

**ATTACHMENT B3**

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

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

**TABLE OF CONTENTS**

|   |                        |
|---|------------------------|
| List of Tables .....  | <a href="#">B3-iii</a> |
| List of Figures .....   | <a href="#">B3-iii</a> |
| B3-1 <u>Validation Methods</u> .....  | <a href="#">B3-1</a>   |
| B3-2 <u>Headspace-Gas Sampling</u> .....  | <a href="#">B3-7</a>   |
| B3-3 <u>Sampling of Homogeneous Solids and Soils/Gravel</u> .....   | <a href="#">B3-9</a>   |
| B3-4 <u>Radiography</u> .....   | <a href="#">B3-11</a>  |
| B3-5 <u>4</u> <u>Gas Volatile Organic Compound Analysis</u> .....   | <a href="#">B3-12</a>  |
| B3-6 <u>5</u> <u>Total Volatile Organic Compound Analysis</u> .....   | <a href="#">B3-14</a>  |
| B3-7 <u>6</u> <u>Total Semivolatile Organic Compound Analysis</u> .....   | <a href="#">B3-15</a>  |
| B3-8 <u>7</u> <u>Total Metal Analysis</u> .....   | <a href="#">B3-17</a>  |
| B3-9 <u>8</u> <u>Acceptable Knowledge</u> .....   | <a href="#">B3-19</a>  |
| B3-10 <u>9</u> <u>Data Review, Validation, and Verification Requirements</u> .....  | <a href="#">B3-20</a>  |
| B3-10 <u>9</u> a <u>Data Generation Level</u> .....   | <a href="#">B3-21</a>  |
| B3-10 <u>9</u> a(1) <u>Independent Technical Review</u> .....   | <a href="#">B3-23</a>  |
| B3-10 a(2) <u>Technical Supervisor Review</u> .....   | <a href="#">B3-24</a>  |
| B3-10 a(3) <u>QA Officer Review</u> .....   | <a href="#">B3-25</a>  |
| B3-10 <u>9</u> b <u>Project Level</u> .....   | <a href="#">B3-25</a>  |
| B3-10 b(1) <u>Site Project QA Officer</u> .....   | <a href="#">B3-25</a>  |
| B3-10 <u>9</u> b(2 <u>1</u> ) <u>Site Project Manager</u> .....   | <a href="#">B3-26</a>  |
| B3-10 <u>9</u> b(3 <u>2</u> ) <u>Prepare Site Project QA Officer <b>Manager</b> Summary and Data<br/>Validation Summary</u> ..... | <a href="#">B3-27</a>  |
| B3-10 <u>9</u> b(4 <u>3</u> ) <u>Prepare Waste Stream Characterization <b>Waste Analysis</b><br/>Package</u> .....                | <a href="#">B3-28</a>  |
| B3-10 <u>9</u> c <u>Permittee Level</u> .....   | <a href="#">B3-28</a>  |
| B3-11 <u>10</u> <u>Reconciliation with Data Quality Objectives</u> .....  | <a href="#">B3-29</a>  |
| B3-11 <u>10</u> a <u>Reconciliation at the Project Level</u> .....  | <a href="#">B3-29</a>  |

|       |           |  |       |                       |
|-------|-----------|--|-------|-----------------------|
| B3-11 | <u>10</u> | b Reconciliation at the Permittee Level                              | ..... | <a href="#">B3-31</a> |
| B3-12 | <u>11</u> | Data Reporting Requirements  | ..... | <a href="#">B3-31</a> |
|       | <u>11</u> | a Data Generation Level  | ..... | <a href="#">B3-32</a> |
|       | <u>11</u> | b Project Level  | ..... | <a href="#">B3-32</a> |
|       | <u>11</u> | b(1) Waste Stream Profile Form                                       | ..... | <a href="#">B3-32</a> |
|       | <u>11</u> | b(2) Characterization <del>Waste Analysis</del> Information Summary  | ....  | <a href="#">B3-33</a> |
|       | <u>11</u> | b(3) Waste Stream Characterization <del>Waste Analysis</del> Package | ..    | <a href="#">B3-34</a> |
|       | <u>11</u> | b(4) WIPP Waste Information System ( <b>WWIS</b> ) Data Reporting    | ..    | <a href="#">B3-35</a> |
| B3-13 | <u>12</u> | Nonconformances  | ..... | <a href="#">B3-35</a> |
| B3-14 | <u>13</u> | Special Training Requirements and Certifications                     | ..... | <a href="#">B3-38</a> |
| B3-15 | <u>14</u> | Changes to WAP-Related Plans or Procedures                           | ..... | <a href="#">B3-38</a> |
| B3-16 | <u>15</u> | List of References   | ..... | <a href="#">B3-39</a> |

## 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                           |
| B3-5                | Summary of Laboratory Quality Control Samples and Frequencies for Volatile Organic Compound Analysis      |
| B3-6                | Semivolatile Organic Compound Target Analyte List and Quality Assurance Objectives                        |
| B3-7                | Summary of Laboratory Quality Control Samples and Frequencies for Semi-Volatile Organic Compound 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  |
| <del>B3-11</del>    | <del>Testing Batch Data Report Contents</del>   |
| B3-12 <sup>11</sup> | Sampling Batch Data Report Contents   |
| B3-13 <sup>12</sup> | Analytical Batch Data Report Contents   |
| B3-14 <sup>13</sup> | Data Reporting Flags  |

## List of Figures

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

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

1 B3-1 Validation Methods

2  
3 The Permittees shall require the generator/storage sites (**sites**) to perform validation of all  
4 sampling and analytical data (qualitative as well as quantitative) generated in accordance with  
5 the requirements of the Waste Analysis Plan (WAP) so that data used for Waste Isolation Pilot  
6 Plant (**WIPP**) compliance programs will be of known and acceptable quality. Validation includes  
7 a quantitative determination of precision, accuracy, completeness, and method detection limits  
8 (as appropriate) for analytical data (headspace Volatile Organics Compounds (**VOC**), total  
9 VOCs, Semivolatile Organic Compounds (**SVOC**), and metals data). Quantitative data  
10 validations shall be performed according to the conventional methods outlined below (equations  
11 B3-1 through B3-8). These quantitative determinations will be compared to the Quality  
12 Assurance Objectives (**QAOs**) specified in Sections B3-2 through B3-9~~8~~. A qualitative  
13 determination of comparability and representativeness will also be performed.  
14

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

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

26 Precision

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

32 **[No changes proposed to Equation B3-1]**

(B3-1)

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

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

5 [No changes proposed to Equation B3-2] (B3-2)

6 where  $s$  is the standard deviation and  $y_{\text{mean}}$  is the mean of the replicate sample analyses.

7 The standard deviation,  $s$ , is calculated as follows:

8 [No changes proposed to Equation B3-3] (B3-3)

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

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

14 [No changes proposed to Equation B3-4] (B3-4)

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

16 Accuracy

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

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

2 **[No changes proposed to Equation B3-5]** (B3-5)

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

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

4 **[No changes proposed to Equation B3-6]** (B3-6)

5 where S is the measured concentration in the spiked aliquot, U is the measured concentration in  
6 the unspiked aliquot, and  $C_{sc}$  is the actual concentration of the spike added.

#### 7 Method Detection Limit

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

12 **[No changes proposed to Equation B3-7]** (B3-7)

13 where  $T_{(n-1, 1-\alpha=.99)}$  is the t-distribution value appropriate to a 99 percent confidence level and a  
14 standard deviation estimate with n-1 degrees of freedom, n is the number of observations, and s  
15 is the standard deviation of replicate measurements.

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

$$17 \text{MDL} = 3s \quad (\text{B3-8})$$

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

#### 22 Completeness

23 Completeness is a measure of the amount of valid data obtained from the overall measurement  
24 system compared to the amount of data collected and submitted for analysis. Completeness  
25 must be expressed as the number of samples analyzed with valid results as a percent of the  
26 total number of samples submitted for analysis. Completeness, expressed as the percent  
27 complete (**%C**), is calculated as follows:  
28

29 **[No changes proposed to Equation B3-9]** (B3-9)

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

### 3 Comparability

4 Comparability is the degree to which one data set can be compared to another. Comparability of  
5 data generated at different sites will be assured through the use of standardized, ~~approved~~  
6 ~~testing~~, sampling, preservation, and analytical techniques and by meeting the QAOs specified in  
7 Sections B3-2 through B3-98.

8 The comparability of waste characterization analysis data shall be ensured through the use of  
9 generator/storage site data usability criteria. The Permittees shall ensure that data usability  
10 criteria are consistently established and used by the generator/storage sites to assess the  
11 usability of analytical ~~and testing~~ data. The criteria shall address, as appropriate, the following:  
12

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

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

### 27 Representativeness

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

32 Representativeness of waste containers from waste streams subjected to ~~visual examination~~  
33 ~~and headspace gas~~, homogeneous solids, and soil/gravel sampling and analysis will be  
34 validated, through documentation, that a true random sample with an adequate population was  
35 identified and collected consistent with Permit Attachment B2, Section B2-1. Since  
36 representativeness is a quality characteristic that expresses the degree to which a sample or  
37 group of samples represents the population being studied, the random selection of waste  
38 containers ensures representativeness on a Program level. The Permittees shall require the site

1 Project Manager to document that the selected waste containers from within a waste stream  
2 were randomly selected. Sampling personnel shall verify that proper procedures are followed to  
3 ensure that samples are representative of the waste contained in a particular waste container or  
4 a waste stream.

