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STATE OF NEVADA
Department of Conservation & Natural Resources
DIVISION OF ENVIRONMENTAL PROTECTION

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Re: **BMI Plant Sites and Common Areas Projects, Henderson, Nevada**
Guidance on Data Validation for Asbestos Data in Soils

Dear Messrs.:

Enclosed in this letter is NDEP's *Data Validation Guidance for Asbestos Data in Soils for the Basic Management Incorporated (BMI) Complex and Common Areas* dated June 2012. Please note that this guidance is also posted on NDEP's website at http://ndep.nv.gov/bmi/technical.htm under "Data Validation".

Please contact the undersigned with any questions at sharbour@ndep.nv.gov or 775-687-9332.

Sincerely,

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Data Validation Guidance for Asbestos Data in Soils for the Basic Management Incorporated (BMI) Complex and Common Areas

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Table of Contents

List of Acronyms

BMI - Basic Management, Incorporated
BRC - Basic Remediation Company
COC - Chain of Custody
DVSR - Data Validation Summary Report
EDXA - Energy Dispersive X-ray Analysis
ED - Electron Diffraction
IST - Isokinetic Sampling Tube
ME - Main Exit
MI - Midget Impinger
NDEP - Nevada Division of Environmental Protection
PCM - Phase Contrast Microscopy
SEM - Scanning Electron Microscopy
TEM - Transmission Electron Microscopy
USEPA - United State Environmental Protection Agency

1.0 Overview

The purpose of this document is to provide guidance for validating asbestos concentration data to ensure data integrity and evaluate data usability. This guidance is an expansion of the recommendations made in Appendix A of the NDEP (2011) technical guidance for asbestos related risk assessment. This asbestos data validation guidance has been developed in response to counting errors that have previously been found in reported asbestos data provided by the Companies that operate the BMI Complex and Common Areas. If the total number of asbestos structures reported by the Companies is less than the number found in laboratory reports, this is considered a fatal flaw according to BMI Complex and Common Areas Technical Review Guidance (NDEP, 2012). Additionally, the individual final reports for each asbestos sample have been found to include errors in the number of primary structure counts recorded, with respect to total structure counts. Consequently, this guidance document provides a step-by-step procedure that must be used by the Companies to verify the accurate reporting of asbestos laboratory results.

2.0 Introduction

Asbestos is the term used to describe a group of naturally occurring hydrated metal silicate minerals of fibrous habit (Berman and Crump, 2003), some of which have been found to cause serious health issues. Inhalation of asbestos fibers is associated with serious illnesses, such as lung cancer, mesothelioma and asbestosis. Consequently, potential exposure to the existing large quantities of asbestos products in public buildings and the natural presence of asbestos in large communities is of major concern to the scientific/medical community and the public (Berman and Crump, 2008a). For assessing health-related risks, collection, analysis and reporting of asbestos samples must be executed with little or no error. Additionally, the reported asbestos data from those samples should be verified via data validation to ensure accuracy.

2.1 Asbestos Mineral Types

Asbestos is generally considered as a description of 6 minerals that can be categorized into two types: chrysotile and amphibole. Chrysotile, which is from the serpentine mineral (magnesium silicate), is the most common type of asbestos. The 5 remaining minerals are all amphiboles (ferro-magnesium silicates) and are classified as crocidolite (fibrous reibeckite), amosite (fibrous grunerite), anthophyllite, tremolite and actinolite (Berman and Crump, 2003). The use of asbestos in commercial applications became widespread in the 19th century with chrysotile making up over 90% of its use (Berman and Crump, 2003). The toxicity of asbestos is considered based on its physical and chemical properties including fiber size, shape, and mineral type. Amphibole fibers are considered by some to be more potent than chrysotile fibers; it has been estimated that chrysotile potency for both mesothelioma and lung cancer is 0.0013 and 0.27 times, respectively, that for amphibole (Berman and Crump, 2003). However, the possibility that chrysotile and amphibole are equal in potency has not been completely discarded (Berman and Crump, 2003).

