ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 3: Asbestos Testing

TNI Standard

P.O. Box 2439
Weatherford, TX 76086
817-598-1624
www.nelac-institute.org

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VOLUME 1, MODULE 3
Quality Systems for Asbestos Testing

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VOLUME 1, MODULE 3
Quality Systems for Asbestos Testing

1.0 Introduction

This Standard applies to laboratories undertaking the examination of asbestos samples. This Standard is organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos.

2.0 Scope

The essential quality control (QC) procedures applicable to asbestos measurements are included in this document. Additional QC requirements that are specified by method, regulation or project shall be met by laboratories.

3.0 Terms and Definitions

The relevant definitions from TNI, Volume 1, Module 2, Section 3.0 are the preferred references. Definitions related to this document, which are used differently or do not exist in the above references, are defined below.

3.1 Additional Terms and Definitions

Asbestos: "Asbestos" refers to the asbestiform varieties of chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite-gruenerite); anthrophyllite; tremolite; and actinolite.

Asbestos Containing Material (ACM): Any material containing more than 1% asbestos.

Friable: "Friable" materials are those materials that, when dry, may be crumbled, pulverized, or reduced to powder by hand pressure. This includes previously non-friable material, after such previously non-friable material becomes damaged to the extent that when dry it may be crumbled, pulverized, or reduced to powder by hand pressure.

Non-friable Organically Bound (NOB): This term (NOB) refers to a wide range of non-friable building materials embedded in flexible-to-rigid asphalt or vinyl matrices. This includes vinyl asbestos tiles (VAT), mastic, asphalt shingles, roofing materials, paint chips, caulking and glazing, etc.

3.2 Exclusions and Exceptions

Reserved

4.0 Method Selection

Refer to TNI Standard Volume 1 Module 2 (i.e., EL-V1M2), Sections 5.4.2, 5.4.3 and 5.4.4.

The inclusion of the analyte in the method shall meet all required calibration requirements of the method and the QC requirements of the method to which the analyte is being added. If no QC

exists in the method, the laboratory shall adhere to the requirements outlined in a similar reference method (when available). A method that meets these requirements shall be identified in such a way so that there is no confusion that the method has been modified.

When it is necessary to use methods not covered by reference methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test. The method developed shall have been validated appropriately before use.

5.0 Method Validation

Prior to acceptance and institution of any method for which data will be reported, all methods shall be validated. Laboratories shall participate in proficiency test programs where available. The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data. Non-standard methods must comply with the requirements in Volume 1, Module 2, and Section 5.4.5.

6.0 Demonstration of Capability (DOC)

6.1 General

a) An individual who performs any activity involved with preparation and/or analysis of samples must have constant, close supervision as defined in the laboratory's training procedure until a satisfactory initial DOC is completed (see Section 1.6.2).

b) Thereafter, ongoing DOC (Section 6.3), as per the QC requirements specified by method test variability/reproducibility (such as laboratory control samples) is required.

c) In cases where an individual has prepared and/or analyzed samples using a method that has been in use by the laboratory for at least one (1) year prior to applying for accreditation, and there have been no significant changes in instrument type or method, the on-going DOC shall be acceptable as an initial DOC. The laboratory shall have records on file to demonstrate that an initial DOC is not required.

d) All demonstrations shall be documented. All data applicable to the demonstration shall be retained for a minimum of five (5) years and readily available at the laboratory.

6.2 Initial DOC

An individual must successfully perform an initial DOC prior to using any method (see Section 6.1.a) above), unless exempted through Clause 6.1.c), and at any time there is a change in instrument type or method, or any time that a method has not been performed by the analyst in a twelve (12) month period.

6.2.1 The laboratory shall document each initial DOC in a manner such that the following information is readily available for each affected employee:

a) analyst(s) involved in preparation and/or analysis;

b) matrix;

c) analyte(s), class of analyte(s), or measured parameter(s);

d) identification of method(s) performed;

e) identification of laboratory-specific SOP used for analysis, including revision number;

f) date(s) of analysis; and

g) summary of analyses, including information outlined in Section 6.2.2.c).

