Office of Superfund Remediation and Technology Innovation (OSRTI)
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USEPA

PLM VALIDATION PROCESS GUIDELINES

For Asbestos Data Review

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NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other Governmental employees. They do not constitute rule-making by the USEPA, and may not be relied on to create a substantive or procedural right enforceable by any other person. The Government may take action that is at a variance with the policies and procedures in this manual.

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ACRONYMS

COC	Chain-of-Custody	RI	Refractive Index
DQO	Data Quality Objective	RPD	Relative Percent Difference
NADES	National Asbestos Data Entry Spreadsheet	SAP	Sampling and Analysis Plan
NIST	National Institute of Standards and Technology	SOP	Standard Operating Procedure
PE	Performance Evaluation	SOW	Statement of Work
PLM	Polarized Light Microscopy	SRM	Standard Reference Material
QAPP	Quality Assurance Project Plan	TR	Traffic Report
QC	Quality Control	USEPA	United States Environmental Protection Agency

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the usability of analytical data generated through the Statement of Work (SOW) and/or methods applicable to asbestos sample analysis by Polarized Light Microscopy (PLM), hereinafter referred to as the PLM SOW. The guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process. It should not be used to establish specific contract compliance. Definitive guidance is provided where performance should be fully under a laboratory's control (e.g., blanks, calibration standards, instrument performance checks), while general guidance is provided for evaluating subjective data that is affected by the site conditions.

The guidelines presented in the document will aid the data reviewer in establishing: (a) if data meets the specific technical and quality control (QC) criteria established in the PLM SOW; and (b) the usability of any data not meeting the specific technical and QC criteria established in the PLM SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to, site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for resampling. To make judgments at this level requires the reviewer to have a complete understanding of the intended use of the data. The reviewer is strongly encouraged to establish a dialogue with the user to discuss usability issues and to answer questions regarding the review, prior to, and after data review. It should also be understood that in all cases, data which do not meet specified criteria are never to be fully acceptable without qualification.

The data reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as professional judgment, should also be used to determine the ultimate usability of data, especially in those cases where all data does not meet specific technical criteria. While data verification and validation are instrumental to evaluating the accuracy (i.e. absence of transcription errors) and quality of the reported data, they are only one component of data review. The reviewer should also be aware that minor modifications to the analytical methods may be made to meet site-specific requirements, and that these modifications could affect certain validation criteria. A full copy of a request for modified analysis made to the analytical method should be included in the data package by the laboratory.

DATA QUALIFIER AND REASON CODE DEFINITIONS

The following definitions provide a brief explanation of the data qualifiers and reason codes assigned to results in the data review process. If the data reviewer chooses to use additional qualifiers and/or reason codes, a complete explanation of those qualifiers or reason codes must accompany the data review.

Table 1a. Data Qualifiers

Qualifier	Definition
J	The associated analyte concentrations may be inaccurate or imprecise due to the quality of the data generated because certain Quality Control (QC) criteria were not met.
N	The associated analyte identification may be inaccurate and the associated concentration represents an approximated value.
UJ	The non-detect result may be inaccurate or imprecise due to the quality of the data generated because certain QC criteria were not met.
R	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
X	Auditor defined.

NOTE: Where professional judgment is required, follow-up with the laboratory, technical knowledge and experience, and/or outreach for professional support/guidance may be applicable.

Table 1b. Reason Codes

Code	Definition
MC	Reported concentrations or analyte identification may be inaccurate due to improper or infrequent scope alignment.
IC	Identification may be inaccurate due to improper or infrequent Refractive Index (RI) liquid calibrations.
DR	The reported concentrations or structure/fiber identification may be inaccurate due to infrequent or discordant intra- and/or inter-analyst, laboratory duplicate, and/or reference material analyses.
B	The reported concentration may be inaccurate due to the presence of analyte structures/fibers in the associate contamination check or a contamination check was not performed as required.
SC	The reported concentration may be inaccurate due to the condition of samples upon receipt at the laboratory and/or improper storage prior to sample preparation and/or analysis.
DL	The number fields of view or points counted/analyzed are insufficient to meet the required limit of detection (LOD).
ID	The asbestos identification and concentrations may be inaccurate because the recorded optical properties (PLM) are not consistent with those described in SOW.
MA	The laboratory procedure did not follow the method-specific requirements.

