

ATTACHMENT B2

STATISTICAL METHODS USED IN SAMPLING AND ANALYSIS

Waste Isolation Pilot Plant
Hazardous Waste Permit
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Figure B2-1 Approach for Solid and Headspace Gas Sampling and Analysis to Obtain Additional Waste Characterization Information

1 The sampling and analysis strategy is illustrated in Figure B2-1. Preliminary estimates of the
2 mean concentration and variance of each RCRA regulated contaminant in the waste will be
3 used to determine the number of waste containers to select for sampling and analysis.
4 Preliminary estimates will be based on a minimum of five samples selected randomly from the
5 waste stream. If the entire waste stream is not accessible for sampling then a minimum of five
6 preliminary samples will be selected randomly from the accessible population. As the rest of the
7 waste stream is retrieved or generated, additional selected containers will be sampled as
8 provided below and the analytical results will be reported to the Permittees. Samples collected
9 to establish preliminary estimates that are selected, sampled, and analyzed using a Permittee
10 approved laboratory in accordance with applicable provisions of the WAP may be used as part
11 of the required number of samples to be collected. The applicability of the preliminary estimates
12 to the waste stream to be sampled shall be justified and documented. The preliminary estimates
13 will be determined in accordance with the following equations:

14
$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i \quad (\text{B2-1})$$

15
$$s^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 \quad (\text{B2-2})$$

16 Where:

17 \bar{x} = the calculated mean.

18 s^2 = the calculated concentration variance.

19 n = the number of samples analyzed.

20 x_i = the concentration determined in the *i*th sample.

21 i = an index from 1 to n .

22 Based upon the preliminary estimates of \bar{x} and s^2 for each chemical contaminant of concern,
23 estimate the appropriate minimum number of samples (n) to be collected for each contaminant
24 using the following formula from SW-846 (EPA 1996):

25
$$n = \frac{t_{\alpha, n_0-1}^2 s^2}{(RT - \bar{x})^2} \quad (\text{B2-3})$$

26 Where:

27 n_0 = the initial number of samples used to calculate the preliminary estimates.

28 n = the calculated minimum number of samples to be collected.

29 $t_{\alpha, n-1}$ = the 90th percentile for the t distribution with n_0-1 degrees of freedom.

30 RT = the Regulatory Threshold of the contaminant (TC limit for toxicity characteristic wastes,
31 PRQL for listed wastes)

1 The number of samples to be collected will be based upon the largest n calculated for each of
2 the contaminants of concern. The actual number of samples collected shall be adjusted as
3 necessary to ensure that an adequate number of samples are collected to allow for acceptable
4 levels of completeness.

5 Non-integer results of calculations for the required sample size should be rounded up to the
6 next integer. A minimum of five containers shall be sampled and analyzed in each waste
7 stream. If there are fewer containers than the minimum or required number of samples in a
8 waste stream, one or more randomly selected containers shall be sampled more than once to
9 obtain the number of needed samples of the waste. Otherwise any one container may be
10 selected for sampling only once.

11 The calculated total number of required waste containers will then be randomly sampled and
12 analyzed using a Permittee approved laboratory. Waste container samples from the preliminary
13 mean and variance estimates may be counted as part of the total number of calculated required
14 samples if and only if:

- 15 • There is documented evidence that the waste containers for the preliminary estimate
16 samples were selected in the same random manner as is chosen for the required
17 samples.
- 18 • There is documented evidence that the method of sample collection in the preliminary
19 estimate samples were identical to the methodology to be employed for the required
20 samples.
- 21 • There is documented evidence that the method of sample analysis in the preliminary
22 estimate samples were identical to the analytical methodology employed for the
23 required samples.
- 24 • There is documented evidence that the validation of the sample analyses in the
25 preliminary estimate samples were comparable to the validation employed for the
26 required samples. In addition, the validated samples results shall indicate that all
27 sample results were valid according to the analytical methodology.

28 If only a portion of a waste stream is accessible for sampling (e.g., the remainder of the waste
29 stream will be recovered from storage at the generator/storage site, or only a portion of the
30 waste stream has been repackaged, treated, or generated), the calculated number of samples
31 will be randomly selected from the accessible portion of the waste stream. A minimum of five
32 randomly selected samples will be obtained and analyzed from the accessible portion of the
33 waste stream. The Permittees may approve the WSPF and authorize the generator/storage site
34 to begin shipping the waste stream to WIPP once the analytical data for the randomly selected
35 samples from the accessible portion of the waste stream have been obtained.

36 The generator/storage site will also randomly select the calculated number of sample locations
37 from the waste stream as a whole. A minimum of five randomly selected sample locations will
38 be selected from the waste stream as a whole. As those randomly selected locations (e.g.,
39 buried or newly generated waste containers) become accessible for sampling, samples will be
40 obtained and analyzed.

