Office of Superfund Remediation and Technology Innovation (OSRTI)
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USEPA

TEM 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

AHERA	Asbestos Hazard Emergency Response Act	QAM	Quality Assurance Manual
ASTM	American Society for Testing and Materials	PE	Performance Evaluation
COC	Chain-of-Custody	QAPP	Quality Assurance Project Plan
DQO	Data Quality Objective	QC	Quality Control
EDXA	Energy Dispersive X-Ray Analysis	SAED	Selected Area Electron Diffraction
FWHM	Full Width at Half Maximum	SAP	Sampling and Analysis Plan
ISO	International Organization for Standardization	SOW	Statement of Work
LOD	Limit of Detection	SRM	Standard Reference Material
MCE	Mixed Cellulose Esters	TEM	Transmission Electron Microscopy
NADES	National Asbestos Data Entry Spreadsheet	TR	Traffic Report
NIST	National Institute of Standards and Technology		

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 Transmission Electron Microscopy (TEM), hereinafter referred to as the TEM 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 meet the specific technical and quality control (QC) criteria established in the TEM SOW; and (b) the usability of any data not meeting the specific technical and QC criteria established in the TEM 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	Structure/fiber counts and reported concentrations may be inaccurate due to improper or infrequent scope alignment and/or magnification calibrations.
IC	Identification by elemental composition, diffraction pattern or optical properties may be inaccurate due to improper or infrequent EDXA, and camera constant.
PA	Structure/fiber counts and reported concentrations may be inaccurate due to improper or infrequent calibration of the plasma asher.
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.
FB	The reported concentration may be inaccurate due to the presence of analyte structures/fibers in the associate field blank.
LB	The reported concentration may be inaccurate due to the presence of analyte structures/fibers in the associate laboratory blank.
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 of grid openings analyzed is insufficient to meet the required limit of detection (LOD).
ID	The asbestos identification and concentrations may be inaccurate because the recorded structure types are not consistent with those described in TEM SOW and/or method.

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 TEM 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 field blanks, field duplicates, and Performance Evaluation (PE) samples (if available), 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 (i.e., water samples)*;
    c. Sample matrix;
    d. Sample volumes;
    e. Field blanks*;
    f. Field duplicates*;
    g. PE 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.

TRANSMISSION ELECTRON MICROSCOPY (TEM) DATA REVIEW

The data requirements to be checked are listed below:

I. Sample Receipt

II. Sample Preparation

III. Microscope Alignment

IV. Instrument/Standard Calibration

V. Analytical Sensitivity

VI. Structure Recording & Identification

VII. Blank Analysis

VIII. Recount/Repreparation Analysis

IX. 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 not packaged in untreated polystyrene foam (peanuts), vermiculite, paper shreds, or excelsior packing materials; top covers and end plugs were in place for each cassette; and samples were properly sealed and undamaged, neither shipped nor stored with bulk samples, 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 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 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 and/or USEPA personnel 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 listed above whether the sample filters subjected to preparation were intact, whether the required blanks were prepared with the samples, whether sample filters were prepared for analysis using either the direct-transfer or the indirect-transfer procedure, and whether appropriate supporting preparation and communication documents are provided.

C. Criteria:

1. Filter samples must not be punctured, overloaded, or mishandled so as to disturb the fibers collected on the filter. If applicable, wet samples must be dried (i.e. by dessicator or oven).

2. A laboratory preparation blank must be prepared with each Laboratory Job prepared, for each preparation-type (direct and indirect), as applicable.

3. Sample specimen grids must meet the following specified quality criteria or the criteria defined in the applicable SOW and/or method:

    - A minimum of two sample specimen grids must be cleared of filter medium during the filter dissolution step.

- The sample grid must not be overloaded with particulate at >25%, or have loose debris.

- The particulate deposits on the specimen grid must be uniformly distributed from one grid opening to the next.

