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CCP-TP-003

Revision 18

CCP
Data Analysis for S3000, S4000, and S5000 Characterization

EFFECTIVE DATE: 12/29/2010

Larry Porter

PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
4	01/17/2002	Added trans-1,2-Dichloroethylene to Attachment 2.
5	03/18/2002	Changed the word “normally” to “normality” in 4.2.6 note.
6	03/20/2002	Changed methyl chloride to methylene chloride in Attachment 2.
7	06/03/2002	Numerous changes throughout the procedure.
8	08/23/2002	Deleted last sentence of note above step 4.9.3.
9	10/10/2002	Deleted Waste Stream Lot Determination Memorandums as a record since no longer used. Made minor revisions to Attachments 3, 4, 5, and 6.
10	12/04/2002	Revised to address the UCL_{90} statistics and the normalization of data.
11	01/20/2003	Update steps 4.3.2[C], 4.3.4, and Attachment 5.
12	01/25/2003	Updated NOTE above 1.1 and step 1.1
13	06/28/2003	Updated to include solids information. Separated electronically fillable forms and updated references in procedure.
14	09/03/2003	Revised to update cross reference in step 4.1.2[F]. Revised sections 4.1 and 4.3. Revised step 4.1.2[I and J], and added section [K].
15	11/16/2006	Revised to implement the Waste Isolation Pilot Plant Hazardous Waste Facility Permit requirements resulting from the Section 311/Remote-Handled (RH) Permit Modification Request (PMR). Addressed Carlsbad Field Office (CBFO) Document Review Record (DRR) comments.
16	10/02/2007	Revised equations above Steps 4.1.2 [C] and 4.2.2 [C], changed TIC reporting requirements to be consistent with the Waste Isolation Pilot Plant Hazardous Waste Facility Permit (HWFP), Waste Analysis Plan (WAP), and minor editorial changes. Revised to incorporate statistical terminology and text changes included in September 2007 Class 1 HWFP Notifications.
17	11/09/2009	Revised to delete a reference that is no longer applicable and add the new reference. Also to update attachments and correct editorial errors.
18	12/29/2010	Revised to implement the revision of the *Waste Isolation Pilot Plant Hazardous Waste Facility Permit*.

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1.0 PURPOSE

This procedure describes actions for the Central Characterization Project (CCP) to evaluate data on transuranic (TRU) waste streams destined for disposal at the Waste Isolation Pilot Plant (WIPP). Compliance with this procedure ensures that sufficient data of acceptable quality and quantity has been collected to meet applicable requirements from CCP-PO-001, *CCP Quality Assurance Project Plan*, Sections C2-1 and C3-1.

The Headspace Gas Summary Data Report and Solids Summary Data Report (if applicable) developed by this procedure is/are required before assembly of a Waste Stream Profile Form (WSPF) (see CCP-TP-002, *CCP Reconciliation of DQOs and Reporting Characterization Data*).

1.1 Scope

This procedure applies to S3000 homogeneous solids, and S4000 soil/gravel TRU waste streams that require solids sampling and analysis; and S5000 debris TRU waste streams that require headspace gas (HSG) sampling and analysis for disposal at the WIPP. This procedure will be implemented for each sampling activity within a waste stream.

2.0 REQUIREMENTS

2.1 References

- CCP-PO-001, *CCP Transuranic Waste Characterization Quality Assurance Project Plan*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*
- CCP-QP-006, *CCP Corrective Action Reporting and Control*
- CCP-QP-008, *CCP Records Management*
- CCP-TP-002, *CCP Reconciliation of DQOs and Reporting Characterization Data*
- CCP-TP-005, *CCP Acceptable Knowledge Documentation*
- CCP-TP-162, *CCP Random Selection of Containers for Solids and Headspace Gas Sampling and Analysis*
- Clarification Number CAO-00-065, *Determination of Tentatively Identified Compounds (TICS)*

- Title 20 *New Mexico Administrative Code* (NMAC), Chapter 4, *Hazardous Waste,* Part I, *Hazardous Waste Management,* Section 200, *Adoption of 40 CFR Part 261*

- Title 40 *Code of Federal Regulations* (CFR), *Protection of the Environment,* Chapter 1, *Environmental Protection Agency,* Subchapter 1, *Solid Wastes,* Part 261, *Identification and Listing of Hazardous Waste,* Appendix VIII, *Hazardous Constituents*

- Title 40 CFR, Chapter 1, Subchapter I, Part 261, Subpart C, *Characteristics of Hazardous Waste* §261.24, *Toxicity Characteristic*

2.2 Training Requirements

2.2.1 Personnel performing this procedure will be trained and qualified in accordance with CCP-QP-002, *CCP Training and Qualification Plan*, prior to performing this procedure.

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3.0 RESPONSIBILITIES

3.1 Site Project Manager (SPM)

3.1.1 Oversees all program activities within CCP including the following responsibilities associated with process control:

[A] Provides data to support the calculations for each evaluation.

[B] Assigns U.S. Environmental Protection Agency (EPA) Hazardous Waste Numbers based on analysis results.

[C] Assigns Tentatively Identified Compounds (TICs) to the target analyte list.

[D] Calculates the number of samples required for HSG sampling, and analysis of S5000 and solids analysis of S3000 and S4000 waste streams.

3.1.2 Uses data from HSG analysis and solids sampling Batch Data Reports (BDRs) to:

[A] Determine and document the Upper 90 Percent Confidence Level (UCL_{90}) for the mean concentration and associated statistics for each waste constituent and waste stream.

[B] Use the UCL_{90} to assign EPA Hazardous Waste Numbers to containers.