2.2 Asbestos Potency

There is continued debate about which fiber dimensions are most potent and contribute to specific disease endpoints. Berman and Crump (2001) reported that fibers longer than 5 μm and thinner than 0.5 μm are biologically active and have the potential to cause asbestos-related diseases. However, recent studies by Berman and Crump (2008a and 2008b) suggest that fibers longer than 10 μm and thinner than 0.4 μm may have the highest potency with respect to lung cancer and mesothelioma. Berman and Crump also suggest that fiber potency may increase with increasing length up to 20 μm or even 40 μm. Despite the ongoing debate, the USEPA interim guidelines (Berman and Crump, 2003) consider fibers longer than 10 μm and thinner than 0.4 μm to be most likely to cause asbestos-related disease. These fiber dimensions are used for calculating asbestos-related risk for the BMI Complex and related sub-areas (NDEP, 2011). It should be noted that the NDEP (2011) risk assessment guidance differs in approach from the USEPA (2008) *Framework for Investigating Asbestos-Contaminated Superfund Sites*, guidance that the USEPA considers as replacing or superseding the Berman and Crump (2003) USEPA interim guidance. The differences between the two approaches, regarding aspects such as sampling, analysis, counting and risk assessment calculations, are discussed in Appendix C of the NDEP (2011) guidance.

3.0 Data Validation

The following subsections describe the necessary components for validation of asbestos data and provide background for understanding the asbestos data validation process. Below, in Appendix I of this document, is a summarized step-by-step process for performing asbestos data validation.

3.1 Sample Receipt/Handling and Chain of Custody

A Chain of Custody (COC) record must accompany the samples throughout the shipping/handling and analysis. The COC record must provide the sample ID, sample collection date and time, analysis request, personnel contact information, who relinquished the samples and who received them. Additionally, a section for comments/instructions for the sampler can be completed if there are any issues during sample collection or to provide more specific instructions for sample analysis.

3.2 Sample Preparation and Analysis

Preparation and analysis of asbestos found in soil samples is the focus of this guidance, which is specific to the BMI Complex and Common Areas. USEPA Method 540-R-97-028, the reference method for this guidance, is employed by the Companies for analyzing releasable asbestos in soils. This method prepares samples via dust generation and utilizes transmission electron microscopy (TEM) for sample analysis. Although there are other methods for analyzing asbestos samples, such as phase contrast microscopy (PCM), midget impinger (MI) and scanning electron microscopy (SEM), TEM is the focus of this guidance. TEM is the preferred technique because

of its analytical capabilities to determine all of the asbestos characteristics that are associated with risk factors, such as mineral type, fiber size and shape.

3.2.1 Sample Preparation via Elutriator Method

The *Draft Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Material* (Berman and Kolk, 2000) was adapted from EPA Method 540-R-97-028 and includes changes that reduce analytical costs and refine the overall method. This adaptation is used by laboratories (such as EMSL Analytical, Inc.) that routinely analyze asbestos soil samples.

The elutriator method employs isokinetic sampling that will collect only the asbestos structures released from soils that are respirable. For sample preparation records, an elutriator prep worksheet must be provided that includes details such as sample weight (before and after drying), total dried sample weight fractions, tumbling speed, start and stop times, flow rate at the main exit (ME) and isokinetic sampling tube (IST) openings and filter IDs with pre- and post-weights. This information is used for determining the concentration of asbestos per gram of respirable dust (S/g_{PM10}), which must be listed on the final report sheet. Additionally, the rate of release of respirable dust can be calculated using the mass measurements of dust collected over time on the (main exit) ME filters. The mass percent of the respirable dust in the bulk sample can also be calculated from the mass measurements. The details for calculating the concentration, rate of release and mass percent are discussed at length in Section 10 of the modified elutriator method (Berman and Kolk, 2000).

3.2.2 Sample Analysis

For sample analysis, via TEM, a Bench Sheet Data report should be available for each sample. This report will list the sample ID, details about the TEM settings and a list of grids and their respective grid openings. For each grid opening, there will be notation about whether a structure was detected and details about the structure (e.g., dimensions and mineral type). The Bench Sheet Data will be used to verify the correct counting of the detected structures (asbestos and non-asbestos minerals). If a structure is detected, a Structure Sketch Sheet should be included where the identified structures are drawn by hand, or electronically if possible, to represent the image seen in the TEM view screen. If the detected structure is classified as an asbestos mineral, energy dispersive X-ray analysis (EDXA) and electron diffraction (ED) spectra are included to verify the mineral type. In some cases, the Photomicrograph Report (TEM image) is also included with the identified asbestos structures. The specific details for using the aforementioned laboratory reports are discussed in more detail below.

3.3 Structure Counting Criteria

The criteria used for counting asbestos structures is specific and only those fibers/structures meeting the criteria are considered in health-related risk assessments. The counting rules for EPA Method 540-R-97-028 follow ISO 10312:1995(E) (Chatfield, 1995), which is discussed below.

