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**RENEWAL APPLICATION
APPENDIX B2**

STATISTICAL METHODS USED IN SAMPLING AND ANALYSIS

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**RENEWAL APPLICATION
APPENDIX B2**

STATISTICAL METHODS USED IN SAMPLING AND ANALYSIS

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1 **RENEWAL APPLICATION**
2 **APPENDIX B2**

3
4 **STATISTICAL METHODS USED IN SAMPLING AND ANALYSIS**
5

6
7 Introduction

8 The Permittees shall require certified characterization programs generator/storage sites (~~sites~~) to
9 use the following statistical methods for sampling and analysis of transuranic (TRU-TRU) mixed
10 waste which is managed, stored, or disposed at the Waste Isolation Pilot Plant (WIPP-WIPP),
11 unless determined unnecessary by the Permittees as a result of an approved Scenario 1 or 3
12 Acceptable Knowledge (~~AK~~) Sufficiency Determination. These statistical methods include
13 methods for selecting waste containers for totals analysis, selecting waste containers for
14 headspace gas (HSG) sampling and analysis, and setting the upper confidence limit.

15 B2-1 Approach for Selecting Waste Containers for Statistical Sampling

16 B2-1a Statistical Selection of Containers for Totals Analysis

17 The statistical approach for characterizing ~~retrievably stored and newly generated~~ homogeneous
18 solids (S3000) and soil/s/gravel (S4000) waste ~~and repackaged or treated S3000 waste~~ relies on
19 using acceptable knowledge (AK) to segregate waste containers into relatively homogeneous
20 waste streams. ~~Using acceptable knowledge, generator/storage sites will classify the entire waste~~
21 ~~stream as hazardous or nonhazardous rather than individual waste containers.~~ Individual waste
22 containers serve as convenient units for characterizing the combined mass of waste from the
23 waste stream of interest. Once segregated by waste stream, random selection and sampling of
24 the waste containers followed by analysis of the waste samples shall be performed to ensure that
25 the resulting mean contaminant concentration provides an unbiased representation of the true
26 mean contaminant concentration for each waste stream. The Permittees shall require each
27 certified characterization program ~~site project manager~~ Site Project Manager to verify that the
28 samples collected from within a waste stream were selected randomly.

29 An end use of analytical results for ~~retrievably stored~~ homogeneous solids and soil/s/gravel is for
30 assigning the Environmental Protection Agency (EPA) hazardous waste numbers (HWNs)
31 associated with toxicity characteristic (TC) waste (D-numbers) that apply to each mixed waste
32 stream. The ~~toxicity characteristic TC~~ D-numbers are indicators that the waste exhibits the
33 ~~toxicity characteristic TC~~ for specific contaminants under the Resource Conservation and
34 Recovery Act (**RCRA**). The ~~RCRA-toxicity TC~~ determination is made on the basis of sampling
35 and analysis of waste streams and on whether or not the waste stream includes F-listed HWNs
36 (F-number) wastes. If a waste stream includes one or more RCRA F-numbers identified via
37 ~~acceptable knowledge AK~~, ~~toxicity characteristic TC~~ contaminants associated with the F-number
38 waste(s) are not included in the ~~RCRA-toxicity characteristic TC~~ determination. That is, the F-
39 numbers take precedence over ~~RCRA-toxicity TC~~ D-number, and the waste stream is assumed
40 hazardous regardless of the concentration. Therefore, ~~toxicity characteristic TC~~ contaminants

1 associated with F-numbers for a waste stream shall be omitted from all calculations for
2 determining the number of containers to sample because these wastes streams are assumed to be
3 hazardous. In addition, each ~~toxicity characteristic~~ **TC** contaminant associated with the
4 F-number(s) shall be excluded from evaluation of analytical results to determine D-numbers.
5 Contaminants of interest for the sampling, analysis, and ~~RCRA-toxicity~~ **TC** determination of a
6 waste stream, then, excludes contaminants associated with F-numbers that have been assigned to
7 the waste stream.

