level

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

24
25 For each waste stream characterized, the Permittees shall require each certified characterization
26 program Site Project Manager to determine if sufficient data have been collected to determine
27 the following WAP required WAP required waste parameters, as applicable:

- 28
29
- 30 • Waste matrix code
 - 31 • Waste material parameter weights
 - 32 • If each waste container of waste contains TRU radioactive waste
 - 33 • Mean concentrations, 90 percent upper confidence limit UCL₉₀ (UCL₉₀) for the mean
34 concentrations, standard deviations, and the number of samples collected for each VOC
in the HSG headspace gas of waste containers in the waste stream
 - 35 • Mean concentrations, UCL₉₀ for the mean concentrations, standard deviations, and
36 number of samples collected for VOCs, SVOCs, and metals in the waste stream
 - 37 • Whether the waste stream exhibits a ~~toxicity characteristic (TC)~~ TC under 40 CFR Part
38 261, Subpart C

- 1 • Whether the waste stream contains listed waste found in 20.4.1.200 NMAC incorporating
2 40 CFR Part 261, Subpart D
- 3 • Whether the waste stream can be classified as hazardous or nonhazardous at the 90-
4 percent confidence level
- 5 • Whether an appropriate packaging configuration and DAC were applied and documented
6 in the HSG headspace gas sampling documentation, and whether the drum age DAC was
7 met prior to sampling-
- 8 • Whether all TICs were appropriately identified and reported in accordance with the
9 requirements of Section B3-1 prior to submittal of a WSPF for a waste stream or waste
10 stream lot-
- 11 • Whether the overall completeness, comparability, and representativeness QAOs were met
12 for each of the analytical and testing procedures as specified in Sections B3-2 through
13 B3-9 prior to submittal of a WSPF for a waste stream or waste stream lot-
- 14 • Whether the PRQLs for all analyses were met prior to submittal of a WSPF for a waste
15 stream or waste stream lot-

16 If the certified characterization program Site Project Manager determines that insufficient data
17 have been collected to make the determinations listed above, additional data collection efforts
18 must be undertaken. The reconciliation of a waste stream shall be performed, as described in
19 Renewal Application Appendix B4, prior to submittal of WSPF and Characterization
20 Information Summary to the Permittees for that waste stream. The Permittees shall not manage,
21 store, or dispose a TRU mixed waste stream at WIPP unless the certified characterization
22 program Site Project Manager determines that the ~~WAP required~~ WAP required waste
23 parameters listed above have been met for that waste stream.

24
25 The statistical procedure presented in Renewal Application Appendix B2 shall be used by
26 participating certified characterization program Site Project Managers to evaluate and report
27 waste characterization data from the analysis of homogeneous solids and soil/gravel. The
28 procedure, which calculates UCL₉₀ values, shall be used to assess compliance with the DQOs in
29 Renewal Application Chapter B, Section B-4a(1) as well as with Resource Conservation and
30 Recovery Act (RCRA) RCRA regulations. The procedure must be applied to all laboratory
31 analytical data for total VOCs, total SVOCs, and total metals. For RCRA regulatory compliance
32 (40 CFR § 261.24), data from the analysis of the appropriate metals and organic compounds
33 shall be expressed as ~~toxicity characteristic leaching procedure (TCLP)~~ TCLP values or results
34 may also be compared to the TC levels expressed as total values. These total values will be
35 considered the regulatory threshold limit (RTL) values for the WAP. The RTL values are
36 obtained by calculating the weight/weight concentration (in the solid) of a TC analyte that would
37 give the regulatory weight/volume concentration (in the TCLP extract), assuming 100-percent
38 analyte dissolution.

1 B3-11b Reconciliation at the Permittee Level

2 The Permittees must also ensure that data of sufficient type, quality, and quantity are collected to
3 meet WAP DQOs. The Permittees will ensure sufficient data have been collected to determine if
4 the waste characterization information is adequate to demonstrate the Permittees' compliance
5 with Renewal Application Chapter B, Section B-4a(1). This is performed during Permittees'
6 review of the WSPF and Characterization Information Summary.
7