The following sections describe distinguishing which structures are considered the most relevant (i.e., potent) for health-related risk assessment, and discuss those structures that are excluded.

3.3.1 Asbestos Structures

Although the use of the term "fiber" has been used to encompass asbestos structures, there are several different types of structures that exist. These structures are well defined in ISO 10312:1995(E) (Chatfield, 1995). The four main structures are fiber, bundle, cluster (disperse and compact) and matrix (disperse and compact). According to the ISO 10312:1995(E) counting rules (Chatfield, 1995), these structures are defined as follows:

1) *fiber*- any particle with parallel or stepped sides that is at least 0.5 μm in length and has an aspect ratio of 5:1 or greater (note that some laboratories may use the historic definition that is a 3:1 ratio for comparison to historical optical measurements, also known as PCM equivalent),
2) *bundle*- group of attached fibers that are parallel,
3) *cluster*- aggregate of two or more randomly orientated fibers, with or without bundles,
4) *matrix*- one or more fibers or bundles that may be attached or somewhat concealed by a nonfibrous particle.

Each one of these four categories exists as a separate entity that is designated as a primary structure. Matrix and cluster primary structures can contain several structures (e.g., fibers and bundles) within them. For example, on a TEM grid opening one might identify a matrix primary structure that is comprised of two asbestos fibers, which are attached to or overlapping a group of nonfibrous particles. Individually identified structures within a primary structure are each counted and yield a total structure count for the sample.

3.3.2 Protocol Asbestos Structures (>5 μm in length; < 0.4 μm in diameter)

According to Berman and Kolk (2000), biologically relevant asbestos structures are those that are longer than 5 μm and thinner than 0.5 μm; structures satisfying these constraints are considered to be "protocol asbestos structures". However, a more recent report by Berman and Crump (2003) indicates that the diameter discrimination of a structure should be < 0.4 μm for risk assessment. For asbestos related risk assessments performed using NDEP (2011) guidance, the final report for each sample should **only include structures with diameters < 0.4 μm** because the dose-response coefficients (as mentioned below) used by NDEP (2011) guidance are specific to this diameter range. In addition to distinguishing structures by diameter for risk assessment, asbestos structures are also discriminated by length due to potency factors, as discussed below. For the purposes of this guidance, "protocol asbestos structures" will encompass both short and long protocol asbestos structures that are < 0.4 μm in diameter, as defined below, but only "long protocol asbestos structures" will be used to calculate asbestos related risk according to NDEP (2011) guidance.

3.3.2.1 Short Protocol Asbestos Structures (>5 µm, < 10µm in length; < 0.4 µm in diameter)

Protocol asbestos structures that are >5 µm, but ≤ 10 µm in length with a < 0.4 µm diameter are considered "short protocol asbestos structures" for the purpose of this guidance. The short protocol asbestos structures are recorded on the final report for each asbestos sample and are labeled as "asbestos structures >5 µm, ≤ 10 µm". However, the short protocol asbestos structures are not used for asbestos related risk calculations and are distinguished separately from "long" (> 10 µm in length) protocol asbestos structures because the "long" structures are considered to be more potent (Berman and Crump, 2003).

3.3.2.2 Long Protocol Asbestos Structures (> 10µm in length; < 0.4 µm in diameter)

Protocol asbestos structures that are > 10 µm in length with a < 0.4 µm diameter are defined as "long protocol asbestos structures". These are recorded on the final report for each asbestos sample and are labeled as "asbestos structures > 10 µm (Long)". Only long protocol asbestos structures are used when calculating asbestos related risk according to NDEP (2011) guidance. Structures meeting these dimension constraints are considered to be most likely to cause asbestos related diseases (Berman and Crump, 2003).

3.3.3 Structures Excluded from Risk Assessment

The asbestos sample analytical report will include the total protocol asbestos structures, but only a portion of them will be used for the asbestos health-related risk assessment. Regulated asbestos minerals include chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite). For inclusion in the asbestos risk assessment, these regulated mineral structures must also be > 10 µm in length and < 0.4 µm in diameter, as suggested by Berman and Crump (2003) for optimized dose-response coefficients. There are other minerals found in soil samples during asbestos analysis that are excluded from the risk assessment and include: non-asbestos minerals (e.g., apatite and talc) and non-regulated amphiboles (e.g., winchite, richterite and fluoro-edenite).