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

23

$$24 \quad \bar{x} = \frac{1}{n} \sum_{i=1}^n x_i \quad (B2-1)$$

25

$$26 \quad s^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 \quad (B2-2)$$

27 Where:

28 \bar{x} = the calculated mean-

29 s^2 = the calculated concentration variance-

30 n = the number of samples analyzed-

31 x_i = the concentration determined in the i^{th} sample-

32 i = an index from 1 to n -

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

$$4 \quad n = \frac{t_{\alpha, n_0}^2 s^2}{(RT - \bar{x})^2} \quad n = \frac{t_{\alpha, n_0}^2 s^2}{(RL - \bar{x})^2} \quad (B2-3)$$

5 Where:

6 n_0 = the initial number of samples observations used to calculate the preliminary estimates-

7 n = the calculated minimum number of samples to be collected-

8 $t_{\alpha, n}$ = the 90th percentile for the t distribution with $n_0 - 1$ degrees of freedom-

9 RT = the Regulatory Threshold Level of the contaminant (toxicity characteristic TC limit for
10 toxicity characteristic wastes, PRQL for listed wastes)

11 The number of samples to be collected will be based upon the largest n calculated for each of the
12 contaminants of concern that do not have corresponding hazardous waste numbers assigned. The
13 ~~actual number of samples collected shall be adjusted as necessary to ensure that an adequate~~
14 ~~number of samples are collected to allow for acceptable levels of completeness.~~ Non-integer
15 results of calculations for the required sample size should be rounded up to the next integer.

16 A minimum of five containers shall be sampled and analyzed in each waste stream. A container
17 may be selected for sampling only once. For waste streams of five containers or less, a
18 representative sample will be collected from each container in that waste stream. The highest
19 concentration detected for each contaminant of concern will be compared to the RL. When this
20 number exceeds the RL, the associated hazardous waste number will be assigned. ~~If there are~~
21 ~~fewer containers than the minimum or required number of samples in a waste stream, one or~~
22 ~~more randomly selected containers shall be sampled more than once to obtain the number of~~
23 ~~needed samples of the waste. Otherwise any one container may be selected for sampling only~~
24 ~~once.~~

25 For waste streams with more than five containers, ~~the~~ the calculated total number of required waste
26 containers will ~~then~~ be randomly sampled and analyzed using a Permittee approved laboratory.
27 Waste container samples from the preliminary mean and variance estimates may be counted as
28 part of the total number of calculated required samples if and only if:

- 29
- 30 • There is documented evidence that the waste containers for the preliminary estimate
samples were selected in the same random manner as is chosen for the required samples.
 - 31 • There is documented evidence that the method of sample collection in the preliminary
32 estimate samples were identical to the methodology to be employed for the required
33 samples.

- 1 • There is documented evidence that the method of sample analysis in the preliminary
2 estimate samples were identical to the analytical methodology employed for the required
3 samples.
- 4 • There is documented evidence that the validation of the sample analyses in the
5 preliminary estimate samples were comparable to the validation employed for the
6 required samples. In addition, the validated samples results shall indicate that all sample
7 results were valid according to the analytical methodology.

8 For waste streams consisting of more than five containers, when If only a portion of a the waste
9 stream is accessible for sampling (e.g., the remainder of the waste stream will be recovered from
10 storage at the generator/storage TRU waste site, or only a portion of the waste stream has been
11 repackaged, treated, or generated), the calculated number of samples will be randomly selected
12 from the accessible portion of the waste stream. A minimum of five randomly selected samples
13 will be obtained and analyzed from the accessible portion of the waste stream. The Permittees
14 may approve the waste stream profile form (WSPF) and authorize the generator/storage site
15 certified characterization program to begin shipping the waste stream to WIPP once the
16 analytical data for the randomly selected samples from the accessible portion of the waste stream
17 have been obtained.

18 The generator/storage site certified characterization program will also randomly select the
19 calculated number of sample locations from the waste stream as a whole. A minimum of five
20 randomly selected sample locations will be selected from the waste stream as a whole. As those
21 randomly selected locations (e.g., buried ~~or newly generated~~ waste containers) become
22 accessible for sampling, samples will be obtained and analyzed.