6.2.2 For asbestos, if the method or regulation does not specify a DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to DOC are adequate.

a) The analyte(s) shall be diluted in a volume of clean quality system matrix (a sample in which no target analytes or interferences are present at concentrations that will impact the results of a specific method) sufficient to prepare four (4) aliquots.

b) At least four (4) aliquots shall be prepared and analyzed according to the method either concurrently or over a period of days.

c) Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations of the population sample (in the same units) for each analyte of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory shall assess performance against established and documented criteria.

d) Compare the information from c) above to the corresponding acceptance criteria for precision and accuracy in the method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all analytes meet the acceptance criteria, the analysis of actual samples may begin. If any one of the analytes does not meet the acceptance criteria, the performance is unacceptable for that analyte.

e) When one or more of the tested analytes fail at least one of the acceptance criteria, the analyst shall proceed according to i. or ii. below.

i. Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with c) above.

ii. Beginning with c) above, repeat the test for all analytes that failed to meet criteria.

f) Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with b).

6.3 On-Going DOC

6.3.1 The laboratory shall have a documented procedure describing ongoing DOC that includes procedures for how the laboratory will identify data associated with ongoing DOCs. The analyst(s) shall demonstrate on-going capability by routinely meeting the QC requirements of the method, laboratory SOP, client specifications, and/or this Standard. If the method has not been performed by the analyst in a twelve (12) month period, an initial DOC (6.2) shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

6.3.2 For asbestos, this ongoing DOC may be one of the following:

a) acceptable performance of a blind sample (single blind to the analyst) or successful analysis of a blind sample on a similar method using the same technology (e.g., EPA Methods 100.1 and 100.2);

b) another initial DOC;

c) at least four (4) consecutive laboratory control samples (LCS) with acceptable levels of precision and accuracy. The laboratory shall determine the acceptable limits for precision and accuracy prior to analysis. The laboratory shall tabulate or be able to readily retrieve four (4) consecutive passing LCS or reference sample(s) for each method for each analyst each year; or

d) a documented process of analyst review using QC samples. The QC samples can be reviewed to identify patterns for individuals or groups of analysts and to determine if corrective action or retraining is necessary.

7.0 Technical Requirements

7.1 Reference Materials

Refer to methods referenced in the following Sections for specific equipment requirements. If NIST standard reference materials (SRM) specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

7.2 Transmission Electron Microscopy

Refer to methods referenced in the following sections for specific equipment requirements.

7.2.1 Water and Wastewater

7.2.1.1 Calibration

All calibrations listed below (unless otherwise noted) shall be performed under the same analytical conditions used for routine asbestos analysis and shall be recorded and include date and analyst. Frequencies stated below may be reduced to “before next use” if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance.

a) Magnification Calibration. Magnification calibration shall be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A log shall be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data shall be displayed on control charts that show trends over time.

b) Camera Constant. The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode shall be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen shall be at the eucentric position for this calibration. This calibration shall allow accurate (<10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen, camera film/image file, or computer screen. This shall also allow accurate (<5% variation) measurement of zone axis SAED patterns on permanent media (e.g., film). Calibrations shall be performed weekly to establish the stability of the camera constant. Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data shall be displayed on control charts that show trends over time.

c) Spot Size. The diameter of the smallest beam spot at crossover shall be less than 250nm as calibrated quarterly. Calibration data shall be displayed on control charts that show trends over time.

d) Beam Dose. The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril >1µm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least fifteen (15) seconds.

e) Energy Dispersive X-Ray Analysis (EDXA) System

i. The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

ii. The ability of the system to resolve the Na Kα line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid or equivalent.

iii. The k-factors for elements found in asbestos (Na, Mg, Al, Si, Ca, and Fe) relative to Si shall be calibrated semiannually, or anytime the detector geometry may be altered. NIST SRM 2063a or similar standard shall be used for Mg, Si, Ca, Fe, while k-factors for Na and Al may be obtained from suitable materials such as albite, kaersutite, or NIST SRM 99a or equivalent. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca, and Fe, and within 20% relative to the mean value obtained for Na. The k-factor relative to Si for Na shall be between 1.0 and 4.0, for Mg and Fe shall be between 1.0 and 2.0, and for Al and Ca shall be between 1.0 and 1.75. The k-factor for Mg relative to Fe shall be 1.5 or less. Calibration data shall be displayed on control charts that show trends over time.