3.4 Fiber Mineral Identification

Identification of asbestos fibers or structures is achieved by evaluating the structure morphology and analyzing the sample with energy dispersive X-ray analysis (EDXA) and electron diffraction (ED). Note that only a specific level of classification for fiber identification can be obtained because of the nature of a sample (e.g., ED cannot be performed on non-crystalline material) and instrumentation limitations (e.g., grid positioning must be optimal for EDXA to be performed). These classification levels are discussed in detail in Tables D.1 and D.2 and Figures D.2 and D.4 of ISO 10312:1995(E) (Chatfield, 1995). The methods used for identifying asbestos fibers are briefly discussed below.

3.4.1 Morphology

Fiber morphology is based on two types of classification: 1) tubular and 2) non-tubular morphology. Fibers that are identified as having tubular morphology are suspected to be chrysotile, whereas non-tubular fibers are suspected to be amphibole. Once a fiber is suspected to be chrysotile or amphibole based on tubular morphology, ED and EDXA can be utilized to further classify the structure and thus confirm if it is either chrysotile or ampibole.

3.4.2 Electron Diffraction (ED)

ED, which is commonly found on TEM instruments, is used to analyze the crystalline structure of a solid using electron diffraction (i.e., interference) patterns. Section D.4.1 of ISO 10312:1995(E) (Chatfield, 1995) describes the features of the electron diffraction pattern that are used to identify chrysotile structures. Additionally, Figure D.3 of this same section shows an image of the electron diffraction pattern for chrysotile. Confirmation of amphibole presence can only be obtained by quantitative interpretation of zone-axis ED patterns (Chatfield, 1995). Figure D.1 of ISO 10312:1995(E) (Chatfield, 1995) shows an example zone-axis ED pattern and Sections D.3.2 and D.4.2 further discuss identification of amphibole fibers with ED.

3.4.3 Energy Dispersive X-ray Analysis (EDXA)

EDXA, which is commonly found on TEM instruments, is utilized to determine the elemental composition of a sample. According to Section 3.11 of ISO 10312:1995(E) (Chatfield, 1995), the nominal elemental composition of chrysotile is $Mg_3(Si_2O_5)(OH)_4$, but the exact composition in natural chrysotile can deviate from this where Si may be substituted by Al or Mg may be substituted by Fe(II), Fe(III), Ni, Mn, or Co. Additionally, ISO 10312:1995(E) (Chatfield, 1995) defines the nominal elemental composition for amphiboles as $A_{0-1}B_2C_5T_8O_{22}(OH, F, Cl)_2$ where A = K, Na; B = Fe(II), Mn, Mg, Ca, Na; C = Al, Cr, Ti, Fe(II), Fe(III), Mg; T = Si, Al, Cr, Fe(III), Ti; and some of these elements can be substituted by Li, Pb or Zn.

EDXA can provide both qualitative and quantitative analysis. Sections D.2.3, D.4.1 and D.4.2 of ISO 10312:1995(E) (Chatfield, 1995) further discuss EXDA measurements of chrysotile and amphibole fibers. For quantitative EDXA of chrysotile, Section D.4.1 (Chatfield, 1995) indicates that there are only two elements (Si and Mg) that are important and those two should be the prominent peaks (with appropriate area ratio) with minimal peaks from the other elements. Due to the 5 types of regulated amphibole minerals and the variations that may exist in chemical composition, EDXA of amphibole fibers is not as straightforward. However, Sections D.2.3 and D.4.2 of ISO 10312:1995(E) (Chatfield, 1995) provide some guidance for EXDA measurements and reference spectra can be found in the literature (Hayashi *et al.*, 1978).

3.5 Verification of Quality Controls and Quality Assurance

Section 12 of USEPA Method 540-R-97-028, Section 9.7 of ISO 10312:1995(E) (Chatfield, 1995) and Section 11 of Berman and Kolk (2000) discuss the quality assurance and quality

control requirements for asbestos sampling and analysis. These requirements are briefly discussed below.