- The TEM grid loading with fibrous structures must be < 7000 structures/mm^2.

- No more than 25% of the grid openings may have broken carbon film over grid openings.

- If available and with USEPA approval, a low volume filter may be analyzed if the matching high volume filter is overloaded. Use of low volume backup filter due to overloading issues must be documented in the case narrative.

- Unless otherwise specified in the SOW, the appropriate USEPA representative must be contacted by the laboratory prior to applying the indirect-transfer preparation procedure for filter samples which exhibit loading of ≥25% of the filter surface, uneven filter loading, or the presence of loose dust or debris in the sampling cassette. USEPA direction will indicate how to proceed with the sample (e.g., whether to discard the impacted filter or proceed with indirect method ISO 13794).

D. Evaluation:

1. Review the sample preparation documentation, TR/COC, and NADES records to verify filter samples were not punctured, overloaded, or mishandled, and if wet, were dried by appropriate means. Also ensure that all samples listed on the COC for TEM analysis have been prepped for analysis.

2. Review the sample preparation documentation, TR/COC, and NADES records to verify a laboratory preparation blank was prepared in the same batch with the associated samples for each preparation day and method. Preparation blanks should be completed at the frequency described in the SOW or at 4%, if not specified.

3. Valid data cannot be obtained unless the sample specimen grids meet specified quality criteria. Reject grid preparations if any of the following is observed:

    - At least two of three sample specimen grids have not been cleared of filter medium during the filter dissolution step.

    - The sample grid is overloaded (>25%) with particulate or has loose debris.

    - The particulate deposits on the specimen grid are not uniformly distributed from one grid opening to the next (i.e., chi-square test in NADES).

    - The TEM grid is too heavily loaded (>7000 structures/mm^2) with fibrous structures to make an accurate count.

- More than approximately 25% of the grid openings have broken carbon film over grid openings.

- If applicable, review the case narrative for documentation on overloading issues.

4. If applicable, review the SOW, sample preparation documentation, TR/COC, NADES, and communication records to verify the appropriate USEPA representative was contacted for direction on whether or not to apply the indirect-transfer preparation procedure to filter samples exhibiting loading of ≥25% of the filter surface, uneven filter loading, or the presence of loose dust or debris in the sampling cassette.

E. Action:

Table 3. Sample Preparation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Filter(s) damaged or compromised	Qualify as unusable (R)	Qualify as unusable (R)	SC
Blanks were not prepared at the specified frequency	Use professional judgment in qualifying as estimated (J) or unusable (R)	No Action	LB
Grid specimen(s) do not meet specified quality criteria (i.e., >25% loading)	Use professional judgment in qualifying as estimated (J) or unusable (R)	Use professional judgment in qualifying as estimated (UJ) or unusable (R)	SC
For overloaded samples, SOW or USEPA direction was not documented for indirect preparation	Use professional judgment in qualifying as estimated (J)	Use professional judgment in qualifying as estimated (UJ)	SC

III. Microscope Alignment

A. Review Items:

TEM alignment documentation and NADES records.

B. Objective:

The objective is to determine whether the TEM microscope was aligned according to the manufacturer's specifications and instructions, and at the frequencies specified by the client, in the SOW, the laboratory's documented procedures (i.e., QAMs), or other applicable guidance document..

C. Criteria:

The electron gun, apertures, and tilt of the TEM must be aligned before structure counting is performed each day when analyses are being performed.

D. Evaluation:

Review the TEM bench sheets and document the scope numbers and dates used. Verify that the TEM Microscope Calibration Logs include the alignment date and analyst; and the scope alignment is checked and calibrated.

E. Action:

Table 4. Microscope Alignment Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
No alignment is performed on the day of analysis	Qualify as estimated (J)	Qualify as estimated (UJ)	MC
Alignment not performed at the required frequency*	Use professional judgment in qualifying as estimated (J)	Use professional judgment in qualifying as estimated (UJ)	MC

* Follow-up with the laboratory may be required.