8 B3-12 Data Reporting Requirements

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

13 B3-12a Data Generation Level

14 Data shall be transmitted by hard copy or electronically (provided a hard copy is available on
15 demand) from the data generation level to the project level. Transmitted data shall include all
16 ~~BDRs Batch Data Reports~~ and data review checklists. The ~~BDRs Batch Data Reports~~ and
17 checklists used must contain all of the information required by the testing, sampling, and
18 analytical techniques described in Renewal Application Appendixes B1 through B6-, as well as
19 the signature releases to document the review, validation, and verification as described in Section
20 B3-10. All ~~BDRs Batch Data Reports~~ and checklists shall be in approved formats, as provided in
21 TRU waste site-specific documentation.
22

23 Batch Data Reports shall be forwarded to the certified characterization program Site Project
24 Manager. All ~~BDRs Batch Data Reports~~ shall be assigned serial numbers, and each page shall be
25 numbered. The serial number used for ~~BDRs Batch Data Reports~~ can be the same as the testing,
26 sampling, or analytical batch number.
27

28 ~~The~~ QA documentation, including raw data, shall be maintained in ~~either testing, sampling, and~~
29 ~~analytical facility files, or~~ certified characterization program or Permittee approved laboratory
30 ~~site project files for those facilities located on site~~ in accordance with the document storage
31 requirements of site-approved site certified characterization program QAPjPs. Permittee
32 approved laboratories shall forward testing, sampling, and analytical QA documentation along
33 with ~~BDRs Batch Data Reports~~ to the certified characterization program site project office for
34 inclusion in site project files.
35

36 B3-12b Project Level

37 The certified characterization program ~~site project office~~ shall prepare a WSPF for each waste
38 stream certified for shipment to WIPP based on information obtained from ~~acceptable knowledge~~
39 AK and ~~BDRs Batch Data Reports~~, if applicable. In addition, the certified characterization
40 program ~~site project office~~ must ensure that the Characterization Information Summary and the
41 Waste Stream Characterization Package (when requested by the Permittees) are prepared as
42 appropriate. The certified characterization program Site Project Manager must also verify these

1 reports are consistent with information found in analytical batch reports. Summarized testing,
2 sampling, and analytical data are included in the Characterization Information Summary. The
3 contents of the WSPF, Characterization Information Summary, and Waste Stream
4 Characterization Package are discussed in the following sections.

5
6 After approval of a WSPF and the associated Characterization Information Summary by the
7 Permittees, the generator/storage site certified characterization program ~~are~~ is required to
8 maintain a cross-reference of container identification numbers to each BDR ~~Batch Data Report~~.

9
10 A Waste Stream Characterization Package shall be transmitted by hard copy or electronically
11 from the certified characterization program Site Project Manager to the Permittees when
12 requested.

13
14 B3-12b(1) Waste Stream Profile Form

15 The WSPF ~~Waste Stream Profile Form (WSPF, (~~ Renewal Application Chapter B, Figure B-1)
16 shall include the following information:

- 17
- 18 • Generator/storage TRU waste site name
 - 19 • Generator/storage site TRU waste site EPA ID
 - 20 • Date of certified characterization program audit report approval by New Mexico
21 Environment Department (NMED) ~~NMED~~ if obtained)
 - 22 • Original generator of waste stream
 - 23 • Whether waste is Contact-Handled or Remote-Handled
 - 24 • The Waste Stream WIPP Identification Number
 - 25 • Summary Category Group
 - 26 • Waste Matrix Code Group
 - 27 • Waste Material Parameter Weight Estimates per unit of waste
 - 28 • Waste stream name
 - 29 • A description of the waste stream
 - 30 • Applicable EPA HWNs ~~hazardous waste numbers~~
 - 31 • Applicable TRUCON codes
 - 32 • A listing of acceptable knowledge AK documentation used to identify the waste stream

- 1 • The waste characterization procedures used and the reference and date of the procedure
- 2 • Certification signature of certified characterization program Site Project Manager, name,
3 title, and date signed