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4.0 PROCEDURE

NOTE

Steps may be performed concurrently at SPM discretion.

NOTE

Initial and Subsequent Waste Stream Lot Size Determination

The number of containers in any waste stream lot is not a fixed quantity and may vary as determined by the SPM. Prior to initial submittal of a WSPF, the SPM must perform reconciliation with Data Quality Objectives (DQOs) as directed in CCP-TP-002. This activity assures that data of sufficient quantity and quality have been acquired and evaluated to characterize the initial waste stream lot. This procedure will be implemented for each sampling activity within a waste stream.

UCL_{90} and TIC Determinations and Evaluation for Initial Waste Stream Lots

UCL_{90} and TIC determinations and evaluations will be performed using HSG (S5000 debris TRU waste streams) and solids sampling (S3000 homogeneous solids and S4000 soil/gravel TRU waste streams) analysis data from the required number of containers randomly selected from the waste stream lot. If the SPM determines that additional EPA Hazardous Waste Numbers must be added to the WSPF in addition to those determined by Acceptable Knowledge (AK), or that TICs must be added to the target analyte list, then the new EPA Hazardous Waste Numbers or additional target analytes will be applicable to all subsequent containers of the waste stream.

UCL_{90} and TIC Determinations and Evaluation for Subsequent Waste Stream Lots

After characterization and approval of a WSPF for the waste stream lot, waste containers will continue to be characterized in waste stream lots. UCL_{90} and TIC determinations and evaluations will be performed on a running total basis. That is, using HSG and solids sampling analysis data from all randomly selected containers characterized to date prior to a planned shipment including those in the current waste stream lot as well as any previously characterized waste stream lots. If after the evaluation, the SPM determines that additional EPA Hazardous Waste Numbers must be added to the WSPF, or that TICs must be added to the target analyte list, the new EPA Hazardous Waste Numbers or additional target analytes will be applicable to all subsequent containers of the waste stream.

4.1 Determination of Adequate Sample Size for Solids Samples

NOTE

The method used to collect samples of TRU mixed waste, classified as S3000 and S4000 waste streams, from waste containers shall be such that the samples are representative of the waste from which they are taken. A sufficient number of samples shall be collected to adequately represent waste being sampled. A statistically selected portion of each S3000 and S4000 waste stream is sampled and analyzed for Resource Conservation and Recovery Act (RCRA)-regulated total volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), and metals.

NOTE

The analytical results for retrievably stored S3000 and S4000 waste streams are used for assigning the EPA Hazardous Waste D-numbers that apply to each mixed waste stream. The RCRA-toxicity determination may be made on the basis of sampling and analysis of waste streams and on whether or not the waste stream includes F-number wastes. If a waste stream includes one or more RCRA F-numbers identified via AK, toxicity characteristic contaminants associated with the F-number waste(s) are not included in the RCRA-toxicity characteristic determination. Therefore, toxicity characteristic contaminants associated with F-number(s) for a waste stream shall be omitted from all calculations for determining the number of containers to sample because these waste streams are assumed to be hazardous. In addition, each toxicity characteristic contaminant associated with the F-number(s) shall be excluded from evaluation of analytical results to determine D-number. Contaminants of interest for the sampling, analysis, and RCRA-toxicity determination of a waste stream, then, exclude contaminants associated with F-numbers that have been assigned to the waste stream.

4.1.1 Obtain the analytical results of the sample set selected by CCP-TP-162, *CCP Random Selection of Containers of Solids and Headspace Gas Sampling and Analysis* as follows:

[A] Obtain a copy of the Random Solids Sample Selection Memorandum from CCP Records.

[B] Obtain, from CCP Records, a copy of the solids analytical BDR(s) that correspond to the container numbers from the Random Solids Sample Selection Memorandum.

[C] Confirm that the identified containers have successfully passed the project level validation and verification.

[D] From the BDR(s), obtain the analytical results for the containers identified in step 4.1.1[A].

4.1.2 For each chemical contaminant of concern, determine the mean and standard deviation (preliminary estimates) from the results using the following steps:

[A] Input the solid sample results into the Solid Analysis Evaluation Spreadsheet.

NOTE

The mean and variance (squared standard deviation) equations are:

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2$$

Where:

$\bar{x}$ = the calculated mean

s^2 = the calculated concentration variance

n = number of samples analyzed

x_i = concentration determined in the *ith* sample

i = an index from 1 to *n*.

$$n = \frac{t^2_{a,n_o-1}s^2}{(RT - \bar{x})^2}$$

Where:

n_o = the initial number of samples used to calculate the preliminary estimates

n = the calculated minimum number of required samples

$\bar{x}$ = the calculated mean

s^2 = the calculated concentration variance

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

RT = Regulatory Threshold of the contaminate (toxicity characteristic [TC] limit for toxicity characteristic wastes, Program Required Quantitation Limit [PRQL] for listed wastes)

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[B] Based upon the preliminary estimates of $\bar{x}$ and s^2 for each chemical contaminant of concern, estimate the appropriate number of samples (n) to be collected for each contaminant using the Solids Analysis Evaluation Spreadsheet.

NOTE

The number of samples to be collected will be based upon the largest number calculated for each of the contaminants of interest. The actual number of samples collected shall be adjusted, as necessary, to ensure that an adequate number of samples are collected to allow for acceptable levels of completeness.