3.5.1 Blanks

Berman and Kolk (2000), in an adaption of USEPA Method 540-R-97-028, recommend that the following blanks be collected routinely while employing their method: filter lot blanks, laboratory blanks, field blanks, method blanks, equipment blanks, and conditioning filters. The details for generating these blanks are specified in Section 11.1 of Berman and Kolk (2000), and criteria listed there for those blanks is summarized as follows:

- Filter lot blanks: 2 filters tested from each lot of 50; contamination should not exceed 0.2 structures/mm^2; only filters that meet this criterion can be used for sample analysis;
- Laboratory blanks: frequency not listed; ensure that laboratory air is in compliance or analysis halts until the issue is addressed; criterion not specified but reference is made to Section 10.6 of Chatfield and Burman (1990), which also does not specify the criterion; NDEP recommends that contamination does not exceed 0.2 structures/mm^2 similar to filter lot blanks;
- Field blanks: QC criterion is to be project specific; Chatfield (1995) recommends at least one field blank is processed with each sample batch and NDEP recommends that contamination does not exceed 0.2 structures/mm^2 similar to filter lot blanks;
- Method blanks: one per 20 samples analyzed; contamination must not exceed 0.2 structures/mm^2;
- Equipment blanks: interchangeable with method blanks, specifically should be used when issues exist with washed sand; no criteria listed but one should default to those for method blanks since they are considered interchangeable with equipment blanks;
- Conditioning filters: collected at the start of each run; no criteria specified other than these blanks should be used for troubleshooting if issues arise.

The results for the above-mentioned blanks must be reported to NDEP with the applicable field sample results.

3.5.2 Duplicates and Replicates

For duplicates and replicates, Berman and Kolk (2000) advise that 5-10% of field samples should have a spatial duplicate and that 100% of the field samples should be duplicate pairs, where only 2-3% are randomly selected to be analyzed by the laboratory. Additionally, Berman and Kolk (2000) state that the acceptable relative percent difference (%RPD) between duplicates is < 50%. If the %RPD is greater than acceptable, then replicate counts should be performed on chosen samples by different analysts. If re-analysis is not possible, the results for the duplicate pair should be flagged to indicate the lack of precision and the potential to affect data usability. Note, soil samples are naturally heterogeneous, which could affect the reproducibility of duplicate results.

3.5.3 Inter-Laboratory Assessments

BRC SOP-12 (2010) states that soil samples will be analyzed for asbestos using procedures consistent with the modified elutriator method developed by Berman and Kolk (2000). Because asbestos counting can be subjective, Berman and Kolk (2000) recommend that at least two different laboratories analyze the asbestos samples. If this recommendation is followed, then this can be accomplished by exchanging blind field replicates between two or more laboratories to compare counting results. The percentage of samples to be verified by other laboratories is not specified in Berman and Kolk (2000), but given the concerns expressed in Berman and Kolk (2000), NDEP recommends 5-10% of the collected samples be re-analyzed by an independent laboratory when inter-laboratory assessments are included in the sampling plan. NDEP also recommends targeting a %RPD of no greater than 50% when inter-laboratory replicates are analyzed.

3.5.4 Analytical Sensitivity Requirements

Analytical sensitivity represents the amount of airborne asbestos structures per gram of respirable dust (S/g_{PM10}) or the amount of asbestos structures per liter of air (S/l). The calculation for analytical sensitivity is shown in Section 8 of the ISO 10312:1995(E) (Chatfield, 1995). 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. Based on the desired analytical sensitivity and experimental parameters (e.g., volume of air sampled, etc.), the number of grid openings required to be analyzed to achieve this sensitivity can be calculated using equation Section 8 of the ISO 10312:1995(E) (Chatfield, 1995), as mentioned above.

3.5.5 Limit of Detection

Chatfield (1995) defines the limit of detection as the upper limit for a Poisson distribution with a 95% confidence interval where there is a zero structure count. However, NDEP (2011) risk assessment guidance does not use this definition. Instead, a detect is defined as one or more counts of asbestos structures within a sample. A non-detect result is defined as zero structures observed or counted within a sample.

3.6 Commentary Write-Up For Asbestos Data Validation

Basic Remediation Company (BRC) has developed a standard operating procedure (SOP) for reviewers to follow (BRC, 2009) when reviewing and validating concentration data. This SOP is specific to traditional chemical analyses, such as organic and inorganic, and does not necessarily apply to asbestos-related data. The BRC SOP also explains the use of validation qualifiers. Presently, no data qualifiers have been employed for reported asbestos concentrations. Due to the

possibility of sample contamination, e.g., from the laboratory or field equipment, data validation qualifiers must used when appropriate. Data qualifiers are important in situations where there is blank contamination such as a laboratory or field blank that could affect the outcome of samples collected with the contaminated blank. Additionally, disagreement in results between duplicate samples could indicate issues within field and laboratory processes that could adversely affect data quality. Replicate and inter-lab results should also be assessed and if necessary qualifiers applied. At a minimum the validation report should discuss any non-conformance with respect to blanks, replicates, and inter-lab results and the possible affect on the data quality and usability. It is important to note that qualified data could still be used in subsequent calculations, such as a risk-assessment, but the qualifiers would clarify any possible influences that the data may have on decision-making.