IV. Instrument Calibration

A. Review Items:

Instrument Calibration documentation and NADES records.

B. Objective:

The objective is to verify whether calibration of the plasma asher, sample specimen grids, TEM screen magnification, camera constant for the SAED mode, and the EDXA system have been performed in accordance to method requirements and at the required frequencies.

C. Criteria:

1. Initially and at quarterly intervals, the low temperature asher must be calibrated by determining a calibration curve for the weight vs. ashing time of a collapsed mixed cellulose ester (MCE) filter, and adjusted to ash between 5% and 10%. If ashing by this amount generates a texture in the replica that affects structure counting, it is permissible to etch by less than this amount.

2. For each lot of sample specimen grids used, the standard deviation of the mean area of 20 grid openings from 20 sample specimen grids, one grid opening per specimen grid, must be less than 5%. If the standard deviation criterion of less than 5% cannot be demonstrated, the dimensions of each grid opening must be measured in the TEM at a calibrated magnification, recorded, and reported with the associated sample analyses.

3. Initially, and at monthly intervals, calibrate the magnification of the fluorescent screen and photo-negatives must be calibrated at 10,000X and 20,000X, or as specified for the project, using a diffraction or carbon grating replica. The variation in sequential

calibration measurements (i.e., two times the standard deviation), should be <5% of the mean calibration value.

4. The camera constant of the TEM must be calibrated at least monthly using a specimen grid supporting a carbon film on which a thin film of gold has been evaporated. With sequential calibrations, the variation in the camera constant measurements (2F) must be <5% of the mean value.

5. At least quarterly, the crossover spot must be measured to ensure it is less than 250 nanometers (nm) in diameter.

6. At least quarterly, the beam dose must be calibrated to minimize damage to chrysotile. A single chrysotile fibril >1 µm in length, from an NIST SRM sample, must be stable in the electron beam for at least 15 seconds.

7. Daily, prior to analyzing samples, EDXA low- and high-energy checks of the copper (Cu) and aluminum (Al) peak centers must be performed and adjusted as necessary.

8. Semi-annual verification of the EDXA must be performed to ensure a full-width at half maximum (FWHM) resolution of <175 eV for manganese (Mn) k_{α}.

9. Quarterly EDXA low-energy detector sensitivity check must be performed. This is accomplished by producing resolvable sodium (Na) K_a x-ray peaks for NIST SRM 1866 (crocidolite) mounted on a sample specimen grid.

10. Quarterly verification of the detector small-structure sensitivity is required. This is done by producing resolvable magnesium (Mg) K_a and silica (Si) K_a peaks from a single fibril (maximum diameter of ≤0.05 µm) for NIST SRM 1866, 1876a, or 1876b (chrysotile) mounted on a sample specimen grid.

11. Semi-annually k-factors for the elements commonly found in asbestos: Na, Mg, Al, Si, calcium (Ca) and iron (Fe) must be calculated. K-factors, can be measured from NIST SRM 2063a for Mg, Si, Ca, and Fe and Na and Al from materials such as albite, kaersutite, or NIST SRM 99a. The k-factors (relative sensitivity factors) relative to Si for elements found in asbestos must be:

    - Determined to a precision within 10% relative to the mean value obtained for Mg, Al, Si, Fe, and within 20% relative to the mean value obtained for Na.
    - Between 1.0 and 4.0 for Na.
    - Between 1.0 and 2.0 for Mg and Fe.
    - Between 1.0 and 1.75 for Al and Ca.
    - The k-factor for Mg relative to Fe must be ≤1.5.

D. Evaluation:

1. Verify that the low temperature asher is properly calibrated at quarterly intervals (if available; this information is typically verified during an on-site audit of the laboratory).

2. Verify that the average grid opening of sample specimen grids used was properly determined and within criteria (if available; this information is typically verified during an on-site audit of the laboratory).