4 B3-12b(2) Characterization Information Summary

5 The Characterization Information Summary shall include the following elements, if applicable:
6

- 7 • Data reconciliation with DQOs
- 8 • The HSG ~~Headspace gas~~ summary data listing the identification numbers of samples used
9 in the statistical reduction, the maximum, mean, standard deviation, UCL₉₀, RTL, and
10 associated EPA HWNs ~~hazardous waste numbers~~ that must be applied to the waste
11 stream:-
- 12 • Total metal, VOC, and SVOC analytical results for homogeneous solids and soil/gravel
13 (if applicable):-
- 14 • The TIC listing and evaluation:-
- 15 • Radiography and ~~visual examination~~ VE summary to document that all prohibited items
16 are absent in the waste (if applicable):-
- 17 • A complete listing of all container identification numbers used to generate the WSPF,
18 cross-referenced to each BDR ~~Batch Data Report~~
- 19 • Complete AK summary, including stream name and number, point of generation, waste
20 stream volume (current and projected), generation dates, TRUCON codes, Summary
21 Category Group, Waste Matrix Code(s) and Waste Matrix Code Group, other TWBIR
22 relevant inventory information, waste stream description, areas of operation, generating
23 processes, RCRA determinations, radionuclide information, all references used to
24 generate the AK summary, and any other information required by Renewal Application
25 Appendix B4, Section B4-2b:-
- 26 • Method for determining Waste Material Parameter Weights per unit of waste, when
27 necessary:-
- 28 • List of any AK Acceptable Knowledge Sufficiency Determinations requested for the
29 waste stream:-
- 30 • Certification through ~~acceptable knowledge~~ AK or testing and/or analysis that any waste
31 assigned the ~~hazardous waste number~~ HWN of U134 (hydrofluoric acid) no longer
32 exhibits the characteristic of corrosivity: (This is verified by ensuring that no liquid is
33 present in U134 waste):-

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

2 The Waste Stream Characterization Package includes the following information:

- 3
- 4 • The WSPF ~~Waste Stream Profile Form (WSPF,~~ (Section B3-12b(1))
 - 5 • Accompanying Characterization Information Summary (Section B3-12b(2))
 - 6 • Complete AK summary (Section B3-12b(2))
 - 7 • The BDRs ~~Batch Data Reports~~ supporting the characterization of the waste stream and
 - 8 any others requested by the Permittees
 - 9 • Raw analytical data requested by the Permittees

10 B3-12b(4) WIPP Waste Information System (~~WWIS~~) Data Reporting

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

18

19 B3-13 Nonconformances

20 The Permittees shall require the status of ~~work and the~~ WAP activities at participating
21 ~~generator/storage~~ TRU waste sites to be monitored and controlled by the certified
22 characterization program Site Project Manager. This monitoring and control shall include
23 nonconformance identification, documentation, and reporting.

24

25 The nonconformances and corrective action processes specified in this section describe
26 procedures between the Permittees and the ~~generator/storage sites~~ certified characterization
27 programs.

28

29 Nonconformances

30

31 Nonconformances are uncontrolled and unapproved deviations from an approved plan or
32 procedure. Nonconforming items and activities are those that do not meet the WAP
33 requirements, procurement document criteria, or approved work procedures. Nonconforming
34 items shall be identified by marking, tagging, or segregating, and the affected ~~generator/storage~~
35 ~~site(s)~~ certified characterization program(s) shall be notified. The Permittees shall require
36 participating sites certified characterization programs to reconcile and correct nonconforming
37 items as appropriate in accordance with the Permittees' Quality Assurance Program Description
38 (QAPD). Disposition of nonconforming items shall be identified and documented. The QAPjPs

1 shall identify the person(s) responsible for evaluating and dispositioning nonconforming items
2 and shall include referenced procedures for handling them.
3

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

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

14 A NCR nonconformance report shall be prepared for each nonconformance identified. Each
15 ~~nonconformance report~~ NCR shall be initiated by the individual(s) identifying the
16 nonconformance. The ~~nonconformance report~~ NCR shall then be processed by knowledgeable
17 and appropriate personnel. For this purpose, a ~~nonconformance report~~ NCR including, or
18 referencing as appropriate, results of laboratory analysis, QC tests, audit reports, internal
19 memoranda, or letters shall be prepared. The ~~nonconformance report~~ NCR must provide the
20 following information:
21

- 22 • Identification of the individual(s) identifying or originating the nonconformance
- 23 • Description of the nonconformance
- 24 • Method(s) or suggestions for correcting the nonconformance (corrective action)
- 25 • Schedule for completing the corrective action
- 26 • An indication of the potential ramifications and overall usability ~~useability~~ the data, if
27 applicable
- 28 • Any approval signatures specified in the site certified characterization program
29 nonconformance procedures

30 The Permittees shall require the certified characterization program Site Project Manager to
31 oversee the ~~NCR nonconformance report~~ process and be responsible for developing a plan to
32 identify and track all nonconformances and report this information to the Permittees. The
33 certified characterization program Site Project Manager is also responsible for notifying project
34 personnel of the nonconformance and verifying completion of the corrective action for
35 nonconformances.
36
37