References

Berman DW and Crump KS, (2001). *Technical Support Document for a Protocol to Assess Asbestos-Related Risk.* Prepared for Mark Raney, Volpe Center, U.S. Department of Transportation, 55 Broadway, Kendall Square, Cambridge, MA 02142. Under USEPA review.

Berman DW and Crump KS, (2003). *Final draft: Technical support document for a protocol to assess asbestos-related risk.* Prepared for Mark Follensbee, Syracuse Research Corporation, Syracuse, NY, and the Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, DC. USEPA #9345.4-06. Limited revision draft.

Berman DW and Crump KS, (2008a). Update of potency factors for asbestos-related lung cancer and mesothelioma. *Critical Reviews in Toxicology*, 38(Suppl 1):1-47.

Berman DW and Crump KS, (2008b). A meta-analysis of asbestos-related cancer risk that addresses fiber size and mineral type. *Critical Reviews in Toxicology*, 38(Suppl 1):49-73.

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.

Basic Remediation Company (BRC), (2010). *SOP-12 Surface Soil Sampling and Analysis for Asbestos.* Basic Remediation Standard Operating Procedures, BMI Common Areas, Clark County, Nevada, December 2010.

Basic Remediation Company (BRC), (2009). *SOP-40 Data Review/Validation.* Basic Remediation Standard Operating Procedures, BMI Common Areas, Clark County, Nevada, May 2009.

Chatfield EJ, (1995). *Ambient Air: Determination of Asbestos Fibers, Direct Transfer Transmission Electron Microscopy Procedure.* Submitted to the International Standards Organization: ISO 10312:1995(E).

Chatfield EJ and Berman DW, (1990). *Interim Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method.* USEPA publication: 540/2-90/005a, May 1990.

Hayashi H, Aita S and Suzuki M, (1978). Semiquantitative chemical analysis of asbestos fibers and clay minerals with an analytical electron microscope. *Clays and Clay Minerals, 26*(3), 181-188.

Nevada Division of Environmental Protection (NDEP), (2011). *Technical Guidance for the Calculation of Asbestos Related Risk in Soils for the Basic Management Incorporated (BMI) Complex and Common Areas.* Prepared For: Nevada Division of Environmental Protection Bureau of Corrective Actions Special Projects Branch 2030 East Flamingo Road, Suite 230 Las Vegas, NV 89119.

Nevada Division of Environmental Protection (NDEP), (2012). BMI Complex and Common Areas Projects, Henderson, Nevada: *Technical Review Comment Guidance Revision 1* (January 2012).

USEPA, (2008) *Framework for Investigating Asbestos-Contaminated Superfund Sites*, Office of Solid Waste and Emergency Response, Asbestos Committee of the Technical Review Workgroup, OSWER Directive 9200.0-68, September 2008.

U.S. Environmental Protection Agency (USEPA), Method 540-R-97-028. *Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials.* Solid Waste and Emergency Response: Interim Version.