3. Verify that the fluorescent screen and photo-negatives are calibrated for 10,000X and 20,000X magnification (or as specified otherwise) at monthly intervals, and that sequential calibration measurements are within criteria.

4. Verify that the camera constant is calibrated at least monthly and that the sequential calibration measurements are within criteria.

5. Verify that the crossover spot is measured quarterly to ensure it is less than 250 nanometers (nm) in diameter.

6. Verify that the beam dose is calibrated at least quarterly to minimize damage to chrysotile fibers.

7. Verify that the EDXA low- and high-energy check of the copper (Cu) and aluminum (Al) peak centers generated from the electronic beam are checked and adjusted (if necessary) daily, prior to analyzing samples.

8. Verify that on a semi-annual basis a verification to ensure a full-width at half maximum (FWHM) resolution of <175 eV for manganese (Mn) k_{α} is performed.

9. Verify that a low-energy sensitivity check is performed quarterly by producing resolvable sodium (Na) K_a x-ray peaks from NIST SRM.

10. Verify the detectors small-structure sensitivity on a quarterly basis by producing resolvable magnesium (Mg) K_a and silica (Si) K_a peaks from a single chrysotile fibril.

11. Verify that the k-factors, relative to Si for elements commonly found in asbestos (Na, Mg, Al, Si, Ca, and Fe), are calculated semi-annually and meet the following criteria:

    - Determined to a precision within 10% relative to the mean value obtained for Mg, Al, Si, Fe, and within 20% relative to the mean value obtained for Na.

    - Between 1.0 and 4.0 for Na.

    - Between 1.0 and 2.0 for Mg and Fe.

    - Between 1.0 and 1.75 for Al and Ca.

    - The k-factor for Mg relative to Fe must be ≤1.5.

E. Action:

Table 5. Instrument/Standard Calibration Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Magnification not calibrated at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	MC
Camera constant not calibrated at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
Spot size not measured at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
Beam dose not calibrated at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
Cu/Al high-, low-energy peaks not checked at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
Mn resolution not checked at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
Low energy sensitivity check of resolvable Na not performed at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
Small structure sensitivity check not performed at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC
K-factors are not calculated at the required frequency	Qualify as estimated (J)	Qualify as estimated (UJ)	IC

V. Analytical Sensitivity

A. Review Items:

NADES records, bench sheets, and raw data (i.e., calibration documentation, preparation logs for indirect preparation).

B. Objective:

The objective is to determine whether project-specific analytical sensitivity has been achieved by analyzing a sufficient number of grid openings.

C. Criteria:

The analytical sensitivity for both TEM preparation procedures corresponds to the detection of one (1) structure in the area of the specimen grid examined (i.e., air (cc^{-1})). The sensitivity can theoretically be lowered indefinitely by filtering larger volumes of air, concentrating the sample during specimen grid preparation (indirect transfer procedure only), or increasing the area of the sample specimen grid examined (i.e., examining more grid openings).

D. Evaluation:

Verify that a sufficient number of grid openings have been analyzed to achieve the required analytical sensitivity.

E. Action:

Table 6. Analytical Sensitivity Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Analytical sensitivity not achieved; insufficient grid openings analyzed *; appropriate stopping rules not applied	Qualify as estimated (J)	Qualify as estimated (UJ)	DL

* Additional grid openings can be analyzed post-hoc at USEPA discretion.

VI. Structure Recording and Identification

A. Review Items:

NADES records, bench sheets, and raw data.

B. Objective:

Fibrous asbestos structures are classified on the basis of morphology, diffraction pattern, and elemental composition, which must be properly recorded and supported by the applicable diffraction pattern (SAED) photographs and EDXA spectra.

C. Criteria:

Note: The criteria listed herein are specific to analyses by the ISO10312 method, and serve as an example of criteria to evaluate for all TEM methods. Refer to the criteria specified in the analytical method applicable to the specific project under evaluation (i.e., AHERA, ASTM5755, EPA Method 100.2).