DATA PACKAGE INSPECTION

If there are any concerns with the data package regarding apparent missing or incorrect information, contact the appropriate USEPA personnel for the project.

Items typically included in a data package include:

- Narrative
- Traffic Report/Chain-of-Custody (TR/COC) records
- Cross-reference to laboratory identification (ID)
- EDD print-outs
- Laboratory bench sheets (if applicable)
- Calibration documents
- Data completeness checklist
- Communications (if applicable)

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the PLM SOW and any future editorial revisions thereof. To use this document effectively, the reviewer should have an understanding of the analytical method used and a general overview of the sample set or Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analyses are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the data package (e.g., flexible analysis approval notices, airbills, Traffic Report/Chain of Custody Records (TR/COCs), Case Narratives).

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP), SOW, or similar document for the project for which samples were analyzed. If applicable, the reviewer should contact the appropriate USEPA personnel to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

Sample sets, or cases, routinely have unique quality control (QC) samples which require special attention from the reviewer. These include laboratory preparation samples, field duplicates, and Performance Evaluation (PE) samples, which must be identified. The sampling records (e.g., TR/COC Records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken;
2. Case number;
3. A complete list of samples, with information on:

    a. Collection and shipping dates;
    b. Preservation*;
    c. Sample matrix;
    d. Sample volumes;
    e. Field duplicates*;
    f. PE samples*;
    g. QC samples*; and
    h. Types of analysis.

(*if applicable)

The TR/COC documentation includes sample descriptions, date(s) and time(s) of sampling, sample location, and sample matrix. The laboratory's Case Narrative is another source of general information. The Case Narrative is required for inclusion in the data package and should contain comments that clearly describe the analyses and any unusual problems associated with a sample set or project, and state the limitations of the data. Unusual problems may include:

- Problems with matrices;
- Insufficient sample volume for analysis or reanalysis;
- Samples received in broken containers; and
- Unusual events.

The reviewer should also inspect telephone or communication logs detailing any discussions of sample or analysis issues between the laboratory and the USEPA Region.

POLARIZED LIGHT MICROSCOPY (PLM) DATA REVIEW

The data requirements to be checked are listed below:

I. Sample Receipt

II. Sample Preparation

III. Microscope Alignment

IV. Refractive Index Liquid Calibration

V. Fiber Identification Criteria

VI. Blank Analysis

VII. Reference Sample Analysis

VIII. Replicate Analysis

IX. Point Counting

X. Overall Assessment of Data

I. Sample Receipt

A. Review Items:

Traffic Report/Chain-of-Custody (TR/COC) records.

B. Objective:

The objective is to ascertain the validity of sample results based on the condition, packaging, and storage of the sample from time of collection to time of sample preparation and/or analysis.

C. Criteria:

Analyst inspection documentation must include verification that samples were properly packaged, sealed, are undamaged, and were labeled upon receipt at the laboratory.

D. Evaluation:

1. Verify that the TR/COC documentation indicates that the samples were received intact. Note in the Case Narrative if the samples were not packaged correctly, there were any problems with the samples upon receipt, or if the sample condition could affect the data.

2. Verify that the information recorded on the COC records, shipping documents, and sample containers are complete and in agreement.

3. Verify that the COC records have been signed and dated.

E. Action

Table 2. Sample Receipt Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Shipment and/or storage conditions are exceeded	Qualify as estimated (J)	Qualify as estimated (UJ)	SC
COC records, shipping documents, and sample container information are not in agreement*	Use professional judgment	Use professional judgment	SC
COC records not signed and dated*	Use professional judgment	Use professional judgment	SC

* Follow-up with laboratory may be required.

II. Sample Preparation

A. Review Items:

Sample preparation documentation, TR/COC, and NADES records (or equivalent).