1 Nonconformance to DQOs

2
3 For any non-administrative nonconformance related to applicable requirements specified in this
4 WAP which are first identified at the certified characterization program Site Project Manager
5 signature release level (i.e., a failure to meet a ~~data quality objective~~ DQO), the Permittees shall
6 receive written notification within seven (7) ~~five (5)~~ calendar days of identification and shall also
7 receive a ~~nonconformance report~~ NCR within ~~thirty (30)~~ calendar days of identification of the
8 incident. The Permittees shall require the ~~generator/storage site~~ certified characterization
9 program to implement a corrective action which remedies the nonconformance prior to
10 management, storage, or disposal of the waste at WIPP. The Permittees shall send NMED a
11 monthly summary of nonconformances identified during the previous month, indicating the
12 number of nonconformances received and the ~~generator/storage sites~~ certified characterization
13 programs responsible.

14
15 Permittees' Corrective Action Process

16
17 The Permittees shall initiate a corrective action process when internal nonconformances and
18 nonconformances at the ~~generator/storage~~ certified characterization program or Permittee
19 approved laboratory sites are identified. Activities and processes that do not meet requirements
20 are documented as deficiencies.

21
22 When a deficiency is identified by the Permittees, the following process action steps are
23 required:

- 24
- 25 • The condition is documented on a Corrective Action Report (**CAR**) by the individual
26 identifying the problem.
 - 27 • The Permittees have designated the CAR Initiator and Assessment Team Leader to
28 review the CAR, determine validity of the finding (determine that a requirement has been
29 violated), classify the significance of the condition, assign a response due date, and issue
30 the CAR to the responsible party.
 - 31 • The responsible organization reviews the CAR, evaluates the extent and cause of the
32 deficiency and provides a response to the Permittees, indicating remedial actions and
33 actions to preclude recurrence that will be taken.
 - 34 • The Permittees review the response from the responsible organization and, if acceptable,
35 communicate the acceptance to the responsible organization.
 - 36 • The responsible organization completes remedial actions and actions to preclude
37 recurrence of the condition.
 - 38 • After all corrective actions have been completed, the Permittees schedule and perform a
39 verification to ensure that corrective actions have been completed and are effective.
40 When all actions have been completed and verified as being effective, the CAR is closed
41 by the CAR Initiator and Assessment Team Leader on behalf of the Permittees.

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

B3-14 Special Training Requirements and Certifications

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

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

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

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

B3-15 Changes to Waste Analysis Plan WAP-Related Plans or Procedures

Controlled changes to ~~WAP-related~~ WAP related plans or procedures shall be managed through the document control process described in the QAPD. The certified characterization program Site Project Manager shall review all non-administrative changes and evaluate whether those

1 changes could impact DQOs specified in the Renewal Application. After site characterization
2 program certification, any changes to ~~WAP-related~~ WAP related plans or procedures that could
3 positively or negatively impact DQOs (i.e., those changes that require prior approval of the
4 Permittees as defined in Appendix B5, Section B5-2) shall be reported to the Permittees within
5 seven (7) ~~five (5)~~ days of identification by the project level review. The Permittees shall send
6 NMED a monthly summary briefly describing the changes to plans and procedures identified
7 pursuant to this section during the previous month.
8

1 B3-16 List of References

2 Currie, Lloyd A. 1968. "Limits for Qualitative Detection and Quantitative Determination."
3 Analytical Chemistry, No. 40: pp. 586-93.

4
5 ~~DOE, 2001. WIPP Waste Information System User's Manual for Use by Shippers/Generators.~~
6 ~~DOE/CAO 97-2273, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S.~~
7 ~~Department of Energy.~~

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Waste Isolation Pilot Plant
Hazardous Waste Facility Permit
Draft Renewal Application
May 2009

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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)	For example: 55-gal (208-L) drums
Plastics (packaging materials)	For example: 90-mil polyethylene drum liner and plastic bags

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

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

^a Criteria apply to PRQL concentrations.