1. Grid openings must be analyzed from a minimum of two sample specimen grids, with approximately half of the grid openings analyzed from each separate samples specimen grid.

2. Each fibrous structure that is a separate entity must be designated as a primary structure and further categorized as fiber (F), bundle (B), disperse cluster (CD), compact cluster (CC), disperse matrix (MD), or compact matrix (MC), as defined in the applicable SOW and/or method.

3. Disperse clusters and disperse matrices must be open networks in which at least one of the individual fibers or bundles can be separately identified and measured as component structures, as indicated in the laboratory-provided sketches (or similar).

4. Recordable fibers must have parallel or stepped sides, a minimum length of 0.5 µm, and an aspect ratio of 3:1 or greater, or as defined in the applicable SOW and/or method.

5. Recordable bundles can have any dimension provided it contains individual component fibers with an aspect ratio greater than 3:1, or as defined in the applicable SOW and/or method.

6. Cluster and matrices designations must be followed by a two digit-number, the first digit representing the total number of fibers and bundle comprising the structure and the second digit representing the total number of fibers and bundles longer than 5 µm.

7. Structures/fibers must identified by their morphology, diffraction (SAED) characteristics and elemental composition, with the following minimum requirements for the identification of serpentine (Chrysotile) and amphiboles:

    - Chrysotile (C) – The minimal classification will be specified in the SOW or QAPP.

    - Amphibole (A) – Unless specified otherwise, the minimal classification will be by morphology, qualitative SAED, and qualitative EDXA (X), level ADX.

8. For each sample in which a fiber is identified, the level of documentation must include (as specified in the SOW or QAPP):

    - For each sample, a digital photograph and/or sketch of a minimum of one structure for each type recorded in NADES. A minimum of one structure of each type recorded in NADES must have an EDXA and SAED performed, recorded, and documentation provided.

Note: Due to the complexity and variation in which primary and component structures must be recorded and identified, the data reviewer is advised to review the applicable sections of the applicable SOW and/or method.

D. Evaluation:

1. Verify that grid openings are analyzed from a minimum of two sample specimen grids, with approximately half of the grid openings analyzed from each separate samples specimen grid. Verify that adjacent grid openings are not analyzed.

2. Verify that primary structures are designated as fibers (F), bundles (B), disperse clusters (CD), compact clusters (CC), disperse matrices (MD) or compact matrices (MC).

3. Verify that identifiable fibers and bundles within disperse clusters and disperse matrices are separately identified and measured as component structures.

4. Verify that recordable fibers have parallel or stepped sides, a minimum length of 0.5 µm, and an aspect ratio of 3:1 or greater, or as defined in the applicable SOW and/or method.

5. Verify that recordable bundles contain individual component fibers with an aspect ratio greater than 3:1., or as defined in the applicable SOW and/or method.

6. Verify that cluster and matrix designations are followed by a two digit-number, the first digit representing the total number of fibers (up to 5) and bundle comprising the structure and the second digit representing the total number of fibers and bundles longer than 5 µm.

7. Verify that counting of structures/fibers is continued for each specimen grid until either 100 asbestos structures have been recorded or a sufficient area of the specimen grid has been examined to achieve the desired analytical sensitivity.

8. Verify the following minimum requirements for the identification of serpentine (Chrysotile) and amphiboles:

    - Chrysotile (C) – Unless specified otherwise, the minimal classification will be by morphology and qualitative SAED (D), level CD.

    - Amphibole (A) – Unless specified otherwise, the minimal classification will be by morphology, qualitative SAED, and qualitative EDXA (X), level ADX.

9. Verify that a few fibers for each sample in which a fiber is identified using ADX, an EDXA spectrum is recorded, as specified in the SOW or QAPP.