B. Objective:

The objective is to determine from the review of the documents whether all samples were prepared, visual estimation procedures were applied, whether preparation procedures were applied, and whether contamination blanks were prepared at the frequency specified in the SOW, the laboratory's documented procedures (i.e., SOPs), or other applicable guidance document.

C. Criteria:

Slide (sample) preparation consists of an initial examination by stereomicroscope and the preparation of random slide mounts from representative sub-samples of the original sample in the appropriate refractive index (RI) liquid. For soil and bulk matrices, a minimum of three slide mounts are prepared.

D. Evaluation:

1. Verify that all samples listed on the COC for PLM analysis have been prepared for analysis.

2. Verify that the entire sample was examined by stereomicroscope to determine both homogeneity and provide a visual estimate of asbestos concentration.

3. Verify that appropriate procedures were used to prepare samples for analysis, and that the necessary gravimetric data, if applicable, have been recorded (i.e., pre- and post-ashing weights).

4. Verify that contamination blank(s) were prepared at the proper frequency.

E. Action:

Table 3. Sample Preparation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Stereomicroscope was not used to examine the entire sample for homogeneity and visual estimate of asbestos concentration	Qualify as estimated (J)	No action	MA
Samples were not prepared and/or recorded using the appropriate procedures	Qualify as unusable (R)	Qualify as estimated (UJ)	MA
Contamination blank(s) were not prepared at the required frequency	Qualify as estimated (J)	No action	B

III. Microscope Alignment

A. Review Items:

PLM alignment documentation.

B. Objective:

The objective is to determine if the PLM instrument was aligned in accordance with the method procedure and frequency requirements. A properly aligned PLM is critical to ensure the instrument is capable of providing acceptable data.

C. Criteria:

The following alignment checks must be performed on a daily basis or when the microscope is determined to be out of alignment by the individual analyst, whichever is more frequent:

- Centering of the stage and objectives
- Centering the optic axis
- Alignment of lower polar
- Alignment of upper polar

D. Evaluation:

Review the provided PLM alignment records to verify the microscope was properly aligned on the day(s) on which the applicable samples were analyzed.

E. Action:

Table 4. Microscope Alignment Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Alignment not performed at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	MC

IV. Refractive Index Liquid Calibration

A. Review Items:

Refractive Index (RI) liquid calibration records and NADES records (or equivalent).

B. Objective:

The objective is to determine whether proper calibration of the RI liquids was performed.

C. Criteria:

1. Each RI liquid used for routine sample preparation and analysis must be calibrated prior to initial use and as required thereafter, as specified in the appropriate method (i.e., PLM NIOSH 9002 requires weekly calibration). Records of these calibration activities must be maintained.

2. The difference between the calibrated RI of the liquid and the original RI of the liquid must not be greater than 0.004. If the difference is greater than 0.004, the liquid may not be used for the analysis of the samples.

D. Evaluation:

1. Verify that each RI liquid used for routine sample preparation and analysis were calibrated prior to initial use, and as applicable thereafter.

2. Ensure the difference between the calibrated RI of the liquid and the original RI of the liquid is not greater than 0.004.

E. Action:

Table 5. Refractive Index Liquid Calibration Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
RI liquid calibration failed criteria	Use professional judgment in qualifying as estimated (J) or unusable (R)	Qualify as estimated (UJ)	IC
RI calibration not performed at required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC

V. Mineral/Fiber Identification Criteria

A. Review Items:

NADES records (or equivalent), bench sheets, and raw data (i.e., calibration records).

B. Objective:

The objective is to determine whether optical properties have been recorded for reported fibers. Positive asbestos identification requires the determination of the following optical properties:

- Morphology
- Pleochroism
- Birefringence
- Angle of extinction
- Sign of elongation
- Refractive Indices (RI)

Asbestos cannot be reported in any quantity, including trace, until its optical properties have been measured and recorded.

C. Criteria:

The optical properties of fibrous material type(s) observed and recorded for samples analyzed are consistent with those described in the applicable SOW and/or method.