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

Waste containers from the preliminary mean and variance estimates may be counted as part of the total number of calculated required samples, if, and only if, the requirements specified in CCP-PO-001, Section C2-1a, are satisfied. The CCP may count waste container samples from the preliminary mean and variance estimates as part of the total number of calculated required samples if and only if:

- 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.
- There is documented evidence that the method of sample collection in the preliminary estimate samples were identical to the methodology to be employed for the required samples.
- There is documented evidence that the method of sample analysis in the preliminary estimate samples was identical to the analytical methodology employed for the required samples.
- There is documented evidence that the validation of the sample analyses, in the preliminary estimate samples, was comparable to the validation employed for the required samples. In addition, the validated sample results indicate that all sample results were valid according to the analytical methodology.

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NOTE

To calculate the percent difference of *n* use the following formula:

$$\% \text{ Difference} = \left(\frac{n_{VOC_i} - n_{VOC_{i-1}}}{n_{VOC_{i-1}}} \right) * 100$$

Where:

n_{VOC_i} = The determination of n_{VOC_i} is an iterative process that continues until the difference between n_{VOC_i} and the previous sample determination is less than 20 percent difference.

[C] Calculate the percent difference between the required number of samples estimated in step 4.1.2[B] and the number of samples actually taken and analyzed.

[D] Based on the percent difference calculations,
IF the observed sample n estimate results in greater than 20 percent more required samples than were originally calculated,
THEN:

- Direct additional samples be taken as required to ensure that the cumulative total number of samples for each analyte equals or exceeds the number estimated in step 4.1.2[B].
- Using the analysis results for the combined sample set, reperform steps 4.1.2[A] through 4.1.2[D].

4.1.3 Upper Confidence Limit Determination for Solids Analysis

NOTE

Normality testing should not be performed using analyte concentrations reported as Minimum Detection Limit (MDL) as identified by a U flag. These should be included in the calculations for UCL_{90} as ½ the reported MDL. Subsequent lots will be added to the UCL_{90} data set as the waste stream is characterized until characterization is complete for the entire waste stream.

NOTE

If for any container the sample was diluted for analysis and the constituent concentration is reported as not detected (U flag), the result is omitted from the distribution tests and subsequent analyses when the sample minimum detectable limit is greater than the PRQL.

[A] Using the Solids Analysis Evaluation Spreadsheet, determine, **AND** record on the appropriate form, Attachment 4, CCP Solids Analysis VOC UCL_{90} Evaluation Form, Attachment 5, CCP Solids Analysis SVOCs UCL_{90} Evaluation Form or Attachment 6, CCP Solids Analysis Metals UCL_{90} Evaluation Form, the following items:

- Number of Samples,
- Number of Samples above MDL,
- Maximum (mg [milligram]/kg [kilogram]) for each target analyte,
- Mean (mg/kg) for each target analyte,
- Standard Deviation (mg/kg) for each target analyte, and
- Additional target analyte compounds (as applicable).

NOTE

For any normality test performed, the probability of a Type I error, α, should not exceed 10 percent.

NOTE

To determine data or transform that is closest to normal for data sets greater than 200, the form with the smallest D value under the Kolmogorov-Smirnov test for normality should be used. For data sets less than or equal to 200, the form with the largest W value under the Shapiro-Wilk test for normality should be used.

NOTE

If transformation of the data is required to achieve normality, the EPA Solid Waste and Sample Plan guidance may be used.

[B] **WHEN** sufficient data has been collected,
THEN perform test(s) for normality on the data.

[C] Use the data or transform that is closest to normal on the Solids Analysis Evaluation Spreadsheet to determine the UCL_{90} for the mean concentration and associated statistics of each VOC, SVOC, and Metals detected in container solid sample for each waste stream or waste stream lot.

NOTE

The formula for calculating UCL_{90} is:

$$UCL_{90} = \bar{x} + \frac{t_{\alpha,n-1}s}{\sqrt{n}}$$

Where:

$\bar{x}$	=	the calculated sample mean
n	=	the number of samples
$t_{\alpha, n_0 - 1}$	=	the 90th percentile for the *t* distribution with n_0-1 degrees of freedom
s	=	the calculated sample standard deviation

NOTE

If transformations were performed on the data, comparisons must be made using the corresponding transformed values for PRQLs.

[D] Record the required UCL_{90} (mg/kg) data on the appropriate UCL_{90} Evaluation Form (Attachment 4, Attachment 5, and Attachment 6, as necessary).

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[E] Compare the UCL_{90} for the mean concentration and PRQL (or transformed PRQL) for each constituent.

[F] Mark YES in the appropriate column on Attachments 4, 5, or 6 for any constituent where the UCL_{90} exceeds the PRQL.

[G] Enter the EPA Hazardous Waste Number for the analyte found for each entry where the UCL_{90} result exceeds the PRQL.

[G.1] **WHEN** there is NO associated EPA Hazardous Waste Number **OR** an EPA Hazardous Waste Number is inappropriate,
THEN explain with a note on Attachments 4, 5, or 6.

[H] Document and provide data analysis methodology and results on Attachment 7, CCP Data Evaluation Narrative.

[H.1] The Data Evaluation Narrative may include the following information (where applicable):

- WSPF Number
- Distribution test(s) used
- Distribution (normality) test result(s)
- Transformation(s), if any, applied to the data to achieve normality
- Comparison of transformed data results to PRQL, where applicable
- How non-detectable measurements are incorporated in the data set
- Proportion of non-detectable measurements
- Non-detectable measurements omitted for dilution reasons

[I] Cross-reference Container Identification (ID) Numbers, Solids Sample ID Numbers, and BDR Numbers by completing Attachment 2, CCP Correlation of Container ID Numbers to Solids Sample ID Numbers.

[J] Repeat step 4.1.3 each time additional sample results are available.

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[K] Print name, sign, and date Attachment 2.