1 For those waste streams where the population of the waste stream as a whole is indeterminate
2 (e.g., continually generated waste streams from ongoing processes) or to facilitate waste
3 processing, the generator/storage site may divide the waste stream into lots. In this case, a
4 minimum of five randomly selected sample locations will be selected from within each
5 subsequent lot. As those randomly selected locations (e.g., buried or newly generated waste
6 containers) become accessible, samples will be obtained and analyzed. As with sampling from
7 the waste stream as a whole, the generator/storage site may ship waste from the lot being
8 generated or retrieved prior to completing sampling and analysis of the lot.

9 The generator/storage site will use the data to update the UCL₉₀ values for the waste stream as
10 described in Section B2-2a and assign EPA hazardous waste numbers as appropriate. The
11 generator/storage sites will submit the analytical data from subsequent sampling to the
12 Permittees for inclusion in the WIPP facility operating record upon completion of project level
13 data validation in Permit Attachment B3, Section B3-10b. If changes to EPA hazardous waste
14 numbers are required as a result of subsequent sampling, the generator/storage site will notify
15 the Permittees and shipments of the affected waste stream shall be suspended until the
16 Permittees approve a revised WSPF for the affected waste stream.

17 Upon collection and analysis of the preliminary samples, or at any time after the preliminary
18 samples have been analyzed, the generator/storage site may presumptively assign hazardous
19 waste numbers to a waste stream even if the calculated number of required samples is greater
20 than the preliminary number of samples collected. For waste streams with calculated upper
21 confidence limits below the regulatory threshold, the site shall collect the required number of
22 samples if the site intends to establish that the constituent is below the regulatory threshold.

23 B2-1b Statistical Selection of Containers for Headspace Gas Analysis

24 Headspace gas sampling of a waste stream may be done on a randomly selected portion of
25 containers in the waste stream. The minimum number of containers, n , that must be sampled is
26 determined by taking an initial VOC sample from ten randomly selected containers. These
27 samples are analyzed for all the target analytes analytes using a Permittee approved laboratory.
28 The standard deviation, s , is calculated for each of the nine VOCs in Module IV, Table IV.D.1.
29 The value of n is determined as the largest number of samples (not to exceed the number of
30 containers in the waste stream or waste stream lot) calculated using the following equation:

$$31 \quad n_{voc_i} = \frac{t_{\alpha, n-1}^2 s^2 e_{voc_i}}{E_{voc_i}^2} \quad (B2-4)$$

32 Where:

33 n_{voc_i} = the number of samples needed to representatively sample the waste stream for the VOC _{i}
34 from Table IV.D.1

35 $t_{\alpha, n-1}$ = the 90th percentile of the t distribution with $n-1$ degrees of freedom

36 s_{evoc_i} = the estimated standard deviation, based on the initial n samples, for VOC _{i} from
37 Table IV.D.1

1 E_{voci} = the allowable error determined as 1 percent of the limiting concentration for VOC_i from
2 Table IV.D.1

3 Non-integer results of calculations for the required sample size should be rounded up to the
4 next integer. A minimum of ten containers shall be sampled and analyzed in each waste stream.
5 If there are fewer containers than the minimum or required number of samples in a waste
6 stream, then each container should be sampled once.

7 The calculated total number of required waste containers will then be randomly sampled and
8 analyzed. Waste container samples from the preliminary mean and variance estimates may be
9 counted as part of the total number of calculated required samples if and only if:

- 10 • There is documented evidence that the waste containers for the preliminary estimate
11 samples were selected in the same random manner as is chosen for the required
12 samples.
- 13 • There is documented evidence that the method of sample collection in the preliminary
14 estimate samples were identical to the methodology to be employed for the required
15 samples.
- 16 • There is documented evidence that the method of sample analysis in the preliminary
17 estimate samples were identical to the analytical methodology employed for the
18 required samples.
- 19 • There is documented evidence that the validation of the sample analyses in the
20 preliminary estimate samples were comparable to the validation employed for the
21 required samples. In addition, the validated samples results shall indicate that all
22 sample results were valid according to the analytical methodology.

23 The mean and standard deviation calculated after sampling n containers can be used to
24 calculate a UCL_{90} for each of the headspace gas VOCs using the methodology presented in
25 Section B2-2b.

26 If only a portion of a waste stream is accessible for sampling (e.g., the remainder of the waste
27 stream will be recovered from storage at the generator/storage site or only a portion of the
28 waste stream has been repackaged or treated), the calculated number of samples will be
29 randomly selected from the accessible portion of the waste stream. A minimum of ten randomly
30 selected samples will be obtained and analyzed from the accessible portion of the waste
31 stream. The Permittees may approve the WSPF and authorize the generator/storage site to
32 begin shipping the waste stream to WIPP once the analytical data for the randomly selected
33 samples from the accessible portion of the waste stream has been obtained.