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

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

#### 16 Identification of Tentatively Identified Compounds

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

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

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

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

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

1 B3-2 Headspace-Gas Sampling

2 Quality Assurance Objectives

3 ~~With the exception of qualifying LANL sealed sources waste containers, headspace-gas~~  
4 ~~sampling will occur from the headspace within each drum of transuranic (TRU) mixed waste or~~  
5 ~~randomly selected containers from waste streams that meet the conditions for reduced~~  
6 ~~headspace-gas sampling listed in Attachment B, Section B-3a(1). The LANL sealed sources~~  
7 ~~waste containers that meet specified conditions must be assigned VOC concentration values in~~  
8 ~~accordance with Section B-3a(1)(iii).~~

9 The precision and accuracy of the drum container headspace-gas sampling operations must be  
10 assessed by analyzing field QC headspace-gas samples. These samples must include  
11 equipment blanks, field reference standards, field blanks, and field duplicates. If the QAOs  
12 described below are not met, a nonconformance report must be prepared, submitted, and  
13 resolved (Section B3-4312).

14 Precision

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

20 Accuracy

21 A field reference standard must be collected using headspace-gas sampling equipment to  
22 assess the accuracy of the headspace-gas sampling operation at a frequency of one field  
23 reference standard for every 20 containers drums sampled or per sampling batch. Corrective  
24 action must be taken if the %R of the field-reference standard is less than 70 or greater than  
25 130.

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

32 Completeness

33 Sampling completeness shall be expressed as the number of valid samples collected as a  
34 percent of the total number of samples collected for each waste stream. ~~The completeness can~~  
35 ~~also be expressed as the number of valid samples collected as a percent of the total number of~~  
36 ~~drums for each waste stream.~~ A valid sample is defined as a sample collected in accordance  
37 with approved sampling methods and the drum container was properly prepared for sampling  
38 (e.g., the polyliner was vented to the drum container headspace). The Permittees shall require  
39 participating sampling facilities to achieve a minimum 90 percent completeness. The amount

1 and type of data that may be lost during the headspace-gas sampling operation cannot be  
2 predicted in advance. The Permittees shall require the Site Project Quality Assurance (QA)  
3 Officer to evaluate the importance of any lost or contaminated headspace-gas samples and take  
4 corrective action as appropriate.

#### 5 Comparability

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

#### 13 Representativeness

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

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

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

2 B3-3 Sampling of Homogeneous Solids and Soils/Gravel

3 Quality Assurance Objectives

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

10 Precision

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

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

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

36 Accuracy

37 Sampling accuracy through the use of standard reference materials shall not be measured.  
38 Because waste containers containing homogeneous solids and soil/gravel with known quantities  
39 of analytes are not available, sampling accuracy cannot be determined. However, sampling

1 methods and requirements described are designed to minimize sample degradation and hence  
2 maximize sampling accuracy.

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

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

### 11 Completeness

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

### 17 Comparability

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

### 22 Representativeness

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

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

30 (B3-10)

31 **[No changes proposed to Equation B3-10]**

32 where

33 x = the depth of the waste in the container

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

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

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

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

#### B3-4 Radiography

##### Quality Assurance Objectives

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

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

##### Precision

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

##### Accuracy

The programmatic accuracy at which the waste matrix code and waste material parameter weights can be determined must be documented through visual examination of a randomly selected statistical portion of waste containers. The Permittees shall require the Site Project QA Officer to calculate and report the miscertification rate of waste containers that require

1 ~~assignment to a different waste matrix code or are found to contain prohibited items after visual~~  
2 ~~examination as a measure of radiography accuracy. The miscertification rate shall be used to~~  
3 ~~determine the number of drums subject to confirmatory visual examination.~~

#### 4 Completeness

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

#### 10 Comparability

11 ~~The comparability of radiography data from different sites shall be enhanced by using~~  
12 ~~standardized radiography procedures and operator qualifications.~~

#### 13 B3-5.4 Gas Volatile Organic Compound Analysis

#### 14 Quality Assurance Objectives

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

#### 22 Precision

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

#### 28 Accuracy

29 Accuracy as %R shall be assessed for the laboratory operations by analyzing PDP blind-audit  
30 samples and laboratory-control samples. Results from these measurements must be compared  
31 to the criteria listed in Table B3-2. These QC measurements will be used to demonstrate  
32 acceptable method performance and to trigger corrective action when control limits are  
33 exceeded.

#### 34 Calibration

35 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated  
36 using the procedures and criteria specified in Table B3-3. These criteria will be used to

1 demonstrate acceptable calibration and to trigger corrective action when control limits are  
2 exceeded.

3 Method Detection Limit

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

7 Program Required Quantitation Limit

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

12 Completeness

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

21 Comparability

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

26 Representativeness

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

30 B3-65 Total Volatile Organic Compound Analysis

31 Quality Assurance Objectives

32 The development of DQOs specifically for this program has resulted in the QAOs listed in Table  
33 B3-4. The specified QAOs represent the required quality of data necessary to draw valid  
34 conclusions regarding program objectives. WAP-required limits, such as the PRQL associated  
35 with VOC analysis, are specified to ensure that the analytical data collected satisfy the

1 requirements of all data users. Key data-quality indicators for laboratory measurements are  
2 defined below.

3 Precision

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

9 Accuracy

10 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control  
11 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from  
12 these measurements for matrix spikes samples must be compared to the %R criteria listed in  
13 Table B3-4. Results for surrogates and internal standards are evaluated as specified in the SW-  
14 846 method (EPA 1996) or Table B3-5. These QC measurements will be used to demonstrate  
15 acceptable method performance and to trigger corrective action when control limits are  
16 exceeded.

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

21 Calibration

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

26 Method Detection Limit

27 MDLs shall be expressed in milligrams per kilogram (mg/kg) for VOCs and must be less than or  
28 equal to those listed in Table B3-4. The detailed procedures for MDL determination shall be  
29 included in site SOPs.

30 Program Required Quantitation Limit

31 Laboratories must demonstrate the capability to quantitate analytes in samples at or below the  
32 PRQLs given in Table B3-4. Laboratories shall set the concentration of at least one calibration  
33 standard below the PRQL. The detailed procedures for PRQL demonstration shall be included  
34 in laboratory SOPs.

1 Completeness

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

8 Comparability

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

17 Representativeness

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

20 B3-7.6 Total Semivolatile Organic Compound Analysis

21 Quality Assurance Objectives

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

29 Precision

30 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,  
31 replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from  
32 measurements on these samples must be compared to the criteria listed in Table B3-6. These  
33 QC measurements will be used to demonstrate acceptable method performance and to trigger  
34 corrective action when control limits are exceeded.

35 Accuracy

36 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control  
37 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from

1 these measurements for matrix spikes samples must be compared to the %R criteria listed in  
2 Table B3-6. Results for surrogates and internal standards are evaluated as specified in the SW-  
3 846 method (EPA 1996) or Table B3-7. These QC measurements will be used to demonstrate  
4 acceptable method performance and to trigger corrective action when control limits are  
5 exceeded.

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

#### 10 Calibration

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

#### 15 Method Detection Limit

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

#### 18 Program Required Quantitation Limit

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

#### 23 Completeness

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

#### 30 Comparability

31 For SVOC analysis, data generated through analysis of samples from different sites shall be  
32 comparable. The Permittees shall require sites to achieve comparability by using standardized  
33 SW-846 sample preparation and methods that meet the QAO requirements in Tables B3-6 and  
34 B3-7, traceable standards, and by requiring all sites to successfully participate in the PDP.  
35 Generator/storage sites may use the most current version of SW-846 if the methods are  
36 consistent with QAO requirements. Any changes to SW-846 methodology that results in the  
37 elimination of sample preparation or analytical methods in use at generator/storage sites must

1 be addressed as a corrective action to address the comparability of data before and after the  
2 SW-846 modification.

3 Representativeness

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

6 B3-87 Total Metal Analysis

7 Quality Assurance Objectives

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

15 Precision

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

21 Accuracy

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

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

31 Calibration

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

1 Program Required Detection Limits

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

11 Program Required Quantitation Limit

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

19 Completeness

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

26 Comparability

27 For metals analysis, data generated through analysis of samples from different sites shall be  
28 comparable. Comparability will be achieved by using standardized SW-846 sample preparation  
29 and methods that meet QAO requirements in Tables B3-8 and B3-9, demonstrating successful  
30 participation in the PDP, and use of traceable standards. Generator/storage sites may use the  
31 most recent SW-846 update. Any changes to SW-846 methodology that results in the  
32 elimination of sample preparation or analytical methods in use at generator/storage sites must  
33 be addressed as a corrective action to address the comparability of data before and after the  
34 SW-846 modification.