NOTE

Upon collection and analysis of the preliminary samples, or at any time after the preliminary samples have been analyzed, the generator/storage site may assign EPA Hazardous Waste Numbers to a waste stream. For waste streams with the calculated upper confidence limits below the regulatory threshold, the generator/storage site shall collect the required number of samples if the generator/storage site intends to establish the constituent is below the regulatory threshold.

4.2 Determination of Adequate Sample Size for HSG Samples

4.2.1 Obtain the analytical results of the sample set selected by CCP-TP-162 as follows:

[A] Obtain a copy of the Random Headspace Gas Sample Selection Memorandum from CCP Records.

[B] Obtain, from CCP Records, a copy of the HSG analytical BDR(s) that corresponds to the container numbers from the Random Headspace Gas Sample Selection Memorandum.

[C] Confirm that the identified containers have successfully passed the project level validation and verification.

[D] From the BDR(s), obtain the analytical results for the containers identified in step 4.2.1[A].

4.2.2 For each chemical contaminant of concern, determine the mean and standard deviation (preliminary estimates) from the results using the following steps:

[A] Input the HSG sample results into the Headspace Gas Evaluation Spreadsheet.

NOTE

The mean and standard deviation equations are:

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2$$

Where:

$\bar{x}$	=	the calculated sample mean
s^2	=	the calculated concentration sample variance
n	=	number of samples analyzed
x_i	=	concentration determined in the ith sample
i	=	an index from 1 to n

$$n_{VOC_i} = \frac{t^2{}_{\alpha,n-1} s^2{}_{e_{VOC_i}}}{E^2{}_{VOC_i}}$$

Where:

n_{VOC_i}	=	the number of samples needed to representatively sample the waste stream for the VOC_i
$t_{\alpha,\, n_0 - 1}$	=	the 90th percentile for the t distribution with n_0-1 degrees of freedom (See Table 1, t Distribution for the 90th Percentile).
$s_{e_{VOC_i}}$	=	the estimated standard deviation, based on the initial n samples for VOC_i
E_{VOC_i}	=	the allowable error determined as 1 percent of the limiting concentration for VOC_i

[B] Based upon the preliminary estimates of $\bar{x}$ and s^2 for each chemical contaminant of concern, estimate the appropriate number of samples (n_{VOC_i}) to be collected for each contaminant using the Headspace Gas Evaluation Spreadsheet.

NOTE

The number of samples to be collected will be based upon the largest number calculated for each of the contaminants of interest.

Non-integer results of calculations for the required sample size should be rounded up to the nearest integer. A minimum of 10 containers shall be sampled and analyzed in each waste stream. If there is fewer than the minimum or required number of containers in a waste stream, then each container should be sampled once.

Waste containers from the preliminary mean and variance estimates may be counted as part of the total number of calculated required samples, if, and only if, the requirements specified in the CCP-PO-001, Section C2-1b, are satisfied. The CCP may count waste container samples from the preliminary mean and variance estimates as part of the total number of calculated required samples if and only if:

- 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
- There is documented evidence that the method of sample collection in the preliminary estimate samples were identical to the methodology to be employed for the required samples
- There is documented evidence that the method of sample analysis in the preliminary estimate samples was identical to the analytical methodology employed for the required samples
- There is documented evidence that the validation of the sample analyses, in the preliminary estimate samples, was comparable to the validation employed for the required samples. In addition, the validated sample results indicate that all sample results were valid according to the analytical methodology

NOTE

To calculate the percent difference of n, use the following formula:

$$\text{\% Difference} = \left(\frac{n_{VOC_i} - n_{VOC_{i-1}}}{n_{VOC_{i-1}}} \right) * 100$$

Where:

n_{VOC_i} = the determination of n_{VOC_i} is an iterative process that continues until the difference between n_{VOC_i} and the previous sample determination is less than 20 percent difference.

[C] Calculate the percent difference between the required number of samples estimated in step 4.2.2[B] and the number of samples actually taken and analyzed.

[D] Based on the percent difference calculations,
IF the observed sample n_{VOC_i} estimate results in greater than 20 percent more required samples than were originally calculated,
THEN:

- Direct additional samples be taken as required to ensure that the cumulative total number of samples for each analyte equals or exceeds the number estimated in step 4.2.2[B].
- Using the analysis results for the combined sample set, reperform steps 4.2.2[A] through [D].

4.2.3 Calculating UCL_{90} for Headspace Gas

NOTE

Normality testing should not be performed using analyte concentrations reported as MDL as identified by a U flag. These should be included in the calculations for UCL_{90} as ½ the reported MDL. Subsequent lots will be added to the UCL_{90} data set as the waste stream is characterized until characterization is complete for the entire waste stream.

NOTE

If for any container the sample was diluted for analysis and the constituent concentration is reported as not detected (U flag), the result is omitted from the distribution tests and subsequent analyses when the sample MDL is greater than the PRQL.

[A] Using the Headspace Gas Evaluation Spreadsheet, determine and record on Attachment 3, CCP Headspace Gas UCL90 Evaluation Form, the following items:

- WSPF Number,
- Number of Samples,
- Number of Samples above MDL,
- Maximum (parts per millions volume [ppmv]) for each target analyte,
- Mean (ppmv) for each target analyte,

- Standard deviation (ppmv) for each target analyte, and
- Additional target analyte compounds (as applicable).

NOTE

For any normality test performed, the probability of a Type I error, α, should not exceed 10 percent.

NOTE

To determine data or transform that is closest to normal for data sets greater than 200, the form with the smallest D value under the Kolmogorov-Smirnov test for normality should be used. For data sets less than or equal to 200 the form with the largest W value under the Shapiro-Wilk test for normality should be used.