34 The generator/storage site will also randomly select the calculated number of sample locations
35 from the waste stream as a whole. A minimum of ten randomly selected sample locations will be
36 selected from the waste stream as a whole. As those randomly selected locations (e.g., buried
37 or newly generated waste containers) become accessible for sampling, samples will be
38 obtained and analyzed.

39 For those waste streams where the population of the waste stream as a whole is indeterminate
40 (e.g., continually generated waste streams from ongoing processes) or to facilitate waste

1 processing, the generator/storage site may divide the waste stream into lots. In this case, a
2 minimum of ten randomly selected containers will be selected from within each subsequent lot.
3 As those randomly selected containers (e.g., buried or newly generated waste containers)
4 become accessible, samples will be obtained and analyzed. As with sampling from the waste
5 stream as a whole, the generator/storage site may ship waste from the lot being generated or
6 retrieved prior to completing sampling and analysis of the lot.

7 The generator/storage site will use the data to update the UCL_{90} values for the waste stream as
8 described in Section B2-2b and assign EPA hazardous waste numbers as appropriate. The
9 generator/storage sites will submit the analytical data from subsequent sampling to the
10 Permittees for inclusion in the WIPP facility operating record upon completion of project level
11 data validation in Permit Attachment B3, Section B3-10b. If changes to EPA hazardous waste
12 numbers are required as a result of subsequent sampling, the generator/storage site will notify
13 the Permittees, and shipments of the affected waste stream shall be suspended until the
14 Permittees approve a revised WSPF for the affected waste stream.

15 Upon collection and analysis of the preliminary samples, or at any time after the preliminary
16 samples have been analyzed, the generator/storage site may presumptively assign hazardous
17 waste numbers to a waste stream even if the calculated number of required samples is greater
18 than the preliminary number of samples collected. For waste streams with calculated upper
19 confidence limits below the regulatory threshold, the site shall collect the required number of
20 samples if the site intends to establish that the constituent is below the regulatory threshold.

21 B2-2 Upper Confidence Limits for Statistical Sampling

22 B2-2a Upper Confidence Limit for Statistical Solid Sampling

23 Upon completion of the required sampling, final mean and variance estimates and the UCL_{90} for
24 the mean concentration for each contaminant shall be determined. The observed sample n^*
25 shall be checked against the preliminary estimate for the number of samples (n) to be collected
26 before proceeding, where n^* is:

$$27 \quad n^* = \frac{t^2_{\alpha, n-1} s^2}{(RT - \bar{x})^2} \quad (B2-5)$$

28 and the right-side terms in the equation are as defined in Section B2-1a.

29 If the observed sample n^* estimate results in greater than 20 percent or more required samples
30 than were originally calculated, then the additional samples required to fulfill the revised sample
31 estimate shall be collected and analyzed. The determination of n^* is an iterative process that
32 follows the collection and analysis of any additional samples and continues until the difference
33 between n^* and the previous sample size determination is less than 20 percent.

34 Once sufficient sampling and analysis has occurred, the waste characterization will proceed.
35 The assessment will be made at the 90 percent confidence level. The UCL_{90} for the mean
36 concentration of each contaminant will be calculated using the following equation from OSWER
37 9285.6-10 (EPA 2002):

1
$$UCL_{90} = \bar{x} + \frac{t_{\alpha, n-1} s}{\sqrt{n}} \quad (\text{B2-6})$$

2 If the UCL_{90} for the mean concentration is less than the regulatory threshold limit, the waste
3 stream is not required to be assigned the hazardous waste number for the associated
4 contaminant. If the UCL_{90} is greater than or equal to the regulatory threshold limit, the waste
5 stream will be assigned the hazardous waste number for the associated contaminant.

6 B2-2b Upper Confidence Limit for Statistical Headspace Gas Sampling

7 A UCL_{90} concentration for each of the headspace gas VOCs must be calculated from the
8 sample data collected. The observed sample n^* shall be checked against the estimate for the
9 number of samples (n) to be collected before proceeding, where n^* is:

10
$$n^* = \frac{t_{\alpha, n-1}^2 s^2}{E^2} \quad (\text{B2-7})$$

11 where E is as defined in Section B2-1b and the remaining right-side terms in the equation are
12 defined in Section B2-1a. When composite headspace gas sample results are used, the mean,
13 standard deviation, and t-statistic are based on the number of composite samples analyzed,
14 rather than the number of containers sampled.