iv. The detector resolution shall be checked quarterly to ensure a full-width half maximum resolution of <175 eV at Mn Kα (5.90 keV). Calibration data shall be displayed on control charts that show trends over time.

v. The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas shall be avoided in asbestos analysis.

vi. The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile or equivalent.

f) Low Temperature Asher. In accordance with AHERA requirements (40CFR, Part 763, Subpart E, App. A), the low temperature ash shall be calibrated quarterly for 5-10% mass loss by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose ester (MCE) filters. Calibration data shall be displayed on control charts that show trends over time.

g) Grid Openings. The area of the TEM grid openings shall be calibrated using an appropriate standard at a frequency of twenty (20) openings per twenty (20) grids per lot of 1000 grids or less, or at least one (1) TEM grid opening per sample.

The variation in the calibration measurements (two times the standard deviation, 2s) shall be <5% of the mean calibration value.

7.2.1.2 Test Variability/Reproducibility

All analyses shall be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks shall be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from inter-laboratory proficiency testing.

NOTE: Relative standard deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at 7 MFL in inter-laboratory proficiency testing, while RSD for chrysotile was higher, 50% at 6 MFL.

a) Replicate. A second, independent analysis shall be performed on the same grids, but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5x of Poisson standard deviation. This shall be performed at a frequency of one (1) per one hundred (100) samples, or at last annually, whichever is more frequent.

b) Duplicate. A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0x of Poisson standard deviation. This shall be performed at a frequency of one (1) per one hundred (100) samples, or at least annually, whichever is more frequent.

c) Verified Analyses. A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two (2) sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of one (1) per twenty (20) samples. Qualified analysts shall maintain an average of ≥ 80% true positives, ≤ 20% false negatives, and ≤ 10% false positives.

7.2.1.3 Analytical Sensitivity

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed. Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

7.2.1.4 Data Reporting

a) The concentration of asbestos in a given sample shall be calculated in accordance with EPA/600/R-94/134, Method 100.2, Section 12.1 or Method 100.1, Section 7.

b) Measurement Uncertainties. The laboratory shall calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

7.2.2 Air

7.2.2.1 Calibration

All calibrations shall be performed in accordance with Section 7.2.1.1, with the exception of: a) magnification used for fiber counting shall be 15,000 to 20,000x and b) camera constant shall be performed at least monthly.

7.2.2.2 Test Variability/Reproducibility

a) All analyses shall be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks shall be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from inter-laboratory proficiency testing.

b) The laboratory and TEM analysts shall obtain mean analytical results on NIST SRM 1876b or equivalent so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section 7.2.1.2.c Verified Analysis, below. NIST SRM 1876b or equivalent shall be analyzed a minimum of once per year by each TEM analyst.

c) The laboratory shall have documentation demonstrating that TEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures greater than or equal to 1 µm in length in known traceable standard materials such as NIST bulk asbestos SRM 1866(or equivalent).

d) Inter-laboratory analyses shall be performed to detect laboratory bias. The frequency of inter-laboratory verified analysis shall correspond to a minimum of one (1) per two hundred (200) grid square analyses for clients.

e) The following QA sample analyses shall be performed:

i. Replicate. A second, independent analysis shall be performed in accordance with Section 7.2.1.2.a);

ii. Duplicate. A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0x of Poisson standard deviation. This shall be performed at a frequency of one (1) per one hundred (100) samples; and

iii. Verified Analyses. A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section 7.2.1.2.c).

f) If more than one TEM is used for asbestos analysis, intermicroscope analyses shall be utilized to detect instrument bias.

7.2.2.3 Analytical Sensitivity

An analytical sensitivity of 0.005 structures/cm^2 is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air

sampled.

7.2.2.4 Data Reporting

a) The concentration of asbestos structures in a given sample shall be calculated in accordance with the method utilized.

b) Measurement Uncertainties. The laboratory shall calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

7.2.3 Bulk Samples

7.2.3.1 Calibration

All calibrations shall be performed in accordance with Section 7.2.2.1.