D. Evaluation:

1. For fibrous materials identified as asbestos, verify that the recorded optical properties are consistent with those provided in applicable SOW and/or method.

2. When non-asbestos fibers are observed, verify that at least one optical property that distinguishes the fiber from asbestos is measured and recorded on the bench sheet.

E. Action:

Table 6. Mineral/Fiber Identification Criteria Evaluation Actions

<table>
<tr><th rowspan="2">Deficiency</th><th colspan="2">Action</th><th rowspan="2">Reason Code</th></tr>
<tr><th colspan="2">Detected Analyte</th><th>Non-Detect Analyte</th></tr>
<tr><td rowspan="2">The recorded optical properties are not consistent with those of the asbestos type reported</td><td>Between amphiboles (i.e. WRTA)</td><td>Qualify as estimated (N)</td><td rowspan="2">No action</td><td rowspan="2">ID</td></tr>
<tr><td>Between serpentine (CH) and amphiboles (i.e. WRTA)</td><td>Qualify as unusable (R)</td></tr>
<tr><td>Optical property not recorded for non-asbestos fibrous material</td><td colspan="2">Qualify as estimated (N)</td><td>No action</td><td>ID</td></tr>
</table>

Note: Refer to Appendix C for a list the optical properties of asbestos fibers.

VI. Blank Analysis

A. Review Items:

NADES records (or equivalent), bench sheets, and raw data.

B. Objective:

The objective is to determine the existence and magnitude of contamination resulting from laboratory or field activities.

C. Criteria:

The following criteria for evaluation of blanks apply to all laboratory method or preparation blanks (contamination checks) associated with the samples.

1. Contamination checks (laboratory blanks) must be prepared and analyzed before the analysis of each set of samples (i.e., Laboratory Job), on days when analysis is performed.

2. Asbestos fibers must not be detected in the associated blanks.

If problems with a blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

D. Evaluation:

1. Verify that no asbestos was detected in the associated contamination blanks.

E. Action:

Table 7. Blank Analysis Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Asbestos fibers detected in contamination blank	Qualify as estimated (J)	Qualify as estimated (UJ)	B

Note: If the reported sample concentration is at or below the blank concentration level, the non-detect qualifier applies, unless otherwise specified in the applicable method, SOW, SOP, etc.

VII. Reference Sample Analysis

A. Review Items:

Reference sample analysis documentation and control charts.

B. Objective:

The objective is to determine whether QA records are maintained for each analyst that demonstrate the analysis of reference slides each day that samples are analyzed.

C. Criteria:

Reference slide results must be within the laboratory provided control limits. Reference slides must be analyzed at a rate of one (1) per sampling event (i.e., one per day, per analyst).

D. Evaluation:

Verify that a reference slide was analyzed at the proper frequency and that the results are within the control chart limits.

E. Action:

Table 8. Reference Material Analysis Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Reference sample analysis not performed at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	DR
Reference sample results fall outside of control limits	Use professional judgment to qualify as estimated (J) or unusable (R)	Qualify as estimated (UJ)	DR

VIII. Replicate Analysis

A. Review Items:

NADES records (or equivalent), bench sheets, and control charts.

B. Objective:

The objective is to determine whether replicate analyses are performed at the required frequency and meet replicate criteria requirements.

C. Criteria:

1. Replicate analyses must be performed at a frequency of one per sample set (i.e., Laboratory Job), one per 10 samples if the sample set exceeds 10 samples, or as directed in the applicable SOW and/or method.

    - Replicate sample analyses must meet the error rate requirement (i.e. RPD, as specified in the laboratory's SOP) on the qualitative analysis for samples containing chrysotile, amosite, and crocidolite. A slightly higher error rate may occur for samples that contain anthophyllite, actinolite, and tremolite, as it can be difficult to distinguish among the three types.

D. Evaluation:

1. Verify that replicate analyses were performed at a frequency of one per sample set <10 samples, one per 10 samples for sets >10 samples, or as defined in the applicable SOW and/or method, and meet the criteria noted above (C.1).