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

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

^d The ethyl benzene PRQL for FTIRS is 20 ppm

CAS = Chemical Abstract Service

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

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

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

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

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet method QAOs	Repeat until acceptable
Laboratory duplicates or on-line duplicates	One (1) per analytical batch or on-line batch	RPD \leq 25 ^b	Nonconformance if RPD >25
Laboratory blanks or on-line blanks	Daily prior to sample analysis for GC/MS and GC/FID. Otherwise, daily prior to sample analysis and one (1) per analytical batch or on-line	Analyte amounts \leq 3 x MDLs for GC/MS and GC/FID; \leq 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 \leq 25 ^b	Nonconformance if RPD > 25
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan
GC/MS	BFB Tune Every 12 hours	Abundance criteria for key ions are met	Repeat until acceptable
GC/MS	Minimum 5-point initial calibration (minimum of five standards) Initially as needed	%RSD of response factor for each target analyte <36	Repeat until acceptable
GC/MS	Continuing calibration every 12 hours	%D for all target analytes < 30 of initial calibration	Repeat until acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
GC/FID	Minimum 3-point calibration (minimum three standards) Initially and as needed	Correlation coefficient > 0.99 or %RSD < 20 for each target analyte and the retention time of each target analyte within an acceptance criteria defined in the method	Repeat until acceptable
GC/FIC	Continuing calibration every 12 hours	%RSD < 15%	Repeat until acceptable

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2 ^a Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.
3 ^b Applies only to concentrations greater than the PRQLs listed in Table B3-2.
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- 5 GC/FID = Gas Chromatography/Flame Ionization Detector
- 6 MDL = Method Detection Limit
- 7 QAO = Quality Assurance Objective
- 8 PDP = ~~Performance Demonstration Program~~ performance demonstration program
- 9 PRQL = Program Required Quantitation Limit
- 10 %R = Percent Recovery
- 11 RPD = Relative Percent Difference
- 12 BFB = 4-Bromoflourobenzene
- 13 %D = Percent difference
- 14 %RSD = Percent relative standard deviation

TABLE B3-4
VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST
AND QUALITY ASSURANCE OBJECTIVES FOR SOLIDS ANALYSIS

Compound	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL ^b (mg/kg)	PRQL ^b (mg/kg)	Completeness (%)
Benzene	71-43-2	≤ 45	37-151	1	10	90
Bromoform	75-25-2	≤ 47	45-169	±	40	90
Carbon disulfide	75-15-0	≤ 50	60-150	±	40	90
Carbon tetrachloride	56-23-5	≤ 30	70-140	1	10	90
Chlorobenzene	108-90-7	≤ 38	37-160	1	10	90
Chloroform	67-66-3	≤ 44	51-138	1	10	90
1,4-Dichlorobenzene ^c	106-46-7	≤ 60	18-190	1	10	90
ortho-Dichlorobenzene^e	95-50-1	≤ 60	18-190	±	40	90
1,2-Dichloroethane	107-06-2	≤ 42	49-155	1	10	90
1,1-Dichloroethylene	75-35-4	≤ 250	D-234 ^d	1	10	90
trans-1,2-Dichloroethylene	156-60-5	≤ 50	60-150	±	40	90
Ethyl benzene	100-41-4	≤ 43	37-162	±	40	90
Methylene chloride	75-09-2	≤ 50	D-221 ^d	±	40	90
1,1,2,2-Tetrachloroethane	79-34-5	≤ 55	46-157	±	40	90
Tetrachloroethylene	127-18-4	≤ 29	64-148	1	10	90
Toluene	108-88-3	≤ 29	47-150	±	40	90
1,1,1-Trichloroethane	71-55-6	≤ 33	52-162	±	40	90
1,1,2-Trichloroethane	79-00-5	≤ 38	52-150	±	40	90
Trichloroethylene	79-01-6	≤ 36	71-157	1	10	90
Trichlorofluoromethane	75-69-4	≤ 110	17-181	±	40	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤ 50	60-150	±	40	90
Vinyl chloride	75-01-4	≤ 200	D-251 ^d	1	4	90
m-xylene	108-38-3	≤ 50	60-150	±	40	90
o-xylene	95-47-6	≤ 50	60-150	±	40	90
p-xylene	106-42-3	≤ 50	60-150	±	40	90
Acetone	67-64-1	≤ 50	60-150	10 ^e	100	90
Butanol	71-36-3	≤ 50	60-150	10 ^e	100	90
Ethyl ether	60-29-7	≤ 50	60-150	10 ^e	100	90
Formaldehyde ^f	50-00-0	≤ 50	60-150	10 ^e	100	90
Hydrazine ^g	302-01-2	≤ 50	60-150	10 ^e	100	90
Isobutanol	78-83-1	≤ 50	60-150	10 ^e	100	90
Methanol	67-56-1	≤ 50	60-150	10 ^e	100	90
Methyl ethyl ketone	78-93-3	≤ 50	60-150	10 ^e	100	90
Pyridine ^c	110-86-1	≤ 50	60-150	10 ^e	100	90

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

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

^c Can also be analyzed as a ~~semi-volatile organic compound~~ SVOC. If analyzed as a ~~semi-volatile compound~~ SVOC, the QAOs of Table B3-6 apply.