NOTE

If transformation of the data is required to achieve normality, the EPA Solid Waste and Sample Plan guidance may be used.

[B] **WHEN** sufficient data has been collected (i.e., a waste lot is complete [a defined group bounding the random selection]), **THEN** perform test(s) for normality on the data.

[C] Use the data or transform that is closest to normal on the Headspace Gas Evaluation Spreadsheet to determine the UCL_{90} for the mean concentration and associated statistics of each VOC detected in the container HSG for each waste stream or waste stream lot.

NOTE

The formula for calculating UCL_{90} is:

$$UCL_{90} = \bar{x} + \frac{t_{\alpha,n-1}s}{\sqrt{n}}$$

Where:

$\bar{x}$	=	the calculated sample mean
n	=	the number of samples
t_{α,n_0-1}	=	the 90th percentile for the t distribution with n_0-1 degrees of freedom
s	=	the calculated sample standard deviation

NOTE

If transformations were performed on the data, comparisons must be made using the corresponding transformed values for PRQLs.

[D] Record the required UCL_{90} (ppmv) data on Attachment 3.

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[E] Compare the UCL_{90} for the mean concentration and PRQL, (or transformed PRQL) for each constituent.

[F] Mark YES in the appropriate column on Attachment 3 for any constituent where the UCL_{90} exceeds the PRQL.

[G] Enter the EPA Hazardous Waste Number for the analyte found for each entry where the UCL_{90} result exceeds the PRQL.

[G.1] **WHEN** there is NO associated EPA Hazardous Waste Number **OR** an EPA Hazardous Waste Number is inappropriate,
THEN explain with a note on Attachment 3.

[H] Document and provide data analysis methodology and results on Attachment 7.

[H.1] The Data Evaluation Narrative may include the following information (where applicable):

- WSPF Number
- Distribution test(s) used
- Distribution (normality) test result(s)
- Transformation(s), if any, applied to the data to achieve normality
- Comparison of transformed data results to PRQL, where applicable
- How non-detectable measurements are incorporated in the data set
- Proportion of non-detectable measurements
- Non-detectable measurements omitted for dilution reasons

[I] Cross-reference Container ID Numbers, HSG Sample ID Numbers, and BDR Numbers by completing Attachment 1, CCP Correlation of Container ID Numbers to Headspace Gas Sample ID Number.

[J] Print name, sign, and date Attachment 1.

4.3 TIC Evaluation

4.3.1 Evaluate the data for HSG or solids (separately), **AND** perform the following:

[A] Document the WSPF # and number of samples on Attachment 8, CCP Waste Stream Headspace Gas Tentatively Identified Compounds, or Attachment 9, CCP Waste Stream Solids Tentatively Identified Compounds.

[B] **IF** TICs were identified in this waste stream, **THEN** document TICs, Chemical Abstract Service (CAS) Number, and Number of Samples containing that TIC on the appropriate form, Attachment 8, or Attachment 9.

[C] Record "Yes" or "No" in the appropriate column on Attachment 8 or Attachment 9, depending on the sample matrix, whether the TICs appear in Title 20 New Mexico Administrative Code (NMAC), Chapter 4, Hazardous Waste, Part I, Hazardous Waste Management, Section 200, Adoption of 40 CFR Part 261 (incorporating Title 40 Code of Federal Regulations (CFR), Protection of the Environment, Chapter 1, Environmental Protection Agency, Subchapter 1, Solid Wastes, Part 261, Identification and Listing of Hazardous Waste, Appendix VIII, Hazardous Constituents) by comparing CAS numbers.

4.3.2 Obtain a copy of the completed Attachment 10, CCP Waste Stream Headspace Gas Tentatively Identified Compounds (Cumulative), or Attachment 11, CCP Waste Stream Solids Tentatively Identified Compounds (Cumulative), from the last sampling activity reviewed from CCP Records, **AND** record the following on current Attachment 10 or Attachment 11 to create accumulative list of TICs in the waste stream:

NOTE

When the evaluation is the first in the waste stream, the information from Attachment 8 or Attachment 9 must be transferred to either Attachment 10 or Attachment 11.

[A] Update the Total number of samples evaluated to date in the waste stream on Attachment 10 or Attachment 11, by adding the Number of Samples on Attachment 8 or Attachment 9 to the total number on the previous Attachment 10 or Attachment 11.

[B] Copy the list of TICs and CAS Numbers from the last sampling activity evaluated, to Columns 1 and 2 of the new Attachment 10 or Attachment 11.

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[C] Total the number of samples containing the TICs by adding the number for each individual TIC (Column 4) from the previous Attachment 10 or Attachment 11 and the number for any additional TICs (third column) from the current evaluation Attachment 8 or Attachment 9 creating the new cumulative number.

[D] Copy any new TICs (CAS number and total number of samples containing the TIC) identified in the waste stream TIC evaluation (Attachment 8 or Attachment 9) to Attachment 10 or Attachment 11, Columns 1, 2, and 4.

4.3.3 Calculate, **AND** document on Attachment 10 or Attachment 11, on an individual TIC basis, the percent of samples in the waste stream that contain the TIC.

[A] Divide the number of samples reporting the TIC from Column 4 by the total number of samples evaluated to date.

[B] Multiply by 100 to obtain the percent and record in Column 5 of Attachment 10 or Attachment 11.

4.3.4 Mark the maximum observed concentration for the TIC in Column 3 on Attachment 10 or Attachment 11.

4.3.5 Mark Yes in Column 6 on Attachment 10 or Attachment 11, those TICs that appear in 25 percent or greater of the samples in the waste stream.