E. Action:

Table 9. Replicate Analysis Evaluation Actions

Deficiency	Action			Reason Code
	Detected Analyte		Non-Detect Analyte	
Replicate samples not analyzed at required frequency	Qualify as estimated (J)		No Action	DR
Replicate sample results fail established acceptance criteria	Between amphiboles (i.e. WRTA)	Qualify as estimated (J)	No Action	DR
	Between serpentine (CH) and amphiboles (i.e. WRTA)	Qualify as unusable (R)		

IX. Point Counting

A. Review Items:

NADES records (or equivalent), bench sheets, and raw data.

B. Objective:

The objective is to determine whether the following information is documented, as specified by the method (i.e. PLM PC-400): magnification; graticule size/type; number of slide mounts prepared; number of empty and/or non-empty points counted; and observance of fibers in a field of view, but not directly under a point.

If no asbestos fibers appear to be present in the sample a scanning option may be substituted for point counting.

C. Criteria:

A minimum of 400 points per sample are required to be counted. This could include a maximum of eight slides with 50 counts each or a minimum of two slides with 200 counts each per sample. Accuracy and precision improve with the number of points counted.

D. Evaluation:

1. Verify that there is a minimum of 400 points counted per sample, and related information documented as applicable.

2. If asbestos is identified during the point count, verify that the % Asbestos reported is calculated using the equation below:

Percent Asbestos for Point Counting

$$\% \text{ asbestos} = (A/N) \times 100$$

Where,

A = number of asbestos counts

N = number of points counted (400 minimum)

If % asbestos = 0, report "no asbestos detected (ND)"

If 0 < % asbestos < 1, report concentration to two decimal places, as "<1% asbestos," or as specified in the applicable method

If % asbestos is greater than 1%, report concentration to the nearest percent, as "≥1% asbestos," or as specified in the applicable method

E. Action:

Table 10. Point Count Analysis Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Less than 400 points counted; improper/missing information and/or documentation	Qualify as estimated (J)	Qualify as estimated (UJ)	DL
Percent asbestos improperly calculated*	Qualify as estimated (J)	Qualify as estimated (UJ)	MA

* Follow-up with laboratory may be required.

X. Overall Assessment of Data

A. Review Items:

Data package, Case Narrative, Quality Assurance Project Plan (QAPP) [specifically, the Data Quality Objectives (DQOs)], Statement of Work (SOW), Sampling and Analysis Plan (SAP), and any communications from the data user that concern the intended use and desired quality of the data.

B. Objective:

The objective of the overall assessment of a data package is to provide a brief narrative of significant data reviewer comments, concerns, and opinions about the quality and usability of the data.

C. Criteria:

All method criteria apply.

D. Evaluation:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

2. Evaluate any technical problems that have not been previously addressed.

3. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate application of the data.

4. If particle size and/or moisture content are requested, check the calculations and transcription of results for accuracy.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which were not already qualified based on the QC criteria previously discussed.

2. Write a brief narrative to give the data user an indication of the analytical limitations of the data. Note for EPA action any inconsistencies between the data and the Case Narrative. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context.

APPENDIX A: GLOSSARY

ACCURACY – The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of precision and bias.

ANALYTICAL SAMPLE – A portion of material to be analyzed that is enclosed in a single container, received from an external source, and identified by a unique sample number. Airborne samples are collected on membrane filters and bulk/soil samples are placed in zip-lock bags.

ANALYTICAL SENSITIVITY – Airborne asbestos concentration represented by one fiber or structure counted under the microscope. It is determined by the air volume collected and the proportion of the filter examined.

ASBESTIFORM (MORPHOLOGY) - A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength, flexibility, or long, thin fibers occurring in bundles.

ASBESTOS – The generic name used for a group of naturally occurring mineral silicate fibers of the serpentine and amphibole series, displaying similar physical characteristics although differing in composition.

BIAS – A systematic error manifested as a consistent positive or negative deviation from the known or true value.