^d Detected; result must be greater than zero.

^e Estimate, to be determined.

^f ~~Required only for homogeneous solids and soil/gravel waste from Savannah River Site, if analysis is required to resolve assignment of EPA hazardous waste numbers.~~

^g ~~Required only for homogeneous solids and soil/gravel waste from Oak Ridge National Laboratory and Savannah River Site, if analysis is required to resolve assignment of EPA hazardous waste numbers.~~

CAS = Chemical Abstract Service

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

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

Waste Isolation Pilot Plant
Hazardous Waste Facility Permit
Draft Renewal Application
May 2009

1 PRQL = Program required quantitation limit; calculated from the ~~toxicity characteristic~~ **TC** level for
2 benzene assuming a 0.9 oz (25-gram [g]) sample, 0.1 gal (0.5 liter [L]) of extraction fluid, and 100
3 percent analyte extraction (milligrams per kilogram)

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

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-4 QAOs	Repeat until acceptable
Laboratory duplicates ^b	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 ^b	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 (EPA, 1996) Method requirements: %RSD for calibration check compounds (CCC) ≤ 30, %RSD for all other compounds ≤ 15% Relative Average response factor (RRF) used if %RSD ≤ 15, use linear regression if %RSD > 15; R or R_t² ≥ 0.990 if using alternative curve System Performance Check Compound (SPCC) minimum RRF as per SW-846 (EPA, 1996) Method; RRF for all other compounds ≥ 0.01	Repeat until acceptable

Waste Isolation Pilot Plant
 Hazardous Waste Facility Permit
 Draft Renewal Application
 May 2009

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
GC/MS Calibration (continued)	Continuing Calibration every 12 hours	%D ≤ 20 for CCC; SPCC minimum RRF as per SW-846 <u>(EPA, 1996)</u> Method; RRF for all other compounds ≥ 0.01 <u>Retention Time (RT)</u> 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
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 ± 3 standard deviations from initial RT calibration per applicable SW-846 <u>(EPA, 1996)</u> Method	Repeat until acceptable.
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples for a given matrix ±3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviation) or > (average %R + 3 standard deviation)
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

^a Corrective Action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.

Nonconformances do not apply to matrix related exceedances.

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

~~CCC~~ = ~~Calibration check compounds~~

GC/FID = Gas Chromatography/Flame Ionization Detector

MDL = Method detection limit

QAO = Quality assurance objective

PDP = ~~Performance Demonstration Program~~ performance demonstration program

%R = Percent recovery

RPD = Relative percent difference

TABLE B3-6
SEMI-VOLATILE ORGANIC COMPOUND TARGET ANALYTE LIST
AND QUALITY ASSURANCE OBJECTIVES FOR SOLIDS ANALYSIS

Compound	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL ^b (mg/kg)	PRQL ^b (mg/kg)	Completeness (%)
Cresols	1319-77-3	≤ 50	25-115	5	40	90
1,4-Dichlorobenzene ^c	106-46-7	≤ 86	20-124	5	40	90
ortho-Dichlorobenzene^e	95-50-1	≤ 64	32-129	5	40	90
2,4-Dinitrophenol	51-28-5	≤ 119	D-172^d	5	40	90
2,4-Dinitrotoluene	121-14-2	≤ 46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤ 319	D-152 ^d	0.3	2.6	90
<u>Hexachlorobutadiene</u>	<u>87-68-3</u>	<u>≤ 64</u>	<u>24-117</u>	<u>2</u>	<u>10</u>	<u>90</u>
Hexachloroethane	67-72-1	≤ 44	40-113	5	40	90
Nitrobenzene	98-95-3	≤ 72	35-180	5	40	90
Pentachlorophenol	87-86-5	≤ 128	14-176	5	40	90
Pyridine ^c	110-86-1	≤ 50	25-115	5	40	90

- 5
 6 CAS = Chemical Abstract Service
 7 %RSD = Percent relative standard deviation
 8 RPD = Relative percent difference
 9 %R = Percent recovery
 10 MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)
 11 PRQL = Program required quantitation limit; calculated from the toxicity characteristic TC level for
 12 nitrobenzene assuming a 100-gram (g) sample, 0.5 gal (2 liter [L]) of extraction fluid, and 100
 13 percent analyte extraction (milligrams per kilograms)
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15 ^a Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material
 16 which has established statistical control limits is used, then the established control limits for that material should be
 17 used for accuracy requirements.