23 For those waste streams where the population of the waste stream as a whole is indeterminate
24 (e.g., continually generated waste streams from ongoing processes) or to facilitate waste
25 processing, the generator/storage site certified characterization program may divide the waste
26 stream into lots. In this case, a minimum of five randomly selected sample locations will be
27 selected from within each subsequent lot. As those randomly selected locations (e.g., buried ~~or~~
28 ~~newly generated~~ waste containers) become accessible, samples will be obtained and analyzed.
29 As with sampling from the waste stream as a whole, the generator/storage site certified
30 characterization program may ship waste from the lot being generated or retrieved prior to
31 completing sampling and analysis of the lot.

32 The generator/storage site certified characterization program will use the data to update the
33 90 percent upper confidence limit UCL₉₀ (UCL₉₀) values for the waste stream as described in
34 Section B2-2a and assign EPA hazardous waste numbers HWNs as appropriate. The
35 generator/storage sites certified characterization programs will submit the analytical data from
36 subsequent sampling to the Permittees for inclusion in the WIPP facility operating record upon
37 completion of project level data validation specified in Renewal Application Appendix B3
38 (Quality Assurance Objectives and Data Validation Techniques for Waste Characterization
39 Sampling and Analytical Methods), Section B3-10b. If changes to EPA hazardous waste
40 numbers HWNs are required as a result of subsequent sampling, the generator/storage site

1 **certified characterization program** will notify the Permittees and shipments of the affected waste
 2 stream shall be suspended until the Permittees approve a revised WSPF for the affected waste
 3 stream.

4 Upon collection and analysis of the preliminary samples, or at any time after the preliminary
 5 samples have been analyzed, the ~~generator/storage site~~ **certified characterization program** may
 6 presumptively assign ~~hazardous waste numbers~~ **HWNs** to a waste stream even if the calculated
 7 number of required samples is greater than the preliminary number of samples collected. For
 8 waste streams with ~~a~~ calculated ~~upper confidence limits~~ **UCL₉₀** below the regulatory **limit**
 9 ~~threshold~~, the site **certified characterization program** shall collect the required number of samples
 10 **for a contaminant of concern** if the site **certified characterization program** intends to establish
 11 that the ~~constituent~~ **contaminant of concern** is below the regulatory **limit** threshold.

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

13 Headspace gas sampling of a waste stream may be done on a randomly selected portion of
 14 containers in the waste stream. The minimum number of containers, *n*, that must be sampled is
 15 determined by taking an initial **volatile organic compound (VOC)** sample from ten randomly
 16 selected containers. These samples are analyzed for all the target analytes using a Permittee
 17 approved laboratory. The ~~mean, \bar{x} , and the~~ standard deviation, *s*, ~~is~~ **are** calculated for **each of**
 18 **the target analytes listed in Renewal Application Appendix B3, Table B3-2** ~~nine VOCs in~~
 19 ~~Renewal Application Chapter N (Volatile Organic Compound Monitoring Plan) Table N-1 in~~
 20 ~~Module IV, Table IV.D.1.~~ The value of *n* is determined as the largest number of samples (not to
 21 exceed the number of containers in the waste stream or waste stream lot) calculated using the
 22 following:

23
$$n_{voc_i} = \frac{t_{\alpha, n-1}^2 S_{evoc_i}^2}{E_{voc_i}^2} \quad n_{voc_i} = \frac{t_{\alpha, n-1}^2 S_{evoc_i}^2}{(RT_{voc_i} - \bar{x}_{voc_i})^2} \quad (B2-4)$$

24
 25 Where:

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

28 $t_{\alpha, n-1}$ = the 90th percentile of the *t* distribution with *n*-1 degrees of freedom

29 s_{evoc_i} = the estimated standard deviation, based on the initial *n* samples, for VOC i ~~from Table~~
 30 ~~IV.D.1~~