7.2.3.2 Test Variability/Reproducibility

Bulk samples with low (< 10%) asbestos content are the most problematic. Laboratories must be able to demonstrate competency quantifying samples in lower concentrations (1%-10%). Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks shall be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from inter-laboratory proficiency testing.

a) Intra-Analyst Precision. At least one (1) out of fifty (50) samples shall be re-analyzed by the same analyst. For single analyst laboratories, at least one (1) out of every ten (10) samples shall be re-analyzed by the same analyst.

b) Inter-Analyst Precision. At least one (1) out of fifteen (15) samples shall be re-analyzed by another analyst. Inter-analyst results will require additional re-analysis, possibly including another analyst, to resolve discrepancies when classification Asbestos Containing Materials (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.

c) Inter-Laboratory Precision. The laboratory shall participate in round robin testing with at least one (1) other laboratory. Samples shall be sent to this other laboratory at least four (4) times per year. These samples shall be samples previously analyzed as QC samples. Results of these analyses shall be assessed in accordance with QC requirements. The QC requirements shall address misclassifications (false positives, false negatives) and misidentification of asbestos types. At a minimum, all analysts shall participate annually.

7.2.3.3 Analytical Sensitivity

The analytical sensitivity is dependent on the type of bulk material being analyzed, preparation method used, and means of analysis.

7.2.3.4 Data Reporting

The concentration of asbestos in a given sample shall be calculated in accordance the method utilized (e.g. EPA 600/M4-82-020 (1982), 40 CFR Part 763, Subpart E, Appendix E (2017)).

NOTE: Measurement Uncertainties. Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction has revealed an inter-laboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

7.2.4 Quality Control

7.2.4.1 Negative Controls

a) Water and Wastewater

i. Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one (1) bottle from each batch, or a minimum of one (1) from each twenty-four (24), shall be tested for background level. When using glass bottles, four (4) bottles from each twenty-four (24) shall be tested. An acceptable bottle blank level is defined as < 0.01 Million Fibers per Liter (MFL) that are greater than 10 µm.

ii. A process blank sample consisting of fiber-free water shall be run before the first field sample. For a 10 grid opening the quantity of blank water shall be greater than or equal to 150 ml for a 25 mm filter and greater than or equal to I liter for a 47 mm filter in Method 100.2 or greater than or equal to 100 ml for a 25 mm filter and greater than or equal to 500 ml for a 47 mm filter in Method 100.1.

b) Air

i. A laboratory blank filter of the same pore size and material shall be prepared for each preparation batch of samples. A laboratory blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared alongside wedges from the sample filters. At minimum, the laboratory blank filter shall be analyzed for each twenty (20) samples analyzed. Maximum contamination on a single polycarbonate blank filter shall be no more than 53 structures/mm^2. Maximum average contamination for all blank polycarbonate filters shall be no more than 18 structures/mm^2. Maximum contamination on a single MCE blank filter shall be less than 15 structures/mm^2. Maximum average contamination for all blank MCE filters shall be less than 5 structures/mm^2.

ii Field blanks shall be provided in accordance with the analytical method utilized and tested for background level if required. A sealed, unopened, blank cassette shall be included with each set of AHERA samples.

c) Bulk Samples

i. Blanks shall be prepared including all the equipment used to prepare and homogenize field samples. Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866, shall be performed at a frequency of one for every twenty samples analyzed. The detection of asbestos at a concentration exceeding 0.1% will require an investigation to detect and remove the source of the asbestos contamination.

ii. The laboratory shall maintain a list of non-asbestos fibers that can be confused with asbestos. The list shall include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos.

iii. The laboratory shall have a set of reference asbestos materials, from which a set of reference diffraction and x-ray spectra are developed.