35 Representativeness

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

1 B3-98 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. QAOs  
4 for analytical results are described in terms of precision, accuracy, completeness, comparability,  
5 and representativeness. Appropriate analytical and testing results will may be used to confirm  
6 supplement the characterization analysis of wastes based on acceptable knowledge (Section  
7 B4-42c of Attachment B4). To ensure that the acceptable knowledge process is consistently  
8 applied, the Permittees shall require sites to comply with the following data quality requirements  
9 for acceptable knowledge documentation:

- 10 ● Precision - Precision is the agreement among a set of replicate measurements  
11 without assumption of the knowledge of a true value. The qualitative  
12 determinations, such as compiling and assessing acceptable knowledge  
13 documentation, do not lend themselves to statistical evaluations of precision.  
14 However, the acceptable knowledge information will be addressed by the  
15 independent review of acceptable knowledge information during internal and  
16 external audits.
- 17 ● Accuracy - Accuracy is the degree of agreement between an observed sample  
18 result and the true value. The percentage of waste containers which require  
19 reassignment to a new waste matrix code and/or designation of different  
20 hazardous waste codes numbers based on an the reevaluation of acceptable  
21 knowledge and sampling and analysis data and waste analysis discrepancies  
22 identified by the Permittees during waste examination will be reported as a  
23 measure of acceptable knowledge accuracy.
- 24 ● Completeness - Completeness is an assessment of the number of waste streams  
25 or number of samples collected to the number of samples determined to be  
26 useable through the data validation process. The acceptable knowledge record  
27 must contain 100 percent of the required information (Permit Attachment B4-3).  
28 The useability of the acceptable knowledge information will be assessed for  
29 completeness during audits.
- 30 ● Comparability - Data are considered comparable when one set of data can be  
31 compared to another set of data. Comparability is ensured through sites meeting  
32 the training requirements and complying with the minimum standards outlined for  
33 procedures that are used to implement the acceptable knowledge process. All  
34 sites must assign hazardous waste codes numbers in accordance with Permit  
35 Attachment B4-4 and provide this information regarding its waste to other sites  
36 who store or generate a similar waste stream.
- 37 ● Representativeness - Representativeness expresses the degree to which sample  
38 data accurately and precisely represent characteristics of a population.  
39 Representativeness is a qualitative parameter that will be satisfied by ensuring  
40 that the process of obtaining, evaluating, and documenting acceptable knowledge  
41 information is performed in accordance with the minimum standards established  
42 in Permit Attachment B4. Sites also must assess and document the limitations of  
43 the acceptable knowledge information used to assign hazardous waste codes

1 numbers (e.g., purpose and scope of information, date of publication, type and  
2 extent to which waste parameters are addressed).

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

7 The Permittees shall require each site to address quality control by tracking its performance with  
8 regard to the use of acceptable knowledge by: 1) assessing the frequency of inconsistencies  
9 among information; and 2) documenting the results of acceptable knowledge confirmation  
10 through radiography, visual examination, headspace gas analyses, and solidified waste  
11 analyses. In addition, the acceptable knowledge process and waste stream documentation must  
12 be evaluated through internal assessments by generator/storage site quality assurance  
13 organizations and assessments by auditors external to the organization (i.e., the Permittees).

#### 14 B3-409 Data Review, Validation, and Verification Requirements

15  
16 Procedures shall be developed for the review, validation, and verification of data at the data  
17 generation level; the validation and verification of data at the project level; and the verification of  
18 data at the Permittee level. Data review determines if raw data have been properly collected and  
19 ensures raw data are properly reduced. Data validation **confirms verifies** that the data reported  
20 satisfy the requirements of this WAP and is accompanied by signature release. Data verification  
21 authenticates that data as presented represent the sampling and analysis activities as performed  
22 and have been subject to the appropriate levels of data review. The requirements presented in  
23 this section ensure that WAP records furnish documentary evidence of quality.

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

- 26 ● ~~A Testing Batch Data Report or equivalent includes all data pertaining to radiography or~~  
27 ~~visual examination for up to 20 waste containers without regard to waste matrix. Table~~  
28 ~~B3-11 lists all of the information required in Testing Batch Data Reports (identified with an~~  
29 ~~"X") and other information that is necessary for data validation, but is optional in Testing~~  
30 ~~Batch Data Reports (identified with an "O").~~
- 31 ● A Sampling Batch Data Report or equivalent includes all sample collection data  
32 pertaining to a group of no more than 20 headspace gas or homogeneous waste samples  
33 that were collected for chemical analysis. Table B3-42<sup>11</sup> lists all of the information  
34 required in Sampling Batch Data Reports (identified with an "X") and other information  
35 that is necessary for data validation, but is optional in Sampling Batch Data Reports  
36 (identified with an "O").
- 37 ● An Analytical Batch Data Report or equivalent includes analytical data from the analysis  
38 of TRU-mixed waste for up to 20 headspace gas or homogeneous waste samples.  
39 Analytical Batch Data Reports or equivalent that contain results for composited  
40 headspace gas samples must contain sufficient information to identify the containers that  
41 were composited for each composite sample and the sample volume that was taken from  
42 each waste container. Because Analytical Batch Data Reports are generated based on

1 the number of samples analyzed, an Analytical Batch Data Report may contain results  
2 that are applicable to more than 20 containers depending on how many composite  
3 samples are part of the report, but may not exceed a total of 20 samples analyzed. Table  
4 B3-~~13~~12 lists all of the information required in Analytical Batch Data Reports (identified  
5 with an "X") and other information that is necessary for data validation, but is optional in  
6 Analytical Batch Data Reports (identified with an "O").

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

- 15 ● An On-line Batch Data Report or equivalent contains the combined information from the  
16 Sampling Batch Data Report and Analytical Batch Data Report that is relevant to the on-  
17 line method used.

#### 18 B3-~~10~~9a Data Generation Level

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

- 21 ● All raw data shall be signed and dated in reproducible ink by the person  
22 generating it. Alternately, unalterable electronic signatures may be used.
- 23 ● All data must be recorded clearly, legibly, and accurately in field and laboratory  
24 records (bench sheets, logbooks), and include applicable sample identification  
25 numbers (for sampling and analytical labs).
- 26 ● All changes to original data must be lined out, initialed, and dated by the individual  
27 making the change. A justification for changing the original data may also be  
28 included. Original data must not be obliterated or otherwise disfigured so as not to  
29 be readable. Data changes shall only be made by the individual who originally  
30 collected the data or an individual authorized to change the data.
- 31 ● All data must be transferred and reduced from field and laboratory records  
32 completely and accurately.
- 33 ● All field and laboratory records must be maintained as specified in Table B-76 of  
34 Attachment B.
- 35 ● Data must be organized into a standard format for reporting purposes (Batch Data  
36 Report), as outlined in specific sampling and analytical procedures.
- 37 ● All electronic and video data must be stored appropriately to ensure that waste  
38 container, sample, and associated QC data are readily retrievable. In the case of

1 classified information, additional security provisions may apply that could restrict  
2 retrievability. The additional security provisions will be documented in  
3 generator/storage site procedures as outlined in the QAPjP in accordance with  
4 prevailing classified information security standards.

5 Data review, validation, and verification at this level involves scrutiny and signature release from  
6 qualified independent technical reviewer(s)<sup>1</sup>, ~~technical supervisors(s), and a QA representative,~~  
7 as specified below. Individuals conducting this data review, validation, and verification must use  
8 checklists that address all of the items included in this section. Checklists must contain or  
9 reference tables showing the results of sampling, analytical or on-line batch QC samples, if  
10 applicable. Checklists must reflect review of all QC samples and quality assurance objective  
11 categories in accordance with criteria established in Tables B3-2 through B3-9 (as applicable to  
12 the methods validated). Completed checklists must be forwarded with Batch Data Reports to the  
13 project level. Analytical raw data must be available and reviewed by the data generation level  
14 reviewer.

15 B3-409a(1) Independent Technical Review

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

20 One hundred percent of the Batch Data Reports must receive an independent technical review.  
21 This review shall be performed by an individual other than the data generator who is qualified to  
22 have performed the initial work. The independent technical review must be performed as soon as  
23 practicably possible in order to determine and correct negative quality trends in the sampling or  
24 analytical process. However at a minimum, the independent technical review must be performed  
25 before any waste associated with the data reviewed is managed, stored, or disposed at WIPP,  
26 unless the data are being obtained from waste sampling and analysis as containers are being  
27 retrieved or generated after initial WSPF approval as described in Attachment B2, Section B2-1.  
28 The reviewer(s) must release the data as evidenced by signature, and as a consequence ensure  
29 the following:

- 30 ● Data generation and reduction were conducted in a technically correct manner in  
31 accordance with the methods used (procedure with revision). Data were reported  
32 in the proper units and correct number of significant figures.
- 33 ● Calculations have been verified by a valid calculation program, a spot check of  
34 verified calculation programs, and/or 100 percent check of all hand calculations.  
35 Values that are not verifiable to within rounding or significant difference  
36 discrepancies must be rectified prior to completion of independent technical  
37 review.
- 38 ● The data have been reviewed for transcription errors.

---

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

- 1           ●       The testing, sampling, or analytical data QA documentation for Batch Data  
2           Reports is complete and includes, as applicable, raw data, DAC and equilibrium  
3           calculations and times, calculation records, chain-of-custody (**COC**) forms,  
4           calibration records (or references to an available calibration package), QC sample  
5           results, and copies or originals of gas canister sample tags. Corrective action will  
6           be taken to ensure that all Batch Data Reports are complete and include all  
7           necessary raw data prior to completion of the independent technical review.
  
- 8           ●       QC sample results are within established control limits, and if not, the data have  
9           been appropriately qualified in accordance with data useability criteria. Data  
10          outside of established control limits will be qualified as appropriate, assigned an  
11          appropriate qualifier flag, discussed in the case narrative, and included as  
12          appropriate in calculations for completeness. QC criteria that were not met are  
13          documented.
  