15 If the observed sample n^* estimate results in greater than 20 percent or more required samples
16 than were originally calculated, then the additional samples required to fulfill the revised sample
17 estimate shall be collected and analyzed. The determination of n^* is an iterative process that
18 follows the collection and analysis of any additional samples and continues until the difference
19 between n^* and the previous sample size determination is less than 20 percent. The UCL_{90} is
20 then calculated using equation B2-6. In this case, UCL_{90} is the 90 percent upper confidence limit
21 for the mean VOC concentration, \bar{x} is the calculated sample mean VOC concentration and s is
22 the calculated sample standard deviation. The value of $t_{(\alpha, n-1)}$ is found in Table 9-2 of Chapter 9
23 of SW-846 (EPA, 1996).

24

1 References

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15 Washington DC.

16

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FIGURES

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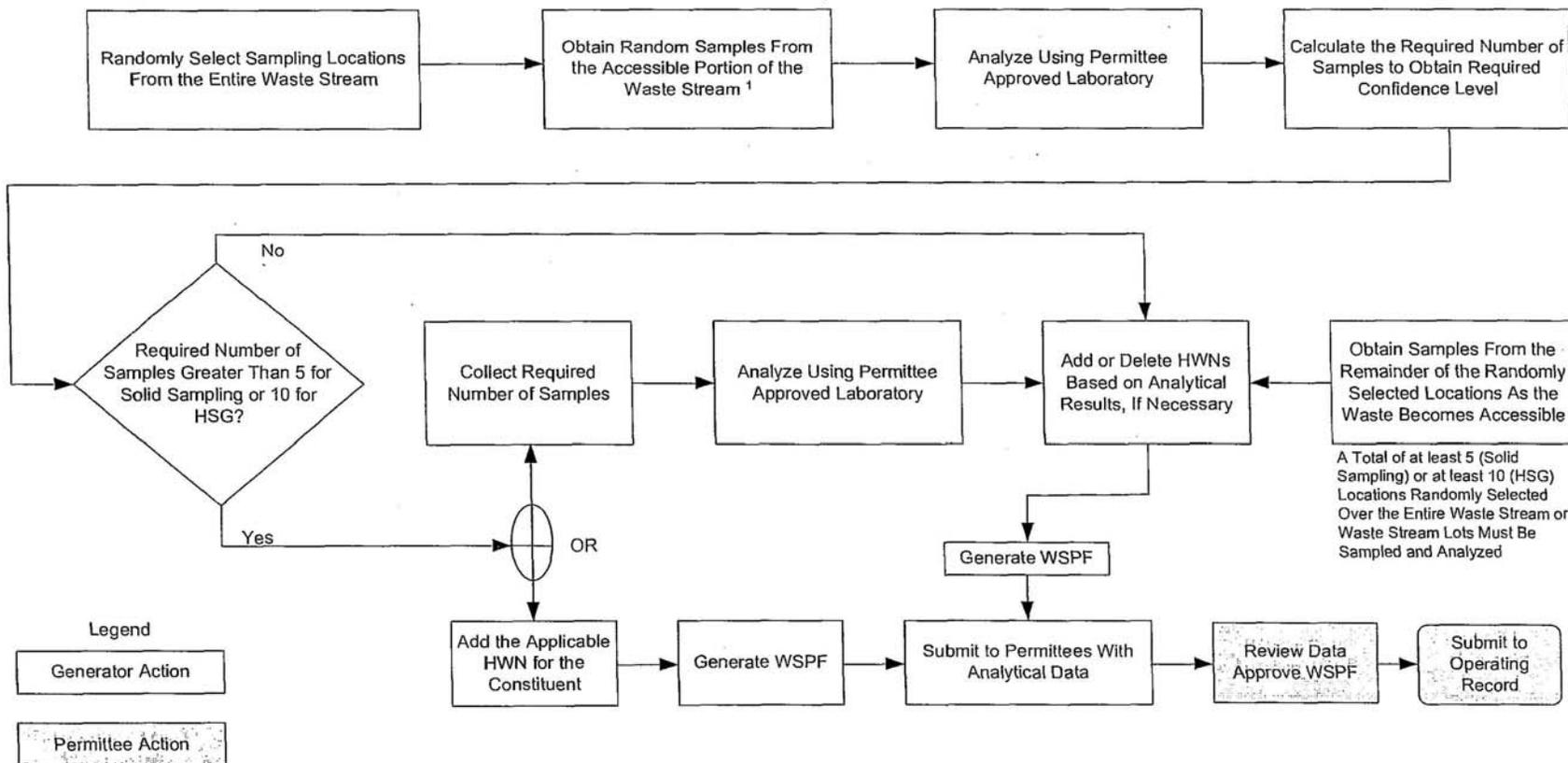


Figure B2-1
Approach for Solid and Headspace Gas Sampling and Analysis to Obtain Additional Waste Characterization Information