7.2.4.2 Other Measures

a) Water and Wastewater

i. Filter preparations shall be made from all six (6) asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between one (1) and twenty (20) structures (>10µm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of one (1) per one hundred (100) samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with S. Turner and E.B. Steel, NISTIR 5351, Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0, 1994.

ii. NIST SRM 1876b or equivalent shall be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM.

b) Air

i. Filter preparations shall be made from all six (6) asbestos types in accordance with Section 7.2.4.2.a)i.

ii. NIST SRM 1876b or equivalent shall be analyzed annually.

c) Bulk Samples

All analysts shall be able to correctly identify the six (6) regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite).

7.2.4.3 Standards and Reagents

a) The QC program shall establish and maintain provisions for asbestos standards.

b) Reference standards that are used in an asbestos laboratory shall be obtained from NIST, EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos, whenever possible. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

c) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

d) All reagents used shall be analytical reagent grade or better.

e) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

7.3 Phase Contrast Microscopy

7.3.1 Calibration

7.3.1.1 At least once daily, the analyst shall use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

7.3.1.2 The phase-shift detection limit of the microscope shall be checked monthly or the next usage (whichever is less often), or after modification or relocation using a phase-contrast test slide for each analyst/microscope combination.

7.3.1.3 Prior to ordering the Walton-Beckett graticule, calibration (in accordance with NIOSH 7400, Issue 3, 14 June 2019, Appendix A), shall be performed to obtain a counting area 100 µm in diameter at the image plane. The diameter, dc (mm), of the circular counting area and the disc diameter shall be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 µm ± 2 µm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter shall be re-measured (or recalibrated) to determine field area (mm2). Recalibration of field diameter shall be in accordance with NIOSH 7400, Issue 3(100 plus or minus 2 micron). Acceptable range for field area shall be 0.00754 mm2 to 0.00817 mm2. The actual field area shall be documented and used.

7.3.2 Test Variability/Reproducibility

a) Inter-Laboratory Precision. Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that includes participation of at least two (2) other independent laboratories. Each laboratory shall participate in round robin testing at least once every six (6) months with at least all the other laboratories in its inter-laboratory quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed. Analysts shall participate at least annually.

b) Intra- and Inter-Analyst Precision. Each analyst shall select and count a prepared slide from a “reference slide library” on each day on which air counts are performed. Reference slides shall be prepared using well-behaved samples taken from the laboratory workload. Fiber densities shall cover the ranges: 5 – 20; >20 – 50; and >50 – 100 fibers per 100 fields. These slides shall be counted by all analysts to establish an original standard deviation and corresponding limits of acceptability. Results from the daily reference sample analysis shall be compared to the statistically derived acceptance limits using a control chart or a database. It is recommended that the labels on the reference slides be periodically changed so that the analysts do not become familiar with the samples. Intra- and inter-analyst precision may be estimated from blind recounts on reference samples. A laboratory developed analytical uncertainty model from inter - and intra- microscopist precision may also be used. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

7.3.3 Analytical Sensitivity

The normal quantitative working range of the method is 0.04 to 0.5 fiber/ cm^2 for a 1000 L air sample. An ideal counting range on the filter shall be 100 to 1300 fibers/mm^2. The limit of detection (LOD) is estimated to be 5.5 fibers per 100 fields or 7 fibers/mm^2. The LOD in fiber/cc

will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/cm^2 for atmospheres free of interferences.

7.3.4 Data Reporting

7.3.4.1 Airborne fiber concentration in a given sample shall be calculated in accordance with NIOSH 7400, Issue 3, 14 June, 2019, Sections 22 and 23.

7.3.4.2 Measurement Uncertainties. The laboratory shall calculate and report the intra-laboratory and inter-laboratory RSD with each set of results (NIOSH 7400, Issue 3, 14 June 2019, Section 24).

7.3.4.3 Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of the filter area covered with particulate shall be reported as “overloaded”, “uncountable” or “probably biased”. Other fiber counts outside the 100-1300 fibers/mm^2 range shall be reported as having “greater than optimal variability” and as being “probably biased”.

7.3.5 Quality Control

At least two (2) field blanks (or 10% of the total samples, whichever is greater) shall be submitted for analysis with each set of samples. Field blanks shall be handled in a manner representative of actual handling of associated samples in the set with a single exception that air shall not be drawn through the blank sample. A blank cassette shall be opened for approximately thirty (30) seconds at the same time other cassettes are opened just prior to analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters shall be unknown to the counter until all counts have been completed. If a field blank yields greater than seven (7) fibers per one hundred (100) graticule fields, report possible contamination of the samples.