- 14          ●       Reporting flags (Table B3-4413) were assigned correctly.
  
- 15          ●       Sample holding time and preservation requirements were met, or exceptions  
16          documented.
  
- 17          ●       ~~Radiography tapes have been reviewed (independent observation) on a waste  
18          container basis at a minimum of once per testing batch or once per day of  
19          operation, whichever is less frequent (Attachment B1, Section B1-3b(2)). The  
20          radiography tape will be reviewed against the data reported on the radiography  
21          form to ensure that the data are correct and complete.~~
  
- 22          ●       Field sampling records are complete. Incomplete or incorrect field sampling  
23          records will be subject to resubmittal prior to completion of the independent  
24          technical review.
  
- 25          ●       QAOs have been met according to the methods outlined in Permit Attachment B3,  
26          Section B3-2 through B3-8.
  
- 27          ●

28       B3-10a(2) Technical Supervisor Review

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

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

- 40       ●       ~~The data are technically reasonable based on the technique used.~~

1 ~~● All data have received independent technical review with the exception of~~  
2 ~~radiography tapes, which shall receive periodic technical review as specified in~~  
3 ~~Attachment B1, Section B1-3b(2).~~

4 ~~● The testing, sampling, or analytical data QA documentation for Batch Data~~  
5 ~~Reports is complete and includes, as applicable, raw data, DAC and equilibrium~~  
6 ~~calculations and times, calculation records, COC forms, calibration records, QC~~  
7 ~~sample results, and original or copies of gas sample canister tags.~~

8 ~~● Sample holding time requirements were met, or exceptions documented.~~

9 ~~● Field sampling records are complete.~~

10 B3-10a(3) QA Officer Review

11 ~~The data generation level QA review ensures that the Batch Data Report is complete, that QC~~  
12 ~~checks meet the acceptance criteria, and that the appropriate QAOs have been met. This review~~  
13 ~~verifies and validates that the characterization results meet the program QA/QC, that instrument~~  
14 ~~performance criteria have been met, and that QAOs for the subject characterization area have~~  
15 ~~been met.~~

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

23 ~~● Independent technical and technical supervisory reviews have been performed as~~  
24 ~~evidenced by the appropriate signature releases.~~

25 ~~● The QA documentation for Batch Data Reports is complete as appropriate for the~~  
26 ~~point of data generation.~~

27 ~~● Sampling and analytical QC checks have been properly performed. QC criteria~~  
28 ~~that were not met are documented.~~

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

30 B3-10**9**b Project Level

31 ~~Data validation and verification at this level involves scrutiny and signature release from the Site~~  
32 ~~Project Manager (or designee) and the Site Project QA Officer (or designee). The Permittees~~  
33 ~~shall require each site to meet the following minimum requirements for each waste container.~~  
34 ~~Any nonconformance identified during this process shall be documented on a nonconformance~~  
35 ~~report (Section B3-1312).~~

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

6 ~~B3-10b(1) Site Project QA Officer~~

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

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

- 16 ~~● Batch Data Reports are complete and data are properly reported (i.e., data are~~  
17 ~~reported in correct units, with correct significant figures, and with correct qualifying~~  
18 ~~flags).~~
- 19 ~~● Sampling batch QC checks (e.g., equipment blanks, field duplicates, field~~  
20 ~~reference standards) were properly performed, and meet the established QAOs~~  
21 ~~and are within established data useability criteria.~~
- 22 ~~● Testing batch QC checks (e.g., replicate scans, measurement system checks)~~  
23 ~~were properly performed. Radiography data are complete and acceptable based~~  
24 ~~on evidence of videotape review of one waste container per day or once per~~  
25 ~~testing batch, whichever is less frequent, as specified in B1-3b(2).~~
- 26 ~~● Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix~~  
27 ~~spikes, matrix spike duplicates, laboratory control samples) were properly~~  
28 ~~performed and meet the established QAOs and are within established data~~  
29 ~~useability criteria.~~
- 30 ~~● On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates, on-~~  
31 ~~line control samples) were properly performed and meet the established QAOs~~  
32 ~~and are within established data useability criteria.~~
- 33 ~~● Proper procedures were followed to ensure representative samples of headspace~~  
34 ~~gas and homogeneous solids and soil/gravel were taken.~~
- 35 ~~● For LANL sealed sources waste streams, the quality control provisions for VOC~~  
36 ~~source term development were properly implemented in accordance with Permit~~  
37 ~~Attachment B, Section B-3a(1)(iii).~~

1 B3-109b(1) Site Project Manager

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

5 One hundred percent of the Batch Data Reports must have Site Project Manager signature  
6 release. ~~The Site Project Manager signature release must occur as soon as practicably possible~~  
7 ~~after the Site Project QA officer signature release in order to determine and correct negative~~  
8 ~~quality trends in the sampling or analytical process. However~~ At a minimum, the Site Project  
9 Manager signature release must be performed before any waste associated with the data  
10 reviewed is managed, stored, or disposed at WIPP, unless the data are being obtained from  
11 waste sampling and analysis as containers are being retrieved or generated as described in  
12 Permit Attachment B2, Section B2-1.

13 This signature release must ensure the following:

- 14 ● ~~The Site Project Manager or designee shall determine~~ The validity of the drum  
15 ~~age criteria (DAC) assignment made at the data generation level based upon an~~  
16 ~~assessment of the data collection and evaluation necessary to make the~~  
17 ~~assignment.~~
- 18 ● ~~For LANL sealed sources waste streams, the VOC source term was properly~~  
19 ~~developed and used in accordance with Permit Attachment B, Section B-3a(1)(iii).~~
- 20 ● Sampling batch QC checks (e.g., equipment blanks, field duplicates, field  
21 reference standards) were properly performed, and meet the established QAOs  
22 and are within established data useability criteria.
- 23 ● Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix  
24 spikes, matrix spike duplicates, laboratory control samples) were properly  
25 performed and meet the established QAOs and are within established data  
26 useability criteria.
- 27 ● On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates, on-  
28 line control samples) were properly performed and meet the established QAOs  
29 and are within established data useability criteria.
- 30 ● Proper procedures were followed to assure representative samples of headspace  
31 gas and homogeneous solids and soil/gravel were taken.
- 32 ● Data generation level independent technical, ~~technical supervisory, and QA officer~~  
33 ~~(or designee)~~ review, validation, and verification have been performed as  
34 evidenced by the completed review checklists and appropriate signature releases.
- 35 ● Batch data review checklists are complete.
- 36
- 37

- 1           ●     Batch Data Reports are complete and data are properly reported (e.g., data are  
2                   reported in the correct units, with the correct number of significant figures, and  
3                   with qualifying flags).
- 4           ●     Verify that data are within established data assessment criteria and meet all  
5                   applicable QAOs (Section B3-112 through B3-8).

6     B3-109b(3) Prepare Site Project QA Officer Manager Summary and Data Validation Summary

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

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

30    B3-109b(4) Prepare Waste Stream Characterization Waste Analysis Package

31    In the event the Permittees request detailed information on a waste stream, the Site Project  
32    Manager will provide a Waste Stream Characterization Waste Analysis Package. The Site  
33    Project Manager can require each characterization area, data generation level technical  
34    supervisor, and QA officer to assist in preparation and review of must assure that the Waste  
35    Stream Characterization Waste Analysis Package (Section B3-121b(2)) as necessary to ensure  
36    the package will support the Site Project Manager's waste characterization analysis  
37    determinations.

38    B3-10-9c Permittee Level

39    The final level of data verification occurs at the Permittee level and must, at a minimum, consist  
40    of an inventory check of the Batch Data Reports to verify completeness. Permittee Level

1 Verification is described in Permit Attachment B7. The Permittees are responsible for the  
2 verification that Batch Data Reports include the following:

- 3 ~~● Project-level signature releases~~
- 4 ~~● Listing of all waste containers being presented in the report~~
- 5 ~~● Listing of all testing, sampling, and analytical batch numbers associated with each~~  
6 ~~waste container being reported in the package~~
- 7 ~~● Analytical Batch Data Report case narratives~~
- 8 ~~● Site Project QA Officer Summary~~
- 9 ~~● Data Validation Summary~~
- 10 ~~● Complete summarized qualitative and quantitative data for all waste containers~~  
11 ~~with data flags and qualifiers.~~

12 For each Waste Stream Profile Form (WSPF) submitted for approval, the Permittees must verify  
13 that each submittal (i.e., WSPF and Characterization Information Summary) is complete and  
14 notify the originating site in writing of the WSPF approval. The Permittees will maintain the data  
15 as appropriate for use in the regulatory compliance programs. At a minimum, the verification  
16 must:

- 17 ~~● Ensure the correct assignment of the waste stream description, Waste Matrix~~  
18 ~~Code Group, Summary Category Groups, and EPA hazardous waste codes~~
- 19 ~~● Reconcile data~~
- 20 ~~● Contain summarized results of characterization~~
- 21 ~~● Contain acceptable knowledge summary documentation~~
- 22 ~~● List the methods used for characterization~~

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

25 B3-11<sup>10</sup> Reconciliation with Data Quality Objectives

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

1 **B3-1110a Reconciliation at the Project Level**

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

12 For each waste stream ~~characterized~~ analyzed, the Permittees shall require each Site Project  
13 Manager to determine if sufficient data have been collected to determine the following WAP-  
14 required waste parameters, as applicable:

- 15 ● Waste matrix code
- 16 ● Waste material parameter weights
- 17 ● If each waste container of waste contains TRU radioactive waste
- 18 ● Mean concentrations, UCL<sub>90</sub> for the mean concentrations, standard deviations,  
19 and the number of samples collected for each VOC in the headspace gas of  
20 waste containers in the waste stream
- 21 ● The potential flammability of TRU waste headspace gases
- 22 ● Mean concentrations, UCL<sub>90</sub> for the mean concentrations, standard deviations,  
23 and number of samples collected for VOCs, SVOCs, and metals in the waste  
24 stream
- 25 ● Whether the waste stream exhibits a toxicity characteristic (**TC**) under 40 CFR  
26 Part 261, Subpart C
- 27 ● Whether the waste stream contains listed waste found in 20.4.1.200 NMAC  
28 incorporating 40 CFR Part 261, Subpart D
- 29 ● Whether the waste stream can be classified as hazardous or nonhazardous at the  
30 90-percent confidence level
- 31 ~~● Whether a sufficient number of waste containers have been visually examined (as~~  
32 ~~a QC check on radiography) to determine with a reasonable level of certainty that~~  
33 ~~the UCL<sub>90</sub> for the miscertification rate is less than 14 percent~~
- 34 ● Whether an appropriate packaging configuration and ~~Drum Age Criteria (DAC)~~  
35 were applied and documented in the headspace gas sampling documentation,  
36 and whether the drum age was met prior to sampling.

- 1           ●     Whether all TICs were appropriately identified and reported in accordance with  
2           the requirements of Section B3-1 prior to submittal of a WSPF for a waste stream  
3           or waste stream lot.
  
- 4           ●     Whether the overall completeness, comparability, and representativeness QAOs  
5           were met for each of the analytical ~~and testing~~ procedures as specified in  
6           Sections B3-2 through B3-9~~8~~ prior to submittal of a WSPF for a waste stream or  
7           waste stream lot.
  
- 8           ●     Whether the PRQLs for all analyses were met prior to submittal of a WSPF for a  
9           waste stream or waste stream lot.

10       If the Site Project Manager determines that insufficient data have been collected to make the  
11       determinations listed above, additional data collection efforts must be undertaken. The  
12       reconciliation of a waste stream shall be performed, as described in Permit Attachment B4, prior  
13       to submittal of WSPF and Waste Stream Information Summary to the Permittees for that waste  
14       stream. ~~For subsequent shipments, data reconciliation is done on all containers or samples prior~~  
15       ~~to shipment to WIPP.~~ The Permittees shall not manage, store, or dispose TRU mixed waste at  
16       WIPP; from any waste stream until ~~unless~~ the Site Project Manager determines that the WAP-  
17       required waste parameters listed above have been met for that waste stream.

18       The statistical procedure presented in Permit Attachment B2 shall be used by participating Site  
19       Project Managers to evaluate and report waste characterization analysis data from the analysis  
20       of homogeneous solids and soil/gravel. The procedure, which calculates UCL<sub>90</sub> values, shall be  
21       used to assess compliance with the DQOs in Attachment B, Section B-4a(1) as well as with  
22       RCRA regulations. The procedure must be applied to all laboratory analytical data for total  
23       VOCs, total SVOCs, and total metals. For RCRA regulatory compliance (40 CFR § 261.24), data  
24       from the analysis of the appropriate metals and organic compounds shall be expressed as  
25       toxicity characteristic leaching procedure (TCLP) values or results may also be compared to the  
26       TC levels expressed as total values. These total values will be considered the regulatory  
27       threshold limit (RTL) values for the WAP. RTL values are obtained by calculating the  
28       weight/weight concentration (in the solid) of a TC analyte that would give the regulatory  
29       weight/volume concentration (in the TCLP extract), assuming 100-percent analyte dissolution.

#### 30       B3-4410b Reconciliation at the Permittee Level

31       The Permittees must also ensure that data of sufficient type, quality, and quantity are collected to  
32       meet WAP DQOs. The Permittees Level data reconciliation is discussed in Permit Attachment  
33       B7. ~~will ensure sufficient data have been collected in accordance with Attachment B, Section B-~~  
34       ~~4a(1) to determine the following:~~

- 35       — ●     The concentration of VOC constituents in the headspace in the total waste  
36       inventory has not exceeded the environment performance standards of 20.4.1.500  
37       NMAC (incorporating 40 CFR §264.601(c)) as specified in Module IV;
  
- 38       — ●     Whether waste streams proposed for disposal in WIPP have been adequately  
39       characterized and

1 Whether data supports the information contained in the WIPP RCRA permit  
2 application

3 B3-4211 Data Reporting Requirements

4 Data reporting requirements define the type of information and the method of transmittal for data  
5 transfer from the data generation level to the project level and from the project level to the  
6 Permittees.

7 B3-4211a Data Generation Level

8 Data shall be transmitted by hard copy or electronically (provided a hard copy is available on  
9 demand) from the data generation level to the project level. Transmitted data shall include all  
10 Batch Data Reports and data review checklists. The Batch Data Reports and checklists used  
11 must contain all of the information required by the ~~testing, sampling, and analytical techniques~~  
12 described in Permit Attachments B1 through B6 , as well as the signature releases to document  
13 the review, validation, and verification as described in Section B3-409. All Batch Data Reports  
14 and checklists shall be in approved formats, as provided in site-specific documentation.

15 Batch Data Reports shall be forwarded to the site project office. Site QAPjPs shall specify the  
16 individual at the site project office who will receive these reports. ~~After review by the Site~~  
17 ~~Project Manager QA Officer, all Batch Data Reports will be forwarded to the Site Project~~  
18 ~~Manager.~~ All Batch Data Reports shall be assigned serial numbers, and each page shall be  
19 numbered. The serial number used for Batch Data Reports can be the same as the ~~testing,~~  
20 ~~sampling, or analytical batch number.~~

21 QA documentation, including raw data, shall be maintained in either ~~testing, sampling, and~~  
22 ~~analytical facility files, or site project files for those facilities located on site in accordance with the~~  
23 ~~document storage requirements of site approved site QAPjPs.~~ Permittee approved laboratories  
24 ~~Contract waste characterization facilities shall forward testing, sampling, and analytical QA~~  
25 ~~documentation along with Batch Data Reports to the site project office for inclusion in site project~~  
26 ~~files.~~

27 B3-4211b Project Level

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

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

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

6 B3-4211b(1) Waste Stream Profile Form

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

- 8 ● Generator/storage site name
- 9 ● Generator/storage site EPA ID
- 10 ● Date of audit report approval by NMED (if obtained)
- 11 ● Original generator of waste stream
- 12 ● Whether waste is Contact-Handled or Remote-Handled
- 13 ● The Waste Stream WIPP Identification Number
- 14 ● Summary Category Group
- 15 ● Waste Matrix Code Group
- 16 ● Waste Material Parameter Weight Estimates per unit of waste
- 17 ● Waste stream name
- 18 ● A description of the waste stream
- 19 ● Applicable EPA hazardous waste codes numbers
- 20 ● Applicable TRUCON codes
- 21 ● A listing of acceptable knowledge documentation used to identify the waste  
22 stream
- 23 ● The waste characterization analysis procedures used and the reference and date  
24 of the procedure
- 25 ● Certification signature of Site Project Manager, name, title, and date signed

26 B3-4211b(2) Characterization Waste Analysis Information Summary

27 The Characterization Waste Analysis Information Summary shall include the following elements,  
28 if applicable:

- 1 ● Data reconciliation with DQOs
- 2 ● Headspace gas summary data listing the identification numbers of samples used  
3 in the statistical reduction, the maximum, mean, standard deviation, UCL<sub>90</sub>, RTL,  
4 and associated EPA hazardous waste codes numbers that must be applied to the  
5 waste stream.
- 6 ~~● For LANL sealed sources waste streams, the VOC source term determination  
7 data (as defined by Attachment B, Section B-3a(1)(iii)) listing one-half the method  
8 detection limit and mean when used to assign concentrations for the headspace  
9 gas target analytes.~~
- 10 ● Total metal, VOC, and SVOC analytical results for homogeneous solids and  
11 soil/gravel (if applicable), and demonstration that control charting cannot be  
12 applied effectively, if this option is implemented.
- 13 ● TIC listing and evaluation, and verification that acceptable knowledge (AK) was  
14 confirmed.
- 15 ~~● Radiography and visual examination summary to document that all prohibited  
16 items are absent in the waste and to confirm AK, and documentation and  
17 justification for the use of radiography in lieu of or in combination with visual  
18 examination/visual examination technique for newly generated waste.~~
- 19 ● A complete listing of all container identification numbers used to generate the  
20 WSPF, cross-referenced to each Batch Data Report
- 21 ● Complete AK summary, including stream name and number, point of generation,  
22 waste stream volume (current and projected), generation dates, TRUCON codes,  
23 Summary Category Group, Waste Matrix Code(s) and Waste Matrix Code Group,  
24 other TWBIR information, waste stream description, areas of operation,  
25 generating processes, RCRA determinations, radionuclide information, all  
26 references used to generate the AK summary, and any other information required  
27 by Permit Attachment B4, Section B4-2b.
- 28 ● Method for determining Waste Material Parameter Weights per unit of waste
- 29 ● Certification through acceptable knowledge or testing and/or analysis that any  
30 waste assigned the hazardous waste number of U134 (hydrofluoric acid) no  
31 longer exhibits the characteristic of corrosivity. This is confirmed verified by  
32 assuring that no liquid is present in U134 waste.