4.3.6 Review Attachment 8, Attachment 9, Attachment 10, and/or Attachment 11.

4.3.7 **IF** TICs were detected in greater than or equal to 25 percent of all samples from the waste stream, and the TICs appear in the Title 20 NMAC, Chapter 4, Part I, Section 200 (incorporating Title 40 CFR, Chapter 1, Subchapter 1, Part 261, Appendix VIII),
THEN instruct the AK Expert (AKE) to perform a re-evaluation of AK in accordance with CCP-TP-005, *CCP Acceptable Knowledge* Documentation to determine whether the TIC can be attributed to waste packaging materials, radiolysis, or other origins.

4.3.8 **IF** (after the re-evaluation of AK) an F-Listed waste constituent TIC can **NOT** be attributed to waste packaging materials, radiolysis, or other origins,

OR

IF the TIC subject to inclusion on the target analyte list exhibits a toxicity characteristic (i.e., those compounds shown in 40 CFR, Chapter 1, Subchapter I, Part 261, Subpart C, Characteristics of Hazardous Waste § 261.24, Toxicity Characteristic as D EPA Hazardous Waste Number),
THEN add the TIC to the target analyte list regardless of the origin since the hazardous waste designation for D codes is not based on source or use.

[A] Issue a memorandum for addition of TICs to target analyte list, directing the HSG and solids analysis personnel (depending on analysis performed) to add the constituent to the target analyte list.

[B] Assign a PRQL as follows:

[B.1] Include the Waste Stream Identification Number and the analytes that will be listed on the new target analyte list.

[B.2] PRQL for Solids: Use 40 parts per million (ppm) for solids without a toxicity characteristic limit (in accordance with Clarification Number CAO-00-065, *Determination of Tentatively Identified Compounds (TICS)*).

[B.3] For Gases: Use 100 ppm for the PRQL for alcohols and ketones, and use 10 ppm for the PRQL for all others.

[C] Submit a copy of the memorandum to CCP Records in accordance with CCP-QP-008.

[D] Request AKE to revise the respective AK Summary Report to add the new EPA Hazardous Waste Number(s) to the waste stream as appropriate.

[E] Send a copy of the memorandum and the revised target analyte list to the Carlsbad Field Office (CBFO) and the WIPP Waste Information System (WWIS).

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4.3.9 **IF** changes to EPA Hazardous Waste Numbers are required as a result of subsequent sampling,
THEN notify the CBFO, **AND** suspend shipments of the affected waste stream until the CBFO approves revised WSPF for the affected waste stream.

4.3.10 Complete a new WSPF in accordance with CCP-TP-002.

4.3.11 Document results on Attachment 12, Site Project Manager Data Evaluation Narrative.

[A] Print name, sign, and date Attachment 12.

4.4 HSG Summary Data Report

4.4.1 Assemble the completed Attachment 1, Attachment 3, Attachment 7, Attachment 8, Attachment 10, and Attachment 12.

4.4.2 Paginate the HSG Summary Data Report.

4.4.3 Add a table of contents to the HSG Summary Data Report.

4.4.4 Transmit the Headspace Gas Summary Data Report as a memorandum to CCP Records in accordance with CCP-QP-008.

4.5 Solids Summary Data Report

4.5.1 Assemble the completed Attachment 2, Attachment 4, Attachment 5, Attachment 6, Attachment 7, Attachment 9, Attachment 11 and Attachment 12.

4.5.2 Paginate the Solids Summary Data Report.

4.5.3 Add a table of contents to the Solids Summary Data Report.

4.5.4 Transmit the Solids Summary Data Report as a memorandum to CCP Records in accordance with CCP-QP-008.

5.0 RECORDS

5.1 Records generated during the performance of this procedure are maintained as Quality Assurance records in accordance with CCP-QP-008. The records are the following:

5.1.1 QA/Lifetime

[A] Headspace Gas Summary Data Report:

[A.1] Attachment 1, CCP Correlation of Container ID Numbers to Headspace Gas Sample ID Number

[A.2] Attachment 3, CCP Headspace Gas UCL_{90} Evaluation Form

[A.3] Attachment 7, CCP Data Evaluation Narrative

[A.4] Attachment 8, CCP Waste Stream Headspace Gas Tentatively Identified Compounds

[A.5] Attachment 10, CCP Waste Stream Headspace Gas Tentatively Identified Compounds (Cumulative)

[A.6] Attachment 12, CCP Site Project Manager Data Evaluation Narrative

[B] Solids Summary Data Report:

[B.1] Attachment 2, CCP Correlation of Container ID Numbers to Solids Sample ID Numbers

[B.2] Attachment 4, CCP Solids Analysis VOC UCL_{90} Evaluation Form

[B.3] Attachment 5, CCP Solids Analysis SVOC UCL_{90} Evaluation Form

[B.4] Attachment 6, CCP Solids Analysis Metals UCL_{90} Evaluation Form

[B.5] Attachment 7, CCP Data Evaluation Narrative

[B.6] Attachment 9, CCP Waste Stream Solids Tentatively Identified Compounds

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[B.7] Attachment 11, CCP Waste Stream Solids Tentatively Identified Compounds (Cumulative)

[B.8] Attachment 12, CCP Site Project Manager Data Evaluation Narrative

5.1.2 QA/Nonpermanent

[A] Memorandum directing HSG and/or solids personnel to add constituent(s) to the target analyte list.