BIREFRINGENCE – The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated, using a Michel-Lévy Chart, from the interference colors observed under crossed polarizers. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below:

Qualitative	Quantitative (N-n)
Weak	≤ 0.010
Moderate	0.011-0.025
Strong	0.026-0.100
Very Strong	0.101-0.200
Extreme	≥ 0.201
None	000 or Isotropic

BLANK – A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

BULK SAMPLE – A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and non-friable materials.

BUNDLE – Asbestos structure consisting of three or more fibers having a common axis of elongation with each fiber closer than one fiber diameter.

CHRYSOTILE – The most prevalent type of asbestos. Chrysotile is a fibrous mineral of the serpentine group which has the nominal composition:

$$Mg_3Si_2O_5(OH)_4$$

NOTE: In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+}, and Co^{2+} may also be present.

CONTAMINATION BLANK – Daily contamination check to determine the existence and magnitude of contamination resulting from laboratory activities.

CONTROL CHART – A graphical plot of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the system is in a state of statistical control.

DETECTION LIMIT – The smallest concentration/amount of the component of interest that can be determined by a single measurement with a stated level of confidence. See Limit of Detection.

DIFFERENTIAL COUNTING – The term applied to the practice of excluding certain types of fibers from the fiber count because they do not appear to be asbestos.

DUPLICATE SAMPLES – Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

ERROR – Difference between the true and the measured value of a quantity or parameter.

EXTINCTION – The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique, symmetrical, and undulose.

EXTINCTION ANGLE – For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

FIBER - With reference to asbestiform morphology, a structure consisting of one or more fibrils.

NOTE: Specifically defined by the method, i.e. (adapted from ISO 10312) a particle that is 0.5 µm or longer, with a length-to-width ratio of at least 3:1 or greater, and with parallel or stepped sides.

FIBRIL – A single fiber of asbestos which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearance.

NOTE: A fiber bundle may exhibit diverging fibers at one or both ends.

FIELD – The area within the graticule circle that is superimposed on the microscope image.

GRATICULE – A microscope slide or eyepiece that contains a grid of lines allowing the size of objects seen under magnification to be measured.

HETEROGENEOUS – Lacking uniformity in composition and/or distribution of material; components not uniform. Does not satisfy the conditions stated for homogeneous; i.e., layered or in clumps, very coarse grained, etc.

HOMOGENEOUS – Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

MATRIX – The predominant material of which the sample to be analyzed is composed.

MORPHOLOGY – The structure and shape of a particle. Characterization may be descriptive (e.g., platy, rod-like, acicular) or dimensional (e.g., length, diameter). See Asbestiform.

NONEMPTY POINT – The visual superposition of a point over any material in the slide preparation.

NVLAP – National Voluntary Laboratory Accreditation Program; program administered by NIST that accredits testing and calibration laboratories.

OFFICE OF LAND AND EMERGENCY MANAGEMENT (OLEM) – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.

PLEOCHROISM – The change in color or hue of colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

POINT COUNTING – A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used with PLM to determine the relative concentrations of asbestos minerals to non-asbestos sample components.

PRECISION – The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to one another. Precision is often expressed as standard deviation, variance, or range, in either absolute or relative terms.

REFERENCE MATERIAL – A material or substance, one or more properties of which are sufficiently well established to be used for the calibration of equipment, the assessment of a measurement method, or for assigning values to materials.

RI – Refractive Index (Index of Refraction); ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as *n* and varies with wavelength and temperature.

REPLICATION – Procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

SERPENTINE – A group of common rock-forming minerals having the nominal formula:

$$Mg_3Si_2O_5(OH)_4$$

Note: Minerals from this family that are important in asbestos analysis include chrysotile, lizardite, and antigorite.

SRM – Standard Reference Material; a reference material certified and distributed by the National Institute of Standards and Technology (NIST).

STATEMENT OF WORK (SOW) – A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

STRUCTURE – A microscopic fiber, fiber bundle, cluster, or matrix which may contain asbestos.

TREMOLITE, ANTHOPHYLLITE, AND ACTINOLITE – The non-asbestos form of these minerals which meet the definition of a fiber. It includes any of these minerals that have been chemically treated and/or altered.

