Data Validation Summary Report for Asbestos Data Associated with the Data Validation Summary Report, February to August 2011, Soil Remediation Completion Sampling, Nevada Environmental Response Trust (NERT)

Prepared for:

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List of Acronyms and Abbreviations

BMI	Basic Management, Inc.
CoC	chain of custody
DVSR	data validation summary report
ED	electron diffraction
EDD	electronic data deliverable
EDXA	energy dispersive X-ray analysis
EPA	Environmental Protection Agency (U.S.)
NAM	non-asbestos mineral
NDEP	Nevada Division of Environmental Protection
NERT	Nevada Environmental Response Trust
PARCCS	precision, accuracy, representativeness, completeness, comparability and sensitivity
%C	percent completeness
QA	quality assurance
QC	quality control
RPD	relative percent difference
S	analytical sensitivity
SOP	standard operating procedure
TEM	transmission electron microscope

1. Introduction

This data validation summary report (DVSR) has been prepared by Neptune and Company, Inc. to assess the validity and usability of asbestos data reported by EMSL Analytical, Inc. for the NERT Data Validation Summary Report February to August 2011 Soil Remediation Completion Sampling (NERT, 2013). This asbestos-only DVSR also includes an electronic data deliverable (EDD) that contains all analytical results and any qualifiers applied during this validation.

The laboratory reports from EMSL contained results for soil samples analyzed for asbestos via transmission electron microscopy (TEM). The asbestos soil samples were collected from the former Tronox Site, which is now known as the Nevada Environmental Response Trust Site, located within the Basic Management Inc., Complex. To determine the amount of releasable asbestos via dust generation, the sampling and analysis U.S. Environmental Protection Agency (EPA) Method 540-R-97-028 (with Berman and Kolk Modifications, 2000) was utilized. Asbestos data for soil samples SSAO5-09-0.0_01_BPC_-A and SSAO5-09-0.0_01_BPC_FD-A were validated for this DVSR.

The laboratory reports included a summary report and a bench data sheet for each asbestos sample. This summary information included elutriator data, TEM specifications, structure counts, classification and sketches. Additionally, chain of custody (CoC) documents, TEM images, electron diffraction (ED) and energy dispersive X-ray analysis (EDXA) results were included with the laboratory reports. A field duplicate was analyzed for precision and included with the reports. EMSL maintains data for blank samples and TEM calibration, which are typically not included in the lab reports due to the nature of elutriator sampling (i.e., it could take months before enough samples are analyzed to warrant a blank, such as filter lot, method, etc.). However, blank analysis and TEM calibration data were provided with this sample delivery group.

The laboratory reports were validated following 2012 Nevada Division of Environmental Protection (NDEP) guidance for validating asbestos data in soils (NDEP, 2012). Acceptance criteria for the QC samples were based upon the associated analytical method (540-R-97-028) and the modified elutriator method (Berman and Kolk, 2000). In cases where the analytical method did not fully describe the quality assurance (QA) criteria or corrective action, the U.S. Department of Defense's *Quality Systems Manual for Environmental Laboratories*, Version 4.2, was used as a guide for acceptance criteria. Professional judgment was also used in some cases to qualify the results.

This DVSR summarizes the QA evaluation of the data according to precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). This DVSR provides an assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability. Data qualifiers and their definitions are provided below.

Qualifiers	
J-	Estimated: The associated numerical value is an estimated quantity with a potentially negative bias. The analyte was detected, but the reported value may not be accurate or precise. The "J-" qualification indicates the data fell outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.
J+	Estimated: The associated numerical value is an estimated quantity with a potentially positive bias. The analyte was detected, but the reported value may not be accurate or precise. The "J+" qualification indicates the data fell outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.
J	Estimated: The associated numerical value is an estimated quantity. It is not possible to assess the direction of the potential bias. The analyte was detected, but the reported value may not be accurate or precise. The "J" qualification indicates the data fell outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.
UJ	Estimated/nondetected: Analyses were performed for the compound or analyte, but it was not detected. This qualification is used to flag possible false negative results in the case where low bias is indicated by a detect in the field duplicate.
R	Rejected: The datum is unusable (the compound or analyte may or may not be present). Use of the "R" qualifier indicates a significant variance from functional guideline acceptance criteria.