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Table 1 – *t* Distribution for the 90th Percentile

Degrees of Freedom (n_o-1)	*t* Value
1	3.078
2	1.886
3	1.638
4	1.533
5	1.476
6	1.440
7	1.415
8	1.397
9	1.383
10	1.372
11	1.363
12	1.356
13	1.350
14	1.345
15	1.341
16	1.337
17	1.333
18	1.330
19	1.328
20	1.325
21	1.323
22	1.321
23	1.319
24	1.318
25	1.316
26	1.315
27	1.314
28	1.313
29	1.311
30	1.303
40	1.310
60	1.296
120	1.289
∞	1.282

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Attachment 1 – CCP Correlation of Container ID Numbers to Headspace Gas Sample ID Numbers (EXAMPLE)

Page ____ of ____

WSPF Number:_____ Lot Number: _____

Container ID Number	HSG Sample ID Number	Headspace Gas BDR (Sample)	Headspace Gas BDR (Analysis)	

Use multiple copies of this attachment as necessary to document all containers discussed in this summary report. Record Waste Stream Profile Form Number and Waste Stream Lot Number on each sheet issued. Number each sheet in the space provided.

Site Project Manager (or designee):

Signature Print Name Date

Attachment 2 – CCP Correlation of Container ID Numbers to Solids Sample ID Numbers (EXAMPLE)

Page ____ of ____

WSPF Number: _____Lot Number: _____

Container ID Number	Sample BDR	VOC Analysis BDR	VOC Analysis Sample ID	SVOC Analysis BDR	SVOC Analysis Sample ID	Non-Halogenated Analysis BDR VOCs	Sample ID Non-Halogenated VOCs	Metals Analysis BDR	Metals Analysis Sample ID

Use multiple copies of this attachment as necessary to document all containers discussed in this summary report. Record Waste Stream Profile Form Number and Waste Stream Lot Number on each sheet issued. Number each sheet in the space provided.

Site Project Manager (or designee):

Signature Print Name Date

Attachment 3 – CCP Headspace Gas UCL_{90} Evaluation Form (EXAMPLE)

Page ____ of ____

WSPF Number: _____ Lot Number: _____

ANALYTE	Transform Data Used (No, Data-Log, SQTI, other)	# Samples	# Samples above MDL	Maximum (ppmv)	Mean (ppmv)	Standard Deviation (ppmv)	UCL_{90} (ppmv)	PRQL (ppmv)	Transformed PRQL (N/A or Value)	UCL_{90} > PRQL Yes	EPA Hazardous Waste Number
Benzene								10			
Bromoform								10			
Carbon tetrachloride								10			
Chlorobenzene								10			
Chloroform								10			
Cyclohexane[a]											
1,1-Dichloroethane								10			
1,2-Dichloroethane								10			
1,1-Dichloroethylene								10			
trans-1,2-Dichloroethylene								10			
Ethyl benzene								10			
Ethyl ether								10			
Methylene chloride								10			
1,1,2,2-Tetrachloroethane								10			
Tetrachloroethylene								10			
Toluene								10			
1,1,1-Trichloroethane								10			
Trichloroethylene								10			
1,1,2-Trichloro-1,2,2-trifluoroethane								10			
1,2,4-Trimethylbenzene[a]								10			

Attachment 3 – CCP Headspace Gas UCL_{90} Evaluation Form (Continued) (EXAMPLE)

Page ____ of ____

WSPF Number: _____ Lot Number: _____

ANALYTE	Transform Data Used (No, Data-Log, SQTI, other)	# Samples	# Samples above MDL	Maximum (ppmv)	Mean (ppmv)	Standard Deviation (ppmv)	UCL_{90} (ppmv)	PRQL (ppmv)	Transformed PRQL (N/A or Value)	UCL_{90} > PRQL Yes	EPA Hazardous Waste Number
1,3,5-Trimethylbenzene[a]								10			
p/m-Xylene[b]								10			
o-Xylene								10			
Acetone								100			
Butanol								100			
Methanol								100			
Methyl ethyl ketone								100			
Methyl isobutyl ketone								100			

[a]These compounds are from CCP-PO-003, CCP Transuranic Authorized Methods for Payload Control (CCP CH-TRAMPAC) and are flammable VOCs that do not appear in CCP-PO-001. These are not part of the target analyte list, but samples may be analyzed for these compounds.
[b]These xylene isomers cannot be resolved by the analytical methods employed in the CCP. M-xylene and p-xylene will be reported as Total m-p-Xylene.

Comments:

_____ _____ _____
Printed name Signature Date

Attachment 4 – CCP Solids Analysis VOC UCL_{90} Evaluation Form (EXAMPLE)

Page ____ of ____

WSPF #_____ Lot Number: _____

ANALYTE	Transform Data Used (No. Data - Log, SQRT, other)	# Samples	# Samples above MDL	Maximum (mg/kg)	Mean (mg/kg)	SD (mg/kg)	UCL_{90} (mg/kg)	PRQL (mg/kg)	Transformed PRQL (N/A or Value)	UCL_{90} > PRQL Yes	EPA Hazardous Waste Number
Benzene								10			
Bromoform								10			
Carbon disulfide								10			
Carbon tetrachloride								10			
Chlorobenzene								10			
Chloroform								10			
1,4-Dichlorobenzene [c]								10			
Ortho-Dichlorobenzene [c]								10			
1,2-Dichloroethane								10			
1,1-Dichloroethylene								10			
Trans-1,2-Dichloroethylene								10			
Ethyl benzene								10			
Methylene chloride								10			
1,1,2,2-Tetrachloroethane								10			
Tetrachloroethylene								10			
Toulene								10			
1,1,1,-Trichloroethane								10			
1,1,2,-Trichloroethane								10			
Trichoroethylene								10			
Trichlorofluoromethane								10			
1,1,2-Trichloro-1,2,2-trifluoroethane								10			
Vinyl chloride								4			
p/m-Xylene								10			