Appendix I: Steps for Validating Reported Asbestos Data

1. **Document Retrieval:** Retrieve final laboratory report, raw laboratory data (bench sheet data, structure sketches, elutriator prep of samples, ED and EDXA files), COC information and the electronic data deliverable (EDD) for all asbestos samples. The laboratory reports should also include all QC samples, such as the blanks described in Section 3.5.1, duplicates and replicates described in Section 3.5.2, and inter-laboratory replicates, if any, described in Section 3.5.3. Note: an EDD may not be available in all cases. In those cases, there should be a summary table for the asbestos data within the written report.
2. **Verify COC:** Compare the samples reported with any Chain-of-Custody (COC) information and ensure that they are consistent, e.g., confirm sampling names, dates and locations match up. The COC must provide the sample ID, sample collection date and time, analysis request, personnel contact information, who relinquished the samples and who received them. Note any issues that may have been recorded on the COC paperwork.
3. **Verify Methods:** Verify that the method being used for sample preparation and analysis is documented on laboratory reports in a manner that can be easily traced to the official document from the USEPA or other applicable source. For asbestos analysis in soil samples, laboratories should be following the modified elutriator method (Berman and Kolk, 2000), which is an adaptation of the USEPA Method 540-R-97-028. Both of these methods are relevant, but the modified elutriator method updates the USEPA Superfund Method.
4. **Verify Sample List:** Verify that the sample names on the laboratory raw data match up with the written report and/or the EDD. Batch identifier information should also be reported with each sample.
5. **Verify Analytical Sensitivity:** Verify that the analytical sensitivity reported for each sample meets the Sampling and Analysis or Work Plan specifications. Analytical sensitivity units should be consistent with the method, (e.g. S/g_{PM10}).
6. **Sample Preparation Sheets:** If any field or lab preparation technique was performed this must be reported. Ensure any mechanical steps used in laboratory sample preparation are included in the reports such as drying and splitting. Documentation of sample preparation must be provided in an elutriator prep worksheet that includes details such as sample weight (before and after drying), total dried sample weight fractions, tumbling speed, start and stop times, flow rate at the ME and IST openings and filter IDs with pre and post weights. From this data, the laboratory can calculate the concentration of asbestos per gram of respirable dust (S/g_{PM10}), which is listed on the final report sheet as "Conc." The mass percent or the amount of respirable dust in the bulk sample can also be calculated from the mass measurements. The details for calculating the concentration, rate of release and mass percent are discussed at length in Section 10 of the modified elutriator method (Berman and Kolk, 2000). Examples of typical mass curves, which are included with the elutriator prep worksheet, can be found in Section 11.2 of USEPA

Method 540-R-97-028 and can be used for comparison to the mass curves shown for each sample.

7. **Sample Analysis Sheets:** The Bench Sheet Data report, which details TEM results, must be available for each sample. This report must list the sample ID, details about the TEM settings and a list of grids and their respective grid openings. For each grid opening, there can be notation about whether a structure was detected and details about the structure (e.g., dimensions and mineral type). The Bench Sheet Data will be used for subsequent steps to verify the correct counting of the detected structures. If a structure is detected, a Structure Sketch Sheet must be included where the identified structures are drawn by hand to represent what is seen in the TEM view screen. If the detected structure is classified as an asbestos mineral, energy dispersive X-ray analysis (EDXA) and electron diffraction (ED) spectra must be included to verify the mineral type. And in some cases, the Photomicrograph Report (TEM image) will also be included with the identified asbestos structures.
8. **Know the Code:** These steps cannot provide all the details that are needed for properly identifying asbestos data on Bench Sheet Data reports. One should become acquainted with the types of primary structures discussed in Section 3.3.1 of this guidance and the codes or abbreviations used to identify them. More complete details, including examples of primary structures, can be found in Annex C of ISO 10312:1995(E) (Chatfield, 1995). For convenience, some of the "structure type" codes are:
    - Primary Structures: F = fiber, B = bundle, MD = matrix diffuse, MC = matrix compact, CD = cluster diffuse, CC = compact cluster;
    - Total Structures within Primary Structures: MF = matrix fiber, MB = matrix bundle, MR = matrix residual, CF = cluster fiber, CB = cluster bundle, CR = cluster residual.

    The primary structure codes MD, MC, CD and CC will be followed by a two-digit number. The first digit is the estimated total number of fibers and bundles in the structure and can range from 1 to 9, or 'I+" if there are more 9 fibers or bundles. The second digit is the total number of fibers and bundles longer than 5 □m within the structure.
9. **Count the Number of Protocol Asbestos Structures:** Find the Bench Sheet Data report (lists fiber types, dimensions and grid openings; EMSL ones are typically in a table format with alternating row colors of blue and white) for all of the samples and focus on them one at time. Looking at the Bench Sheet Data report, find the column listed as "Total" under "Structure Number". This column will sequentially number the total structures found in the sample. Note that this sheet will assign a number to all minerals found, even those that do not qualify as protocol asbestos structures (e.g., NAM or non-asbestos mineral). Verify that the codes (see Step 8 above) used for describing the structures (e.g., MD11) are consistent with the hand-drawn structures on the Structure Sketch Sheet. Next, identify the column "Mineral Type" and only look for chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite) structures. Then,

count all of the chrysotile and amphibole (total structures) that are >5 □m in length and < 0.4 □m in diameter; this will give the total protocol asbestos structures. Now separate the total count into chrysotile and amphibole structures since they are reported separately. The last step for this count is to count the number of primary structures in which the total structures were found. The primary structure numbers are listed under the column "Structure Type" – "Primary". For every total structure there should be one primary structure, but each primary structure can have several structures within it. Note that only primary structures > 5 μm in length and < 0.4 μm in width will be considered "countable" primary structures that will appear in the final report. Verify the determined counts with those recorded in the final and written reports.