Qualifier Reason Codes	
b	The analyte was detected in the associated laboratory blank
fd	Field duplicates did not meet the project control limits

1.1. PARCCS Criteria

Precision is a measure of the agreement or reproducibility of analytical results under a given set of conditions. It is a quantity that cannot be measured directly but is calculated from structure counts. Precision is expressed as the relative percent difference (RPD):

$$RPD = \text{Absolute Value of } (D1-D2)*2/(D1+D2) * 100$$

Where D1 and D2 are the reported structure counts for sample and duplicate analyses.

An RPD outside the 50% QC limit (Berman and Kolk, 2000) between the field duplicates indicates imprecision but does not imply accuracy or allow for directional qualification (e.g., J+ or J-). Note that due to the inherent heterogeneity of soil samples and site locations, a high occurrence of RPD exceedances may be observed. However, it is important for field duplicates to be evaluated to assess site (and possibly sampling) variability. For this report, only duplicate field samples were provided (i.e., no laboratory duplicates were analyzed).

Accuracy is a measure of the agreement of an experimental determination and the true value of the parameter being measured. Due to the nature of asbestos analysis, accuracy cannot easily be assessed. There are no set standards or reference materials that can mimic the type of samples collected at naturally occurring sites. The key component for accuracy is the analyst, who is well trained for the identification and analysis of asbestos structures, including proficiency tests generated by accrediting agencies (e.g., NVLAP). The analyst uses tools such as morphology, ED and EDXA to accurately identify the asbestos structure and visually determines its size using scale bars. Note that the equipment, such as the TEM, ED and EDXA, have manufacturer requirements for maintaining its proper use and calibration; these records are typically maintained by the laboratory and must be requested with data packages. For this DVSR, the instrument calibration (e.g., camera, magnification, K-factors, detector resolution, resolvable Mg-Si and Na peaks, spot size measurement) will not be discussed in detail, but EMSL records show all TEM calibrations passed for the period of sample analysis.

Representativeness is a qualitative parameter that expresses the degree to which the sample data are characteristic of a population. It is evaluated herein by reviewing the QC results of blanks, samples and holding times. Positive detects of compounds in the blank samples identify compounds that may have been introduced into the samples during sample collection, transport, preparation, or analysis. The QC blanks collected and analyzed can include filter lot, field, lab, method, equipment and conditioning filter blanks. The client (i.e., who requests asbestos analysis) determines if field blanks are collected/analyzed, whereas the other blanks are dependent on batch sizes and if contamination is found (e.g., conditioning filters). Holding times and preservation are not established in the EPA Method (540-R-97-028) or Berman and Kolk (2000) modifications; however, the EMSL Elutriator SOP (rev. 2.1, June 2010) recommends samples be shipped on ice and stored at ice temperature if samples are not immediately analyzed. This is to avoid bacterial growth within the samples.

Comparability is a qualitative expression of the confidence with which one data set may be compared with another. In the data validation context, it provides an assessment of the equivalence of the analytical results to data obtained from other analyses. Comparability is also dependent upon other PARCCS criteria because only when precision, accuracy, and representativeness are known can data sets be compared with confidence. Note that the comparability of asbestos is somewhat limited because the accuracy of analysis cannot be easily assessed.

Completeness is defined as the percentage of acceptable sample results compared with the total number of sample results. Completeness equals the total number of sample results for each fraction minus the total number of rejected sample results divided by the total number of sample results multiplied by 100. Percent completeness (%C) is calculated using the following equation:

$$\%C = (T - R)/T \times 100,$$

where:

T = total number of sample results and

R = total number of rejected sample results.

Sensitivity relates to the ability of an analysis method to identify positive results. Specifically for asbestos analysis, sensitivity is measured using a construct called "analytical sensitivity". This is the calculated concentration of airborne asbestos structures, equivalent to counting one asbestos structure in the analysis (Chatfield, 1995). Analytical sensitivity (S) is a function of the volume of air sampled, the active area of the collection filter and the area of TEM grid that structures are counted (as defined below by Chatfield, 1995):

$S = A_f / (k*A_g*V)$

where A_f is the active area of the sample collection filter, A_g is the mean area of grid openings examined, k is the number of grid openings examined and V is the volume of air sampled.