WALTON-BECKETT GRATICULE – An eyepiece graticule specifically designed for asbestos fiber counting. It consists of a circle with a projected diameter of 100 ± 2 µm (area of about 0.00785 mm^2) with a cross hair having tic-marks at 3- µm intervals in one direction and 5- µm in the orthogonal direction.

APPENDIX B: TARGET ANALYTE LIST

"Asbestos" is a commercial term which applies to the asbestiform varieties of a group of naturally occurring silicate minerals. The six minerals listed below are specifically regulated as asbestos by the U.S. government (U.S. CFR, 2003).

- Chrysotile (asbestiform serpentine) CAS # 12001-29-5
- Amosite (asbestiform cummingtonite-grunerite) CAS # 12172-73-5
- Crocidolite (asbestiform riebeckite) CAS # 12001-28-4
- Asbestiform anthophyllite CAS # 77536-67-5
- Asbestiform tremolite CAS # 77536-68-6
- Asbestiform actinolite CAS # 77536-66-4

In addition to the regulated asbestos minerals, 388 minerals are known to occur, at least occasionally, in fibrous form, some of which are asbestiform. The precise chemical formulation of each species will vary with the location from which it was mined; therefore, the analytical sensitivity that can be achieved in asbestos analyses is highly matrix-dependent (Harris, 2007).

The Libby Amphibole (LA) solution series includes winchite, richterite, tremolite, and actinolite, (WRTA).

APPENDIX C: OPTICAL PROPERTIES OF ASBESTOS FIBERS

From Asbestos (bulk): Method 9002, Issue 2, dated 15 August 1994

Table 1. Optical Properties of Asbestos Fibers

		Refractive Index (Approximate Values)		
Mineral	Morphology and Color	⊥ to Elongation	to Elongation	Birefringence
Chrysotile	Wavy fibers with kinks. Splayed ends on larger bundles. Colorless to light brown upon being heated. Nonpleochroic. Aspect ratio typically >10:1.	1.54	1.55	0.002 - 0.014
Cummingtonite-Grunerite (Amosite)	Straight fibers and fiber bundles. Bundle ends appear broom-like or splayed. Colorless to brown upon heating. May be weakly pleochroic. Aspect ratio typically >10:1.	1.67	1.70	0.02 - 0.03
Crocidolite (Riebeckite)	Straight fibers and fiber bundles. Longer fibers show curvature. Splayed ends on bundles. Characteristic blue color. Pleochroic. Aspect ratio typically >10:1.	1.71	1.70	0.014 - 0.016 Interference colors may be masked by blue color.
Anthophyllite	Straight fibers and fiber bundles. Cleavage fragments may be present. Colorless to light brown. Nonpleochroic to weakly pleochroic. Aspect ratio generally <10:1.	1.61	1.63	0.019 - 0.024
Tremolite-Actinolite	Straight and curved fibers. Cleavage fragments common. Large fiber bundles show splayed ends. Tremolite is colorless. Actinolite is green and weakly to moderately pleochroic. Aspect ratio generally <10:1.	1.60 - 1.62 (tremolite) 1.62 - 1.67 (actinolite)	1.62 - 1.64 (tremolite) 1.64 - 1.68 (actinolite)	0.02 - 0.03

Table 1. Optical Properties of Asbestos Fibers (Continued)

			Central Stop Dispersion Staining Colors		
Mineral	Extinction	Sign of Elongation	RI Liquid	⊥ to Vibration	to Vibration
Chrysotile	Parallel to fiber length	+ (length slow)	1.550 HD	Blue	Blue-magenta
Cummingtonite-Grunerite (Amosite)	Parallel to fiber length	+ (length slow)	1.670 Fibers subjected to high temperatures will not dispersion-stain.	Red magenta to blue	Yellow
Cummingtonite Grunerite			1.680 1.680	pale blue blue	blue gold
Crocidolite (Riebeckite)	Parallel to fiber length	- (length fast)	1.700 1.680	Red magenta yellow	Blue-magenta pale yellow
Anthophyllite	Parallel to fiber length	+ (length slow)	1.605 HD 1.620 HD	Blue Blue-green	Gold to gold-magenta Golden-yellow
Tremolite-Actinolite	Oblique - 10 to 20• for fragments. Some composite fibers show extinction.	+ (length slow)	1.605 HD	Pale blue (tremolite) Yellow (actinolite)	Yellow (tremolite) Pale yellow (actinolite)

HD = high-dispersion RI liquid series.