10. **Count the Number of Short Protocol Asbestos Structures:** This will separate out the number of protocol structures that are "short" and not included in the risk assessment. Similar to step 8, look at the Bench Sheet Data report and find the column listed as "Total" under "Structure Number". Now count the chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite) total structures that are >5 □m, but ≤ 10□m in length and < 0.4 □m in diameter. This count will give the total number of short protocol asbestos structures. Now separate the total count into chrysotile and amphibole structures since they are reported separately. The last step is to count the number of primary structures in which the total structures were found. The primary structure numbers are listed under the column "Structure Type" – "Primary". For every total structure there should be one primary structure, but each primary structure can have several structures within it. Note that only primary structures > 5 μm in length and < 0.4 μm in width will be considered "countable" primary structures that will appear in the final report. Verify the determined counts with those recorded in the final and written reports.
11. **Count the Number of Long Protocol Asbestos Structures:** This will distinguish those structures that will be included in the risk assessment calculations. Similar to steps 8 and 9, look at the Bench Sheet Data report and find the column listed as "Total" under "Structure Number". Now count the chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite) total structures that are > 10□m in length and < 0.4 □m in diameter. This count will give the total number of short protocol asbestos structures. Now separate the total count into chrysotile and amphibole structures since they are reported separately. The last step for this count is to count the number of primary structures that the total structures were found in. The primary structure numbers are listed under the column "Structure Type" – "Primary". For every total structure there should be one primary structure, but each primary structure can have several structures within it. Note that only primary structures > 5 μm in length and < 0.4 μm in width will be considered "countable" primary structures and will appear on the final report. Verify the determined counts with those recorded in the final and written reports.

12. **Count the Number of Protocol Non-Asbestos Structures:** This step will count the structures that fall within the dimensions of a protocol asbestos structures, but are not classified as chrysotile or amphibole minerals. Similar to previous steps, look at the Bench Sheet Data report and find the column listed as "Total" under "Structure Number" and count the total non-asbestos structures (NAM or non-asbestos mineral) that are >5 □m length and < 0.4 □m in diameter. This count will give the total number of protocol non-asbestos structures. Similar to before, count the number of primary structures and verify that the NAM total and primary structure counts are reported correctly in the final and written reports.
13. **Verify Fiber Identification:** The laboratory should provide the data used for fiber identification, such as ED, EDXA and morphology from TEM images. However, all of these data are not always available for each fiber identification. Additionally, unless the reviewer has been sufficiently trained in interpreting these data, it will be difficult for the reviewer to verify the fiber identification. It is recommended that the reviewer refer to Sections 3.4.1 through 3.4.3 of this guidance for assistance in verifying fiber identification. If the reviewer suspects there might be an issue with how a fiber was identified, they should discuss this with the project manager for clarification.
14. **Verify Quality Controls:** Ensure that the proper blanks and field duplicates have been performed and meet the criteria specified in the method, which are summarized in Sections 3.5.1 and 3.5.2 of this guidance. Also, verify that 5-10% of the total samples have been sent to other, independent laboratories for count verifications and the data is reported. If the criteria for blanks, duplicates and inter-laboratory assessments are not met, this should be identified in the DVSR. At a minimum the validation report should discuss any non-conformance with respect to blanks, replicates, and inter-lab results and the possible affect on the data quality and usability.
15. **Examine the Final Laboratory Report Sheets:** The final laboratory report sheets typically have the name of the laboratory identifying the analysis and have summarized nearly all of the details included in the raw laboratory data. Looking at the final report for each sample, verify that the determined counts match those in the final report. Verify that the following is included on the final laboratory report: sample name, levels of analysis, magnification for fiber counting, aspect ratio used for fiber definition, mass of respirable dust on filter, area of the sample filter, number of grid openings analyzed, area of grid openings, dimensions used for counting, analyst name, dried sample weights, soil moisture, air flow rate through ME and IST openings, total elutriator flow rate, structure class, counts (primary and total), density, concentration, lower and upper detection limits, non-asbestos structures (primary and total) and a list of asbestiform amphibole present (ones that did not meet the dimension requirements or were non-regulated amphiboles).
16. **Comment Write-Up:** Summarize and formally write-up any issues that were found using the guidelines referenced in Section 3.6 of this document.