E. Action:

Table 7. Mineral/Fiber Identification Criteria Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Grid opening were analyzed from one specimen grid	Qualify as estimated (J)	Qualify as estimated (UJ)	DL
Primary structures not properly recorded	Qualify as estimated (J)	No action	ID
Component structures not properly recorded	Qualify as estimated (J)	No action	ID
Recorded fibers/bundles do not meet minimum requirements	Qualify as estimated (J)	No action	ID
Count terminated prior to the analysis of the required number of GOs	Qualify as estimated (J)	Qualify as estimated (UJ)	DL
Minimum requirement for chrysotile identification not achieved	Qualify as estimated (J)	Qualify as estimated (UJ)	ID
Minimum requirement for amphibole identification not achieved	Qualify as estimated (J)	Qualify as estimated (UJ)	ID
Required SAED photos not provided	Qualify as estimated (J)	Qualify as estimated (UJ)	ID
Required EDXA spectra not provided	Qualify as estimated (J)	Qualify as estimated (UJ)	ID

VII. Blank Analysis

A. Review Items:

NADES records, 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:

1. The analysis of laboratory preparation blanks must be performed at a frequency of one per preparation day, or as specified in the applicable SOW and/or method.

2. The analysis of field blanks must be performed at a frequency of one per sampling event or per 20 samples if the event is >20 samples, or as specified in the applicable SOW and/or method.

3. If samples are prepared using both direct- and indirect-transfer, preparation blanks must be prepared for both procedures.

4. For each blank type, there must be <10 asbestos structures of any type detected per mm^2 and/or <0.1 fibers or bundles per mm^2 for >5 µm in an analysis of 10 grid openings, or as specified in the applicable SOW and/or method.

D. Evaluation:

If any problems with the blanks exist, all data associated with the blank must be carefully evaluated to determine whether or not there is an inherent contamination for the samples in the batch, or the problem is an isolated occurrence not affecting the sample data.

1. Review the blanks reported on the TEM Blank documentation and on the NADES, and compare the results to the blank results on the bench sheets. If the bench sheet and NADES values do not match, contact the laboratory for correction.

2. Verify that both direct- and indirect-transfer preparation blanks are prepared and analyzed if samples are prepared using both procedures.

3. Verify that there are <10 asbestos structures per mm^2 and/or <0.1 fibers or bundles per mm^2 for >5 µm detected in the blanks. If the number of asbestos structures detected exceeds the requirement, corrective action should be initiated, which may include re-preparation and reanalysis of the associated samples.

E. Action:

Table 8. Blank Analysis Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Asbestos structures detected in method blanks	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.

VIII. Recount/Repreparation Analysis

A. Review Items:

NADES records, bench sheets, and raw data.

B. Objective:

The objective is to determine whether recount and repreparation analyses (including inter-laboratory samples, if applicable) were performed at the required frequencies and meet the criteria requirements.

C. Criteria:

1. Each intra-laboratory QC sample type, including Re-analysis Same Analyst and Re-analysis Different Analyst, must be analyzed at a minimum frequency of one per sampling event or one per 20 samples if sampling event is >20 samples, or as directed in the applicable SOW and/or method.

2. Same-and different-analyst analyses must be analyzed at a frequency of one of the samples in the sample set or one per 20 samples if the sample set exceeds 20 samples, or as directed in the applicable SOW and/or method.

    - A sample with countable structures is to be used for re-analysis, as required by the project, with all original sample grid openings and a maximum of 10 structures per GO re-counted in the QC analysis. If no structures were found, an ND sample is to be selected.

    - The number of asbestos structures counted in each grid opening with ≤10 structures that are identical.

    - The number of asbestos structures counted in grid openings with >10 structures match within 10% RPD.

    - The asbestos mineral types agree 100% on chrysotile versus amphibole.

    - Structure lengths agree to within 0.5 µm or 20% (whichever is less stringent) for fibers and bundles.