31 E_{voc_i} ~~RT_{voc_i}~~ = the **PRQL** allowable error determined as 1 percent of the limiting concentration for
 32 VOC i from **Renewal Application Appendix B3, Table B3-2** ~~IV.D.1~~

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

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

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

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

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

32 The ~~generator/storage site~~ **certified characterization program** will also randomly select the
33 calculated number of sample locations from the waste stream as a whole. A minimum of ten
34 randomly selected sample locations will be selected from the waste stream as a whole. As those
35 randomly selected locations (e.g., buried ~~or newly generated~~ waste containers) become
36 accessible for sampling, samples will be 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 certified characterization program may divide the waste
 4 stream into lots. In this case, a minimum of ten randomly selected containers will be selected
 5 from within each subsequent lot. As those randomly selected containers (e.g., buried ~~or newly~~
 6 ~~generated~~ waste containers) become accessible, samples will be obtained and analyzed. As with
 7 sampling from the waste stream as a whole, the generator/storage site certified characterization
 8 program may ship waste from the lot being generated or retrieved prior to completing sampling
 9 and analysis of the lot.

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

20 Upon collection and analysis of the preliminary samples, or at any time after the preliminary
 21 samples have been analyzed, the generator/storage site certified characterization program may
 22 presumptively assign hazardous waste numbers EPA HWNs to a waste stream even if the
 23 calculated number of required samples is greater than the preliminary number of samples
 24 collected. For waste streams with a calculated UCL₉₀ ~~upper confidence limits~~ below the
 25 regulatory limit threshold, the site certified characterization program shall collect the required
 26 number of samples if the site certified characterization program intends to establish that the
 27 constituent is below the regulatory limit threshold.

28 B2-2 Upper Confidence Limits for Statistical Sampling

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

30 Upon completion of the required sampling, final mean and variance estimates and the UCL₉₀ for
 31 the mean concentration for each contaminant shall be determined. The observed sample ~~n~~ n_o
 32 shall be checked against the preliminary estimate for the number of samples (n*) to be collected
 33 before proceeding, where n* is:

34
$$\underline{n^* = \frac{t^2_{\alpha, n-1} S^2}{(RT - \bar{x})^2}} \quad \underline{n^* = \frac{t^2_{\alpha, n_o-1} S^2}{(RL - \bar{x})^2}} \quad (B2-5)$$

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

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

6 Once sufficient sampling and analysis has occurred, the waste characterization will proceed. The
7 assessment will be made at the 90 percent confidence level. The UCL_{90} for the mean
8 concentration of each contaminant will be calculated using the following equation from OSWER
9 9285.6-10 (EPA, 2002):

10
$$UCL_{90} = \bar{x} + \frac{t_{\alpha, n-1} S}{\sqrt{n}} \quad (B2-6)$$

11 If the UCL_{90} for the mean concentration is less than the regulatory ~~threshold~~ limit, the waste
12 stream is not required to be assigned the ~~hazardous waste number~~ **HWN** for the associated
13 contaminant. If the UCL_{90} is greater than or equal to the regulatory ~~threshold~~ limit, the waste
14 stream will be assigned the ~~hazardous waste number~~ **HWN** for the associated contaminant.

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

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

19
$$n^* = \frac{t^2_{\alpha, n-1} S^2}{E^2} \quad n^* = \frac{t^2_{\alpha, n-1} S^2}{(RT - \bar{x})^2} \quad (B2-7)$$

20 where ~~E~~ **RT** is as defined in Section B2-1b and the remaining right-side terms in the equation are
21 defined in Section B2-1a. When composite ~~headspace gas~~ **HSG** sample results are used, the
22 mean, standard deviation, and t-statistic are based on the number of composite samples analyzed,
23 rather than the number of containers sampled.