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

19 ^c Can also be analyzed as a ~~volatile organic compound~~ VOC

20 ^d Detected; result must be greater than zero

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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 ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-6 QAOs	Repeat until acceptable
Laboratory duplicates ^b	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 (EPA, 1996) Method requirements: %RSD for CCC ≤ 30, %RSD for all other compounds ≤ 15% Average response factor (RRF) used if %RSD ≤ 15, use linear regression if >15; R or R ² ≥ 0.990 if using alternative curve System Performance Check Compound (SPCC) minimum RRF as per SW-846 (EPA, 1996) Method; RRF for all other compounds ≥ 0.01 %D ≤ 20 for CCC, SPCC minimum RRF as per SW-846 (EPA, 1996) Method; RRF for all other compounds ≥ 0.01 RT for internal standard must be ± 30 seconds from last daily calibration, internal standard area count must be >50% and <200% of last daily calibration	Repeat until acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
GC/ECD Calibration	5-pt. Calibration initially and as needed Continuing Calibration every 12 hours	Correlation Coefficient \geq 0.990 or %RSD < 20 for all analytes %D or %Drift for all analytes \leq 15 of expected values, RT \pm 3 standard deviations of initial RT calibration per applicable SW-846 (EPA, 1996) Method	Repeat until acceptable
Matrix spike duplicates	One (4) 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 (4) 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

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- 2 ^a Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.
- 3 Nonconformances do not apply to matrix related exceedances.
- 4 ^b May be satisfied by using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the
- 5 PRLs listed in Table B3-6.
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- 7 MDL = Method Detection Limit
- 8 QAO = Quality Assurance Objective
- 9 PDP = ~~Performance Demonstration Program~~ performance demonstration program
- 10 %D = Percent Difference
- 11 %R = Percent Recovery
- 12 RPD = Relative Percent Difference
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**TABLE B3-8
METALS TARGET ANALYTE LIST
AND QUALITY ASSURANCE OBJECTIVES**

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

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^a ≤ 30 percent control limits apply when sample and duplicate concentrations are ≥ 10 x IDL for Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) ICP-AES and Atomic Absorption (AA)-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.

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

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

^d PRDL set such that it is a factor of 10 below the PRQL for 100 percent solid samples, assuming a 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)

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

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-8 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	$\leq 3 \times \text{IDL}$ ($\leq 5 \times \text{IDL}$ for ICP-MS) ^b	Redigest and reanalyze any samples with analyte concentrations which are $\leq 10 \times$ blank value and $\geq 0.5 \times$ PRQL
Matrix spikes	One (1) per analytical batch	Meet Table 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, CVAA, GFAA, HAA	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 (EPA, 1996) Method 6020 criteria	Nonconformance if not reanalyzed at 5 X dilution until criteria are met

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action ^a
Serial Dilution (ICP, ICP-MS)	One (1) per analytical batch	5 X dilution must be $\leq 10\%$ D of initial value for sample $> 50 \times \text{IDL}$	Flag Data if $> 10\%$ and $> 50 \times \text{IDL}$
Interference Correction Verification (ICP, ICP-MS)	Beginning and end of run or every 12 hours (8 for ICP) whichever is more frequent	80-120% recovery for analytes Note: Acceptance Criteria and Corrective Action apply only if interferences found in samples at levels greater than ICS A Solution	Correct problem and recalibrate, nonconformance if not corrected
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

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2 ^a Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.
3 Nonconformances do not apply to matrix related exceedances.
4 ^b Applies only to concentrations greater than the PRQLs listed in Table B3-8.
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- 6 CVAA = Cold vapor atomic absorption
- 7 DFTPP = Decafluorotriphenylphosphine
- 8 FLAA = Flame atomic absorption
- 9 GFAA = Graphite furnace atomic absorption
- 10 HAA = Hydride-generation atomic absorption
- 11 IDL = Instrument Detection Limit
- 12 PDP = ~~Performance Demonstration Program~~ performance demonstration program
- 13 PRQL = Program Required Quantitation Limit
- 14 %R = Percent Recovery
- 15 RPD = Relative Percent Difference

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**TABLE B3-10
 MINIMUM TRAINING AND QUALIFICATIONS REQUIREMENTS ^a**

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

4 ^a Based on requirements contained in USEPA Contract Laboratory Program Statement of Work for Organics
 5 Analysis (Document Number OLM 01.0) and Statement of Work for Inorganics Analysis (Document Number ILM
 6 03.0).