33 B3-1211b(3) Waste Stream Characterization Waste Analysis Package

34 The Waste Stream Characterization Waste Analysis Package includes the following information:

- 35 ● Waste Stream Profile Form (WSPF, Section B3-1211b(1))

- 1 ● Accompanying Characterization Waste Analysis Information Summary (Section  
2 B3-121b(2))
- 3 ~~● Complete AK summary (Section B3-12b(2))~~
- 4 ● Batch Data Reports supporting the confirmation of AK analysis of the waste  
5 stream and any others requested by the Permittees
- 6 ● Raw analytical data requested by the Permittees

#### 7 B3-121b(4) WIPP Waste Information System (WWIS) Data Reporting

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

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

#### 25 B3-1312 Nonconformances

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

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

#### 34 Nonconformances

35 Nonconformances are uncontrolled and unapproved deviations from an approved plan or  
36 procedure. Nonconforming items and activities are those that do not meet the WAP  
37 requirements, procurement document criteria, or approved work procedures. Nonconforming  
38 items shall be identified by marking, tagging, or segregating, and the affected generator/storage

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

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

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

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

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

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

34 Nonconformance to Data Quality Objectives (DQOs)

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

10 The Permittees will receive written notification of all non-administrative nonconformances (i.e., a  
11 failure to meet a DQO) first identified during the Site Project Manager Review within five (5) days  
12 of identification. The Permittees will also receive a nonconformance report within thirty (30) days  
13 of identification. The generator/storage site will implement a corrective action process and  
14 resolve the identified nonconformance prior to the Permittees management, storage, or disposal  
15 of TRU mixed waste at WIPP.

#### 16 ~~Permittees' Corrective Action Process~~

17 The Permittees shall initiate a corrective action process when internal nonconformances and  
18 nonconformances at the generator/storage sites are identified is described in Permit Attachment  
19 B7. ~~Activities and processes that do not meet requirements are documented as deficiencies.~~

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

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

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

#### 4 B3-14 13 Special Training Requirements and Certifications

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

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

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

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

#### 35 B3-15 14 Changes to WAP-Related Plans or Procedures

36 Controlled changes to WAP-related plans or procedures shall be managed through the  
37 document control process described in the QAPD. The Site Project Manager ~~and the Site Project~~  
38 ~~QA Officer~~ shall review all non-administrative changes and evaluate whether those changes  
39 could impact DQOs specified in the Permit. After ~~site certification~~ WSPF approval, any changes  
40 to WAP-related plans or procedures that could positively or negatively impact DQOs (i.e., those  
41 changes that require prior approval of the Permittees as defined in Attachment B5, Section B5-2)  
42 shall be reported to the Permittees within five (5) days of identification by the project level review.

1 The Permittees shall send NMED a monthly summary briefly describing the changes to plans  
2 and procedures identified pursuant to this section during the previous month.

3 ~~B3-46~~<sup>15</sup> List of References

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

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

9 ~~DOE. 1995a. Performance Demonstration Program Plan for the Analysis of Simulated~~  
10 ~~Headspace Gases for the TRU Waste Characterization Program. CAO-95-1076, Current~~  
11 ~~Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.~~

12 ~~DOE. 1995b. Performance Demonstration Program Plan for the Analysis of Solid Wastes for the~~  
13 ~~TRU Waste Characterization Program. CAO-95-1077, Current Revision, Carlsbad, New Mexico,~~  
14 ~~Carlsbad Area Office, U.S. Department of Energy.~~

15 ~~EG&G. 1993a. Preliminary Assessment of Real-Time Radiography and Visual Characterization~~  
16 ~~for Selected Waste Containers. RFP-4604, Golden, Colorado, D. L. Zeigler and R. V. Harder,~~  
17 ~~EG&G Rocky Flats, Rocky Flats Plant.~~

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

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

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

**TABLES**

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**TABLE B3-1  
 WASTE MATERIAL PARAMETERS AND DESCRIPTIONS**

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

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

| Compound                              | CAS Number          | Precision <sup>a</sup><br>(%RSD or RPD) | Accuracy <sup>a</sup><br>(%R) | MDL <sup>b, c, d</sup><br>(ng) | FTIRS<br>MDL <sup>b</sup><br>(ppmv) | PRQL<br>(ppmv) | Completeness<br>(%) |
|---------------------------------------|---------------------|---|-------------------------------|--------------------------------|-------------------------------------|----------------|---------------------|
| Benzene                               | 71-43-2             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Bromoform                             | 75-25-2             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Carbon tetrachloride                  | 56-23-5             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Chlorobenzene                         | 108-90-7            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Chloroform                            | 67-66-3             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| 1,1-Dichloroethane                    | 75-34-3             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| 1,2-Dichloroethane                    | 107-06-2            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| 1,1-Dichloroethylene                  | 75-35-4             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| cis-1,2-Dichloroethylene              | 156-59-2            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| trans-1,2-Dichloroethylene            | 156-60-5            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Ethyl benzene <sup>e, f</sup>         | 100-41-4            | ≤25                                     | 70-130                        | 10                             | 10                                  | 10             | 90                  |
| Ethyl ether                           | 60-29-7             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Formaldehyde <sup>g</sup>             | <del>50-00-0</del>  | <del>≤25</del>                          | <del>70-130</del>             | <del>40</del>                  | <del>40</del>                       | <del>40</del>  | <del>90</del>       |
| Hydrazine <sup>g</sup>                | <del>302-01-2</del> | <del>≤25</del>                          | <del>70-130</del>             | <del>40</del>                  | <del>40</del>                       | <del>40</del>  | <del>90</del>       |
| Methylene chloride                    | 75-09-2             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| 1,1,2,2-Tetrachloroethane             | 79-34-5             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Tetrachloroethylene                   | 127-18-4            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Toluene                               | 108-88-3            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| 1,1,1-Trichloroethane                 | 71-55-6             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Trichloroethylene                     | 79-01-6             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| m-Xylene <sup>e, f</sup>              | 108-38-3            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| o-Xylene                              | 95-47-6             | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| p-Xylene <sup>e, f</sup>              | 106-42-3            | ≤25                                     | 70-130                        | 10                             | 5                                   | 10             | 90                  |
| Acetone                               | 67-64-1             | ≤25                                     | 70-130                        | 150                            | 50                                  | 100            | 90                  |
| Butanol                               | 71-36-3             | ≤25                                     | 70-130                        | 150                            | 50                                  | 100            | 90                  |
| Methanol                              | 67-56-1             | ≤25                                     | 70-130                        | 150                            | 50                                  | 100            | 90                  |
| Methyl ethyl ketone                   | 78-93-3             | ≤25                                     | 70-130                        | 150                            | 50                                  | 100            | 90                  |
| Methyl isobutyl ketone                | 108-10-1            | ≤25                                     | 70-130                        | 150                            | 50                                  | 100            | 90                  |

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

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

<sup>c</sup> ~~Required only for homogeneous solids and soil/gravel waste from Savannah River Site.~~

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

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

<sup>f</sup> The ethyl benzene PRQL for FTIRS is 20 ppm

- CAS = Chemical Abstract Service
- %RSD = Percent relative standard deviation
- RPD = Relative percent difference
- %R = Percent recovery
- MDL = Method detection limit (maximum permissible value), for GC/MS and GC/FID; total number of nanograms delivered to the analytical system per sample (nanograms); for FTIRS based on 1 m sample cell
- PRQL = Program required quantitation limit (parts per million/volume basis)

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

| QC Sample   | Minimum Frequency  | Acceptance Criteria   | Corrective Action <sup>a</sup>   |
|---|--|---|--|
| Method performance samples                            | Seven (7) samples initially and four (4) semiannually  | Meet method QAOs  | Repeat until acceptable  |
| Laboratory duplicates or on-line duplicates           | One (1) per analytical batch or on-line batch  | RPD $\leq$ 25 <sup>b</sup>  | Nonconformance if RPD >25  |
| Laboratory blanks or on-line blanks                   | Daily prior to sample analysis for GC/MS and GC/FID. Otherwise, daily prior to sample analysis and one (1) per analytical batch or on-line | Analyte amounts $\leq$ 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 <sup>b</sup>  | Nonconformance if RPD > 25   |
| Blind audit samples                                   | Samples and frequency controlled by the Gas PDP Plan   | Specified in the Gas PDP Plan   | Specified in the Gas PDP Plan  |

<sup>a</sup> Corrective action per ~~Section B3-13~~ [Permit Attachment B7](#) when final reported QC samples do not meet the acceptance criteria.