APPENDIX C: OPTICAL PROPERTIES OF ASBESTOS FIBERS (Continued)

From SOP SRC-LIBBY-03 (Revision 2), dated 10 October 2008

Mineral	Morphology and Color	Refractive Indices		Birefringence	Extinction	Elongation Sign
		α	γ			
Tremolite[7]	Straight to curved fibers and bundles. Colorless to pale green.	1.600-1.628 1.604-1.612 1.599-1.612 1.6063	1.625-1.655 1.627-1.635 1.625-1.637 1.6343	0.017-0.028	Oblique (up to 21 °);	+ (length slow)
Actinolite[7]		1.600-1.628 1.612-1.668 1.613-1.628 1.6126	1.625-1.655 1.635-1.688 1.638-1.655 1.6393	0.017-0.028		+ (length slow)
Winchite	Straight to curved fibers or bundles. Colorless to pale blue Pleochroism weak to moderate: X-colorless, Y=light blue-violet, Z=light blue[3]	**1.618-1.626**[1] **1.618-1.621**[2] 1.629[3] 1.636[4]	**1.634-1.642**[1] **1.634-1.637**[2] 1.650[3] 1.658[4]	**0.008-0.019**[1] **0.016**[2] 0.021[3] 0.022[4]	Oblique, **22°**[1] **15.8°**[2] Oblique, 7-29°[8]	+ (length slow)
Richterite	Straight to curved fibers or bundles. Colorless, pale yellow, brown, pale to dark green, or violet[8] Pleochroism weak to strong in pale yellow, orange, and red[5]	**1.622-1.623**[1] 1.605-1.624[5] 1.615[6]	**1.638-1.639**[1] 1.627-1.641[5] 1.636[6]	**0.012-0.017**[1] 0.017-0.022[5]	Oblique, **21-22°**[1] Oblique, 5-45°[8]	+ (length slow)
Magnesio-riebeckite	Prismatic to fibrous aggregates. Blue, grey-blue, pale blue to yellow. Can be pleochroic.[8]	1.650-1.673[8]	1.662-1.676[8]	Up to 0.015[8]	Oblique, 8-40°[8]	- (length fast)[8]
Magnesio-arfvedsonite	Prismatic to fibrous aggregates. Yellowish green, brownish green, or grey-blue. Can be pleochroic.[8]	1.623-1.660[8]	1.635-1.680[8]	0.012-0.026[8]	Oblique, 18-45°[8]	- (length fast)[8]

APPENDIX D: ASBESTOS DATA REVIEW SUMMARY

ANALYTICAL TEST REPORT

Bulk Asbestos Analysis by PLM-VE

Prepared For: _____

Address: _____

Laboratory Name: _____

Address: _____

Report Review by: _____

Date

Standard Laboratory Data Package Checklist

Instructions: **For Analytical Test Reports, complete the following checklist and attach supporting documentation as outlined below.**

1 Laboratory Job No.:
2 Chain of Custody No.:
3 Date of sample receipt:
4 Number of samples received:
5 Analytical Method:
6 Test Report Correction No.:
7 Condition of samples:
8 Attachments:
Chain of Custody form(s)
Case Narrative and any modification forms
Analysis Results
Analytical Bench Sheet(s)

Verification: Laboratory and Validator Verification signifies that all laboratory QA/QC tasks were performed for the samples in this Laboratory Job Number and that this Analytical Test Report is accurate and complete. Laboratory Verification is done by the person who performed data entry and test results and Validator Verification is done by the person who performed the QC check of the data entry.

Laboratory Verification (Initials and Date) _____

Validator Verification (Initials and Date) _____