The purpose of the analytical sensitivity is to try to encompass the range of asbestos concentrations that are of concern for asbestos related risk assessment. Berman and Kolk (2000) suggest that an analytical sensitivity of 3×10^6 S/g_{PM10} will encompass most of these concentrations and is adequate for most studies where protocol amphibole structures are suspected. However, they also suggest that a sensitivity of 5×10^7 S/g_{PM10} may be sufficient in cases where only chrysotile structures are suspected due to their lower potency compared to amphibole structures.

1.2. Basis for Qualifying Data

Field Duplicates: Duplicate samples were qualified with a J if the RPD between the sample and its duplicate was above 50%.

Blanks: Per EMSL Elutriator SOP (rev. 2.1, June 2010), the following blanks are analyzed:

- Filter lot blanks: 2 per lot of 50 filters, analyzed prior to sampling; lot rejected if background contamination is > 0.2 fiber/mm^2;
- Field blanks: determined by client;
- Lab blanks: 1) filter to evaluate elutriator prep room air, 2) filter near elutriator sampling ports (always collected, only analyzed if there is a question of contamination); blanks considered contaminated if >10 structures/mm^2;
- Method blank: analyzed 1 every 20 samples (this can take months before 20 samples are analyzed due to the time-intensive elutriator method), washed play sand used to assess tumbler and elutriator, should be 0.2 structures/mm^2;
- Equipment blanks: similar to the method blank, except sand is not used (only air); interchangeable with method blank;

- Conditioning filters: collected at the beginning of every run; not required unless there is case of contamination since these filters can help with troubleshooting.

2. Asbestos via EPA Method 540-R-97-028 with Berman and Kolk (2000) Modifications (soils)

Analytical sensitivities for all samples were around 3 x 10^6 S/g_{PM10}. No qualifications were made based on QC issues, as described below.

2.1. Quality Control Results

2.1.1. Blank Samples

The following blank information was obtained from EMSL or confirmed via phone with Robyn Denton, EMSL Special Projects Manager:

- Filter lot blanks: running average through 12/4/2008 shows no detects;
- Field blanks: no field blank was included with this data set;
- Lab blanks: no issues have been reported;
- Method blank: below the 0.2 structures/mm^2 criterion at a cumulative average of $\leq$ 0.09 structures/mm^2, Aug. 2009 to 2012;
- Conditioning filters: only used for troubleshooting.

No blank issues were reported; therefore no data was qualified due to blank issues.

2.1.2. Duplicate Sample Results

Field duplicate results for samples were within the control limits of 50 RPD (established by Berman and Kolk, 2000) or not applicable due to non-detects (i.e., not protocol structure counts); no qualifications made.

2.2. Unaddressed Issues

This section details issues that need to be addressed in the laboratory reports.

The customer sample number on the lab report for EMSL sample number 041103180-0002 needs to indicate this sample is a field duplicate. All other records for this sample indicate it should be labeled as SSAO5-09-0.0_01_BPC_FD-A.

2.3. Summary

No samples were qualified due to QC issues; however there are some unaddressed issues discussed in Section 2.2. Beyond the unaddressed issues, the data are considered acceptable and no data are rejected. The EDD details the verified (or corrected) structure counts and qualifiers (if applicable); this should be used to report asbestos results for these samples.

3. PARCCS

Precision: Assessments were discussed above and the precision the data are considered acceptable with the included data qualifiers (where applied).

Accuracy: As discussed above, accuracy is not easily assessed; however, EMSL records indicate the data should be accurate within their limitations.

Representativeness: No significant blank contamination has been found in laboratory samples and the representativeness of the project data is considered acceptable.

Comparability: The laboratory used standard analytical methods for the analyses. No information was provided that would conflict with the comparability of the results; therefore, the overall comparability is considered acceptable.

Completeness: No results were rejected based on this data validation. The completeness level attained for the samples was 100%.

Sensitivity: The analytical sensitivity for all samples was around 3×10^6 S/g_{PM10}, which is acceptable for risk assessment.

References

Berman DW and Kolk A, (2000). *Draft Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Material, Revision 1*. Submitted to the U.S. Environmental Protection Agency, Region 8, May 23, 2000, 84 pp.

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