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

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

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

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

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

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

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

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

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

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

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

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

| QC Sample                          | Minimum Frequency   | Acceptance Criteria  | Corrective Action <sup>a</sup>  |
|------------------------------------|---|--|---|
| Method performance samples         | Seven (7) samples initially and four (4) semiannually                             | Meet Table B3-4 QAOs   | Repeat until acceptable   |
| Laboratory duplicates <sup>b</sup> | One (1) per analytical batch  | Meet Table B3-4 precision QAOs   | Nonconformance if RPDs > values in Table B3-4                                 |
| Laboratory blanks                  | One (1) per analytical batch  | Analyte concentrations $\leq 3 \times$ MDLs  | Nonconformance if analyte concentrations > 3 x MDLs                           |
| Matrix spikes <sup>b</sup>         | One (1) per analytical batch  | Meet Table B3-4 accuracy QAOs  | Nonconformance if %Rs are outside the range specified in Table B3-4           |
| Matrix spike duplicates            | One (1) per analytical batch  | Meet Table B3-4 accuracy and precision QAOs  | Nonconformance if RPDs > values and %Rs outside range specified in Table B3-4 |
| Laboratory control samples         | One (1) per analytical batch  | Meet Table B3-4 accuracy QAO's   | Nonconformance if %R < 80 or > 120  |
| GC/MS Calibration                  | BFB Tune every 12 hours<br><br>5-pt. Initial Calibration initially, and as needed | Abundance criteria met as per method<br><br>Calibrate according to SW-846 Method requirements:<br><br>%RSD for CCC $\leq 30$ ,<br>%RSD for all other compounds $\leq 15\%$<br><br>Average response factor (RRF) used if %RSD $\leq 15$ , use linear regression if %RSD > 15; R or R <sup>2</sup> $\geq 0.990$ if using alternative curve<br><br>System Performance Check Compound (SPCC) minimum RRF as per SW-846 Method; RRF for all other compounds $\geq 0.01$ | Repeat until acceptable   |

Waste Isolation Pilot Plant  
Class 3 Permit Modification Request  
June 2005

| QC Sample                     | Minimum Frequency  | Acceptance Criteria   | Corrective Action <sup>a</sup>  |
|-------------------------------|--|---|---|
| GC/MS Calibration (continued) | Continuing Calibration every 12 hours  | %D ≤ 20 for CCC;<br><br>SPCC minimum RRF as per SW-846 Method; RRF for all other compounds ≥ 0.01<br><br>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   |
| GC/FID Calibration            | 3-pt. Initial Calibration initially and as needed<br><br>Continuing Calibration every 12 hours | Correlation Coefficient ≥ 0.990 or %RSD ≤ 20 for all analytes<br><br>%D or %Drift for all analytes ≤ 15 of expected values,<br><br>RT ± 3 standard deviations from initial RT calibration per applicable SW-846 Method  | Repeat until acceptable.  |
| Surrogate compounds           | Each analytical sample   | Average %R from minimum of 30 samples for a given matrix ±3 standard deviations   | Nonconformance if %R < (average %R - 3 standard deviation) or > (average %R + 3 standard deviation) |
| Blind audit samples           | Samples and frequency controlled by the Solid PDP Plan   | Specified in the Solid PDP Plan   | Specified in the Solid PDP Plan   |

<sup>a</sup> Corrective Action per ~~section B3-13~~ [Permit Attachment B7](#) when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

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

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

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

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

CAS = Chemical Abstract Service  
 %RSD = Percent relative standard deviation  
 RPD = Relative percent difference  
 %R = Percent recovery  
 MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)  
 PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for nitrobenzene assuming a 100-gram (g) sample, 0.5 gal (2 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilograms)

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

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

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

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

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

| QC Sample                          | Minimum Frequency  | Acceptance Criteria   | Corrective Action <sup>a</sup>                                      |
|------------------------------------|--|---|---|
| Method performance samples         | Seven (7) samples initially and four (4) semiannually  | Meet Table B3-6 QAOs  | Repeat until acceptable   |
| Laboratory duplicates <sup>b</sup> | One (1) per analytical batch   | Meet Table B3-6 precision QAOs  | Nonconformance if RPDs > values in Table B3-6                       |
| Laboratory blanks                  | One (1) per analytical batch   | Analyte concentrations $\leq 3 \times$ 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<br><br>5-pt. Initial Calibration initially, and as needed<br><br><br><br><br><br><br><br><br><br>Continuing Calibration every 12 hours | Abundance criteria met as per method<br><br>Calibrate according to SW-846 Method requirements:<br><br>%RSD for CCC $\leq 30$ ,<br>%RSD for all other compounds $\leq 15\%$<br>Average response factor (RRF) used if %RSD $\leq 15$ , use linear regression if >15; R or R <sup>2</sup> $\geq 0.990$ if using alternative curve<br><br>System Performance Check Compound (SPCC) minimum RRF as per SW-846 Method; RRF for all other compounds $\geq 0.01$<br><br>%D $\leq 20$ for CCC,<br><br>SPCC minimum RRF as per SW-846 Method; RRF for all other compounds $\geq 0.01$<br><br>RT for internal standard must be $\pm 30$ seconds from last daily calibration, internal standard area count must be >50% and <200% of last daily calibration | Repeat until acceptable   |

Waste Isolation Pilot Plant  
Class 3 Permit Modification Request  
June 2005

| QC Sample                  | Minimum Frequency  | Acceptance Criteria  | Corrective Action <sup>a</sup>  |
|----------------------------|--|--|---|
| GC/ECD Calibration         | 5-pt. Calibration initially and as needed<br><br>Continuing Calibration every 12 hours | Correlation Coefficient $\geq$ 0.990 or %RSD < 20 for all analytes<br><br>%D or %Drift for all analytes $\leq$ 15 of expected values,<br><br>RT $\pm$ 3 standard deviations of initial RT calibration per applicable SW-846 Method | Repeat until acceptable   |
| Matrix spike duplicates    | One (1) per analytical batch   | Meet Table B3-6 accuracy and precision QAOs  | Nonconformance if RPDs > values and %Rs outside range specified in Table B3-6                         |
| Laboratory control samples | One (1) per analytical batch   | Meet Table B3-6 accuracy QAO's   | Nonconformance if %R < 80 or > 120  |
| Surrogate compounds        | Each analytical sample   | Average %R from minimum of 30 samples from a given matrix $\pm$ 3 standard deviations  | Nonconformance if %R < (average %R - 3 standard deviations) or > (average %R + 3 standard deviations) |
| Blind audit samples        | Samples and frequency controlled by the Solid PDP Plan                                 | Specified in the Solid PDP Plan  | Specified in the Solid PDP Plan   |

<sup>a</sup> Corrective action per ~~section B3-13~~ **Permit Attachment B7** when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

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

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

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

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

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

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

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

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

- CAS = Chemical Abstract Service
- %RSD = Percent relative standard deviation
- RPD = Relative percent difference
- %R = Percent recovery
- PRDL = Program required detection limit (i.e., maximum permissible value for IDL) (micrograms per liter)
- PRQL = Program required quantitation limit (milligrams per kilogram)

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

| QC Sample   | Minimum Frequency                                     | Acceptance Criteria  | Corrective Action <sup>a</sup>   |
|---|---|--|--|
| Method performance samples  | Seven (7) samples initially and four (4) semiannually | Meet Table B3-8 QAOs   | Repeat until acceptable  |
| Laboratory blanks   | One (1) per analytical batch                          | $\leq 3 \times \text{IDL}$ ( $\leq 5 \times \text{IDL}$ for ICP-MS) <sup>b</sup>   | 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 $\leq 5$ ; 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<br>1 blank, 1 standard (ICP, ICP-MS)<br>3 standard, 1 blank (GFAA, FLAA)<br>5 standard, 1 blank (CVAA, HAA) | Daily   | 90-110 %R (80-120% for CVAA, GFAA, HAA, FLAA) for initial calibration verification solution.<br>Regression coefficient $\geq 0.995$ for FLAA, CVA, GFAA, MAA | Correct problem and recalibrate; repeat initial calibration  |
| Continuing Calibration  | Every 10 samples and beginning and end of run         | 90-110% for continuing calibration verification solution.<br>(80-120% for CVAA, GFAA, HAA, FLAA)   | Correct problem and recalibrate; rerun last 10 samples   |
| Internal Standard Area Verification (ICP-MS)  | Every Sample  | Meet SW-846 Method 6020 criteria   | Nonconformance if not reanalyzed at 5 X dilution until criteria are met  |
| Serial Dilution (ICP, ICP-MS)   | One (1) per analytical batch                          | 5 X dilution must be $\leq 10\%$ D of initial value for sample > 50xIDL  | Flag Data if >10% and > 50xIDL   |

| QC Sample  | Minimum Frequency   | Acceptance Criteria   | Corrective Action <sup>a</sup>  |
|--|---|---|---|
| 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<br><br>Note: Acceptance Criteria and Corrective Action apply only if interferences found in samples at levels greater than ICS A Solution | Correct problem and recalibrate, nonconformance if not corrected                |
| Laboratory Control Samples                         | One (1) per analytical batch  | Table B3-8 accuracy QAOs  | Redigest and reanalyze for affected analytes; non conformance if not reanalyzed |
| Blind audit samples                                | Samples and frequency controlled by the Solid PDP Plan                            | Specified in the Solid PDP Plan   | Specified in the Solid PDP Plan   |

<sup>a</sup> Corrective action per ~~section B3-13~~ [Permit Attachment B7](#) when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