Attachment 4 – CCP Solids Analysis VOC UCL_{90} Evaluation Form (Continued) (EXAMPLE)

Page ____ of ____

WSPF #_____ Lot Number: _____

ANALYTE	Transform Data Used (No. Data - Log, SQRT, other)	# Samples	# Samples above MDL	Maximum (mg/kg)	Mean (mg/kg)	Standard Deviation (mg/kg)	UCL_{90} (mg/kg)	PRQL (mg/kg)	Transformed PRQL (N/A or Value)	UCL_{90} > PRQL Yes	EPA Hazardous Waste Number
o-Xylene								10			
Acetone								100			
Butanol								100			
Ethyl ether								100			
Formaldehyde [(a)]								100			
Hydrazine [(b)]								100			
Isobutanol								100			
Methanol								100			
Methyl ethyl ketone								100			
Pyridine[(c)]								100			

(a) Required only for homogenous solids and soil/gravel waste from Savannah River Site.
(b) Required only for homogenous solids and soil/gravel waste from Oak Ridge National Laboratory and Savannah River Site.
(c) Can also be analyzed as an SVOC. If analyzed as an SVOC, the QAOs of CCP-PO-001, Table C3-6 apply.

Comments:

Printed name Signature Date

Attachment 5 – CCP Solids Analysis SVOC UCL_{90} Evaluation Form (EXAMPLE)

Page ____ of ____

WSPF #_____ Lot Number: _____

ANALYTE	Transform Data Used (No. Data Log, SQRT, other)	# Samples	# Samples above MDL	Maximum (mg/kg)	Mean (mg/kg)	Standard Deviation (mg/kg)	UCL_{90} (mg/kg)	PRQL (mg/kg)	Transformed PRQL (N/A or Value)	UCL_9 > PRQL Yes	EPA Hazardous Waste Number
2-Methylphenol (cresols)								40			
3&4-Methylphenol (cresols)								40			
1,4-Dichlorobenzene [(a)]								40			
Ortho-Dichlorobenzene [(a)]								40			
2,4-Dinitrophenol								40			
2,4-Dinitrotoluene								2.6			
Hexachlorobenzene								2.6			
Hexachloroethane								40			
Nitrobenzene								40			
Pentachlorophenol								40			
Pyridine[(a)]								40			

(a) Can also be analyzed as a VOC.

COMMENTS:

_____ _____ _____

Printed name Signature Date

Attachment 6 – CCP Solids Analysis Metals UCL_{90} Evaluation Form (EXAMPLE)

Page ____ of ____

WSPF #_____ Lot Number: _____

ANALYTE	Transform Data Used (No. Data Log, SQRT, other)	# Samples	# Samples above MDL	Maximum (mg/kg)	Mean (mg/kg)	Standard Deviation (mg/kg)	UCL_{90} (mg/kg)	PRQL (mg/kg)	Transformed PRQL (N/A or Value)	UCL_{90} > PRQL Yes	EPA Hazardous Waste Number
Antimony								100			
Arsenic								100			
Barium								2000			
Berylium								100			
Cadmium								20			
Chromium								100			
Lead								100			
Mercury								4			
Nickel								100			
Selenium								20			
Silver								100			
Thallium								100			
Vanadium								100			
Zinc								100			

Comments:

_____ _____ _____

Printed name Signature Date

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Attachment 7 – CCP Data Evaluation Narrative (EXAMPLE)

Page ____ of ____

WSPF Number: _____Lot Number: _____

Approved By:

_____ _____ _____
Signature Printed Name Date

Attachment 8 – CCP Waste Stream Headspace Gas Tentatively Identified Compounds (EXAMPLE)

Page ____ of ____

Tentatively Identified Compound (TICs)	Chemical Abstract Service (CAS) Number	Number of Samples Containing TIC

Approved By:

Signature Printed Name Date

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Attachment 9 – CCP Waste Stream Solids Tentatively Identified Compounds (EXAMPLE)

Page ____ of ____

Tentatively Identified Compound (TICs)	Chemical Abstract Service (CAS) Number	Number of Samples Containing TIC

Approved By:

Signature Printed Name Date

Attachment 10 – CCP Waste Stream Headspace Gas Tentatively Identified Compounds (Cumulative) (EXAMPLE)

Page ____ of ____

WSPF Number:_____Lot Number(s):_____

Total number of samples evaluated to date in this waste stream: _____

The following TICs were detected during Headspace Gas analysis of this waste stream:

Tentatively Identified Compound	CAS Number	Maximum Observed Estimated Concentrations (ppmv)	Total Number of Samples Containing TIC	% TIC Detected in Waste Stream	Greater than 25% Yes

Approved by:

_____ _____ _____

Signature Printed Name Date

Attachment 11 – CCP Waste Stream Solids Tentatively Identified Compounds (Cumulative) (EXAMPLE)

Page ____ of ____

WSPF Number:_____Lot Number(s):_____

Total number of samples evaluated to date in this waste stream: _____

The following TICs were detected during Headspace Gas analysis of this waste stream:

Tentatively Identified Compound	CAS Number	Maximum Observed Estimated Concentrations (ppmv)	Total Number of Samples Containing TIC	% TIC Detected in Waste Stream	Greater than 25% Yes

Approved by:

_____ _____ _____

Signature Printed Name Date

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Attachment 12 – CCP Site Project Manager Data Evaluation Narrative (EXAMPLE)

Page ____ of ____

WSPF Number: _____ Lot Number: _____

Approved By:

_____	_____	_____
Signature	Printed Name	Date