- For MD, MC, CD, and CC, the overall dimensions of the matrix/cluster, in two perpendicular directions representing the maximum dimensions, shall be recorded.

- For MR and CR, a maximum of five (5) matrix/cluster residuals for any matrix/cluster shall be recorded.

D. Evaluation:

1. Verify that intra-laboratory QC samples are prepared and analyzed for each sample set at a minimum frequency of one per 20 samples, or as defined in the applicable SOW and/or method, and meet the following criteria:

    - Select and examine the same grid openings that were examined as the original analysis. Note that project specific requirements may be applied.

    - The original sample with the highest number of countable structures is used for re-analysis.

    - The number of asbestos structures counted in each grid opening with ≤10 structures are identical.

    - The number of asbestos structures counted in grid openings with >10 structures match within 10%.

    - The asbestos mineral types agree 100% on chrysotile versus amphibole.

    - Structure lengths agree to within 0.5 µm or 20% (whichever is less stringent) for fibers and bundles.

    - For MD, MC, CD, and CC, the overall dimensions of the matrix/cluster, in two perpendicular directions representing the maximum dimensions are recorded.

    - For MR and CR, a maximum of five (5) matrix/cluster residuals for the matrix/cluster are recorded.

E. Action:

Table 9. Recount/Repreparation Analysis Evaluation Actions

Deficiency	Action		Reason Code
	Detected Analyte	Non-Detect Analyte	
Recount/Repreparation samples not analyzed at required frequency*	Use professional judgment in qualifying as estimated (J)	Use professional judgment in qualifying as estimated (UJ)	DR
Recount/Repreparation sample results fall outside of established acceptance criteria	Qualify as estimated (J)	Qualifying as estimated (UJ)	DR

* Post-hoc analyses may be an appropriate action to meet project requirements.
NOTE: If samples require qualification, both the original and QC sample are qualified.

IX. 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 the 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.

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 any 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 or sample jars.

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.

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, which can both occur naturally and used commercially. 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.

CLUSTER – A structure in which two or more fibers or fiber bundles are randomly oriented in a connected grouping.

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 from observance of morphology, SAED patterns, and EDS spectrum.

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

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 BLANK – An analyte-free matrix (e.g., sampling cassette, filter) carried to the sampling site, exposed to the sampling conditions for ≤ 30 seconds, returned to the laboratory, and carried through all steps of the preparation and analysis. Field blanks may or may not be identified as such when delivered to the laboratory, and should be treated and reported as a routine sample.

FILTER LOT BLANK – An unopened sampling cassette with filter, or a filter from a new lot analyzed to verify that the matrix is contaminant-free.

FWHM – Full width at half maximum; a measure of the width of a line in a spectrum, either emission or absorption. It is the width of the line at a point that is half the line's peak value and is used when measuring spectrum peaks in EDXA.

GRID – An open structure on which a sample specimen is mounted to aid in its examination of a TEM grid opening).

LOD – Limit of Detection; calculated airborne asbestos structure concentration, equivalent to counting 2.99 asbestos structures in the analysis.

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

MATRIX – FOR TEM: Structure in which one or more fibers or fiber bundles touch, are attached to, or are partially concealed by a single particle or connected group of non-fibrous particles.

MCE – Mixed Cellulose Esters; one type of matrix for sample collection or sample analytical filters.

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.

PREPARATION BLANK – An unused filter, obtained from a lot of filters which has been shown to be free from contamination, which is exposed while a set of sample filters are processed, and is taken through all of the preparation, analysis, and reporting steps simultaneously with the sample set.

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.

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

SAED – Selected area electron diffraction; technique in electron microscopy in which the crystal structure of a small area of a sample is examined.

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 the required analytical methods and quality control procedures to be followed in order for the data to support its intended use.

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

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: ASBESTOS DATA REVIEW SUMMARY

ANALYTICAL TEST REPORT

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) _____