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

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

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

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

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

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

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

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

| Required Information  | Radiography | Visual Examination as QC-Check on Radiography | Visual Verification of Acceptable Knowledge | Comment   |
|---|-------------|---|---|---|
| Batch Data Report Date  | ✕           | ✕   | ✕   |   |
| Batch number  | ✕           | ✕   | ✕   |   |
| Waste container number  | ✕           | ✕   | ✕   |   |
| Waste stream name and/or number                               | ⊖           | ⊖   | ⊖   |   |
| Waste Matrix Code   | ✕           | ✕   | ✕   | Summary Category Group included in waste matrix code  |
| Implementing procedure (specific version used)                | ✕           | ✕   | ✕   | 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  | ⊖           | ⊖   | ⊖   | Drums, Standard Waste Box, Ten-Drum Overpack, etc.  |
| Videotape reference   | ✕           | ✕   |   | Reference to Videotape(s) applicable to each container. For visual examination (for characterization) of newly generated waste, videotape not required if two trained operators review the contents of the waste container to ensure correct reporting. |
| Imaging check   | ⊖           |   |   |   |
| Camera check  |             | ⊖   |   |   |
| Audio check   | ⊖           | ⊖   |   |   |
| QC check of scales  |             | ⊖   | ⊖   | Available documented evidence calibrated scale(s) were used. Only applicable if items are weighed during the visual examination.  |
| QC documentation  | ✕           | ✕   | ✕   |   |
| Description of liners and layers of confinement (if possible) | ✕           | ✕   | ✕   |   |
| Indication of vented rigid liners                             | ✕           | ✕   | ✕   | Only required for containers with rigid liners. If radiography is used to verify, then include in Testing Batch Data Report.  |
| Description of container contents                             | ✕           | ✕   | ✕   | Provide enough detail to identify all discernible waste items, etc., and to verify estimated weights for the 12 waste material parameters.  |

Waste Isolation Pilot Plant  
Class 3 Permit Modification Request  
June 2005

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

LEGEND:

✖-- Required in batch data report.

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

**TABLE B3-4211**  
**SAMPLING BATCH DATA REPORT CONTENTS**

| Required Information              | Headspace Gas | Solid Sampling | Comment  |
|-----------------------------------|---------------|----------------|--|
| Batch Data Report Date            | X             | X              |  |
| Batch number                      | X             | X              |  |
| Waste stream name and/or number   | O             | O              |  |
| Waste Matrix Code                 |               | X              | Summary Category Group included in Waste Matrix Code   |
| Procedure (specific version used) | X             | X              | If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.  |
| Container number                  | X             | X              |  |
| Container type                    | O             | O              | Drums, Standard Waste Box, Ten Drum Overpack, etc.   |
| Sample matrix and type            | X             | X              |  |
| Analyses requested and laboratory | X             | X              |  |
| Point of origin for sampling      | X             | X              | Location where sample was taken (e.g., building number, room)  |
| Sample number                     | X             | X              |  |
| Sample size                       | X             | X              |  |
| Sample location                   | X             | X              | Location within container where sample is taken. (For HSG, specify what layer of confinement was sampled. For solids, physical location within container.)   |
| Sample preservation               | X             | X              |  |
| Person collecting sample          | X             | X              |  |
| Person attaching custody seal     | O             | O              | May or may not be the same as the person collecting the sample   |
| Chain of custody record           | X             | X              | Original or copy is allowed  |
| Sampling equipment numbers        | X             | X              | For disposable equipment, a reference to the lot   |
| 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  
Class 3 Permit Modification Request  
June 2005

| 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 headspace gas volume when it can be estimated   |
| Scale Calibration  |               | O              |  |
| Depth of waste   |               | X              | For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a representative sample has been taken.  |
| Calculation of core recovery   |               | X              | For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a representative sample has been taken.  |
| Co-located core description  |               | X              | For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a QC sample has been taken.  |
| Time between coring and subsampling  |               | X              | Only applicable to coring.   |
| OVA calibration and reading  | O             |                | Only applicable to manifold systems. Must be done in accordance with manufacturer's specifications   |
| Field Records  | X             | X              | Must contain the following as applicable to the sampling method used: Collection problems, Sequence of sampling collection, Inspection of the solids sampling area, Inspection of the solids sampling equipment, Coring tool test, random location of sub-sample, canister pressure, and ambient temperature and pressure. |
| Reference to or copy of associated NCRs, if any                                      | X             | X              | Copies of associated NCRs must be available.   |
| Operator Signature and date and time of sampling                                     | X             | X              |  |
| Data review checklists   | X             | X              | All data review checklists will be identified  |

<sup>a</sup> ~~The headspace gas sampling batch data report is not required for the LANL sealed sources waste containers that meet specified conditions and are assigned VOC concentration values in accordance with Section B-3a(1)(iii).~~

LEGEND:

X - Required in batch data report.

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

**TABLE B3-13<sup>12</sup>**  
**ANALYTICAL BATCH DATA REPORT CONTENTS**

| Required Information  | Headspace Gas <sup>a</sup> | Solid Sampling | Comment   |
|---|----------------------------|----------------|---|
| Batch Data Report Date  | X                          | X              |   |
| Batch number  | X                          | X              |   |
| Sample numbers  | X                          | X              |   |
| QC designation for sample                                     | X                          | X              |   |
| Implementing procedure (specific version used)                | X                          | X              | If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used. |
| QC sample results   | X                          | X              |   |
| Sample data forms   | X                          | X              | Form should contain reduced data for target analytes and TICs   |
| Chain of custody  | X                          | X              | Original or copy  |
| Gas canister tags   | X                          |                | Original or copy  |
| Sample preservation   | X                          | X              |   |
| Holding time  |                            | X              |   |
| Cross-reference of field numbers to laboratory sample numbers | X                          | X              |   |
| Date and time analyzed  | X                          | X              |   |
| <b>Confirmation Verification</b> 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 <sup>13</sup> 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   |

<sup>a</sup> ~~The headspace gas analytical batch data report is not required for the LANL sealed sources waste containers that meet specified conditions and are assigned VOC concentration values in accordance with Section B-3a(1)(iii).~~

LEGEND:

X - Required in batch data report.

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

**TABLE B3-14**<sup>13</sup>  
**DATA REPORTING FLAGS**

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

**FIGURES**

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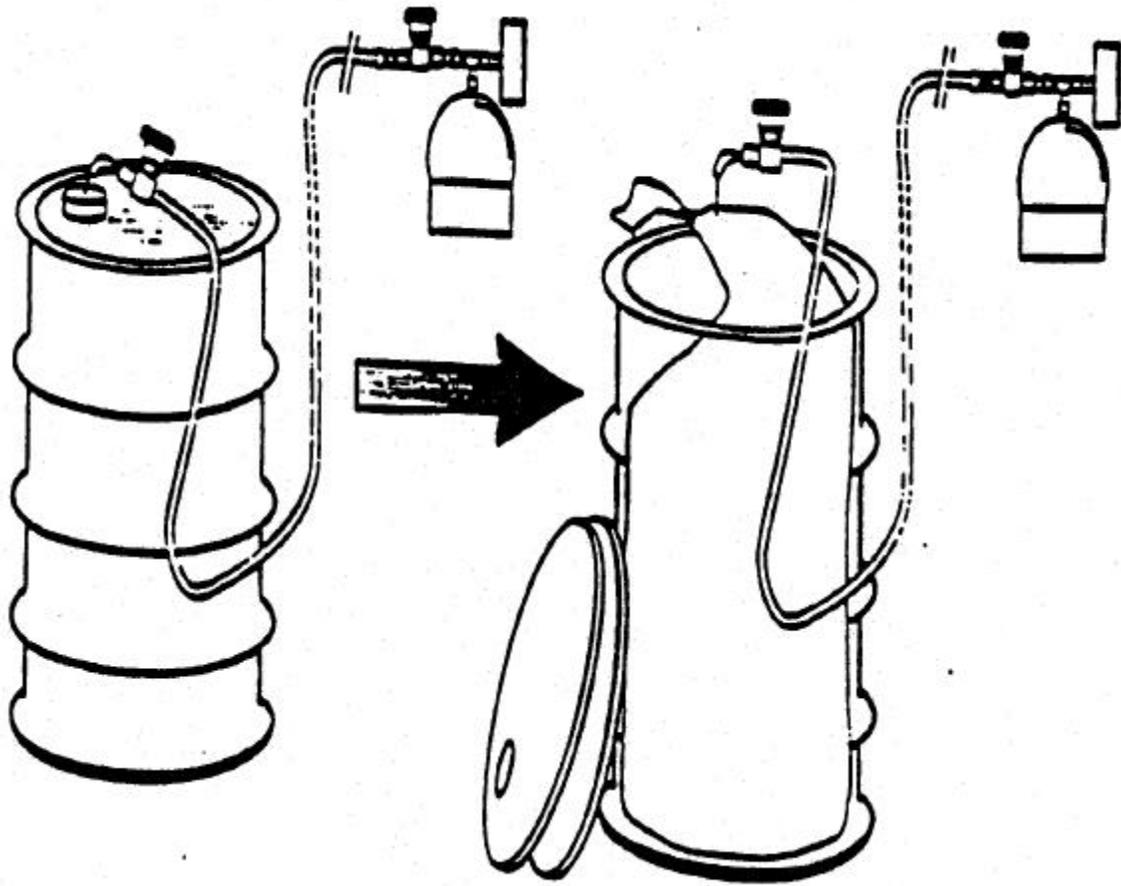


Figure B3-1  
Overall Headspace-Gas Sampling Scheme Illustrating Manifold Sampling