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

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

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

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

Required Information	Radiography	Visual Examination	Comment
Operator signature and date of test	X	X	Signatures of both operators required for Visual Verification of Acceptable Knowledge <u>AK</u>
Data review checklists	X	X	All data review checklists will be identified

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LEGEND:

X - Required in BDR batch data report.

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

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**TABLE B3-12
SAMPLING BATCH DATA REPORT CONTENTS**

Required Information	Headspace Gas	Solid Sampling	Comment
BDR 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
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.

Waste Isolation Pilot Plant
 Hazardous Waste Facility Permit
 Draft Renewal Application
 May 2009

Required Information	Headspace Gas	Solid Sampling	Comment
Cross-reference of sampling equipment numbers with associated cleaning batch numbers	O	X	As applicable to the equipment used for the sampling. For disposable equipment, a reference to the lot and procurement records to support cleanliness is sufficient
Drum age	X		
Equilibration time	X		
Verification of rigid liner venting	X		Only applicable to containers with rigid liners
Verification that sample volume taken is small in comparison to the available volume	X		Must include HSG 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.
Organic Vapor Analyzer (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

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LEGEND:

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

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**TABLE B3-13
 ANALYTICAL BATCH DATA REPORT CONTENTS**

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

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LEGEND:

X - Required in ~~BDR batch data report~~.

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

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**TABLE B3-14
 DATA REPORTING FLAGS**

DATA FLAG	INDICATOR
B	Analyte detected in blank (Organics/ <u>HSGs</u> 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/ <u>HSGs</u> Headspace-gases)
J	Analyte less than PRQL but greater than or equal to MDL (Organics/ <u>HSGs</u> 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/ <u>HSGs</u> Headspace-gases)
Z	One or more QC samples do not meet acceptance criteria
H	Holding time exceeded

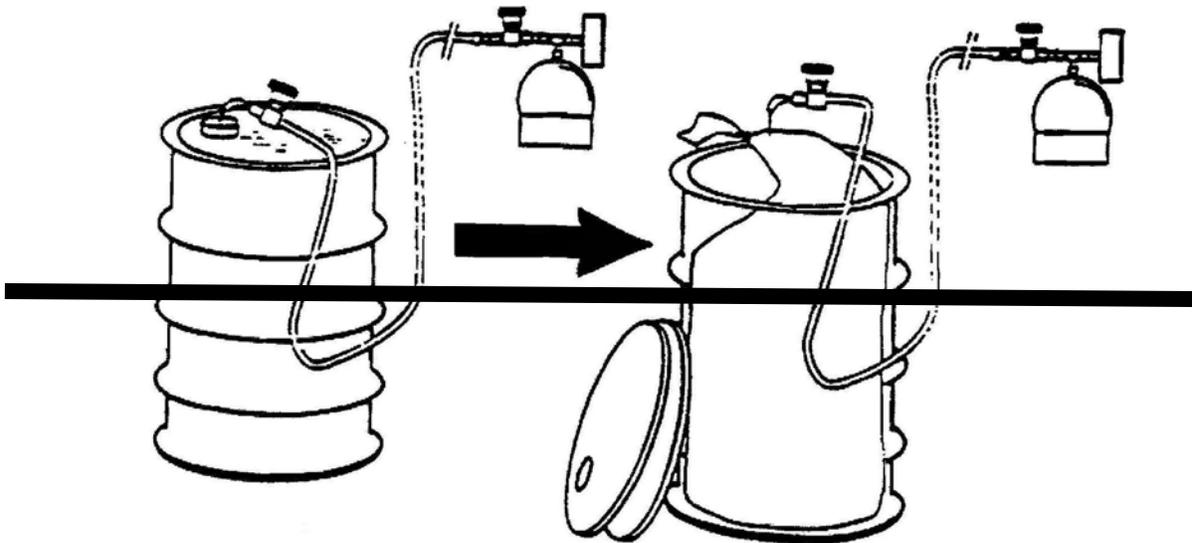
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FIGURES

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