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

1

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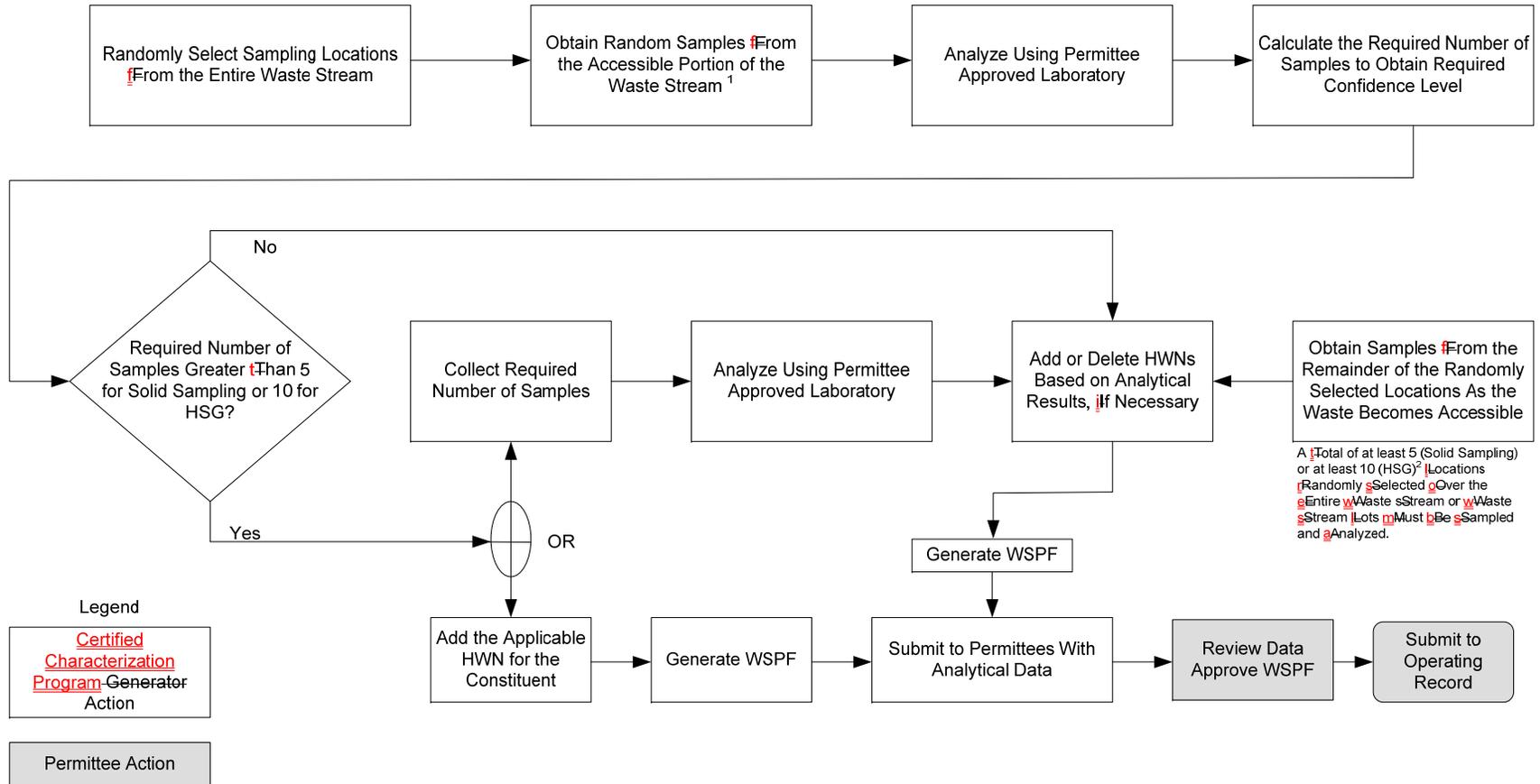
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FIGURES

1

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A Total of at least 5 (Solid Sampling) or at least 10 (HSG)² Locations Randomly Selected Over the Entire Waste Stream or Waste Stream Lots Must Be Sampled and Analyzed.

¹ Samples Are Obtained From the First Five Accessible Random Locations for Solid Sampling and the First Ten Accessible Random Locations for Headspace Gas Sampling.
² For waste streams with less than five S3000 or S4000 containers or less than ten S5000 containers each container will be sampled only once.

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