7.3.6 Other Measures

a) Test for Non-Random Fiber Distribution; blind recounts by the same analyst shall be performed on 10% of the filters counted. A person other than the counter shall re-label slides before the second count. A test for type II error shall be performed to determine whether a pair of counts by the same analyst on the same slide shall be rejected due to non-random fiber distribution. If a pair of counts is rejected by this test, the remaining samples in the set shall be recounted and the new counts shall be tested against first counts. All rejected paired counts shall be discarded.

NOTE: It shall not be necessary to use this statistic on blank recounts.

b) All laboratories shall participate in a national sample testing scheme such as the Proficiency Analytical Testing (PAT) program or the Asbestos Analysts Registry (AAR) program, both sponsored by the American Industrial Hygiene Association (AIHA).

7.3.7 Standards and Reagents

Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples shall be utilized as reference samples (refer to Section 7.3.6.b) to standardize the optical system and analyst. All other testing reagents and devices (phase shift test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (NIOSH 7400, Issue 3, 14 June, 2019).

7.4 Polarized Light Microscopy

7.4.1 Calibration

7.4.1.1 Microscope Alignment. To accurately measure the required optical properties, a properly aligned polarized light microscope (PLM) shall be utilized. The PLM shall be aligned before each use.

7.4.1.2 Refractive Index Liquids. Laboratories must have a reference series of n_D = 1.49 through 1.72 in intervals less than or equal to 0.005, as well as refractive index liquids for routine dispersion staining: high-dispersion series 1.550, 1.605, and 1.680. The accurate measurement of the refractive index (RI) of a substance requires the use of calibrated refractive index liquids. These liquids shall be calibrated at first use and semiannually or next use, whichever is less frequent, to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or RI glass beads.

7.4.2 Test Variability/Reproducibility

a) Intra-Analyst Precision. For laboratories that can participate in Inter-Analyst determinations, at least one (1) out of fifty (50) samples shall be re-analyzed by the same analyst. For single analyst laboratories, at least one (1) out of every ten (10) samples shall be re-analyzed by the same analyst.

b) Inter-Analyst Precision. At least one (1) out of fifteen (15) samples shall be re-analyzed by another analyst. Inter-analyst results will require additional re-analysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.

c) Inter-Laboratory Precision. The laboratory shall participate in round robin testing with at least one (1) other laboratory. Samples shall be sent to this other laboratory at least four (4) times per year. These samples shall be samples previously analyzed as QC samples. Results of these analyses shall be assessed in accordance with QC requirements. The QC requirements shall address misclassifications (false positives, false negatives) and misidentification of asbestos types. All analysts shall participate at least annually.

7.4.3 Analytical Sensitivity

The laboratory shall utilize a method that provides a LOD that is appropriate and relevant for the intended use of the data. LOD shall be determined by the protocol in the method or applicable regulation.

7.4.4 Data Reporting

7.4.4.1 The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (40 CFR Part 763, Subpart E, Appendix E).

7.4.4.2 Method Uncertainties. Precision and accuracy shall be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors shall be generated for different concentrations of asbestos.

7.4.5 Quality Control

a) Friable Materials. At least one (1) blank slide shall be prepared daily or with every fifty (50) samples analyzed, whichever is less. This is prepared by mounting a sub-sample of an

isotropic verified non-asbestos-containing material (non-ACM) (e.g., fiberglass in SRM 1866) in a drop of immersion oils normally used on a clean slide, rubbing preparation tools (forceps, dissecting needles, etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip shall be scanned to detect any asbestos contamination. A similar check shall be made after every twenty (20) uses of each piece of homogenization equipment. An isotropic verified non-ACM shall be homogenized in the clean equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this Section.)

b) Non-Friable Materials. At least one (1) non-ACM non-friable material shall be prepared and analyzed with every twenty (20) samples analyzed. This non-ACM shall go through the full preparation and analysis regimen for the type of analysis being performed.

7.4.6 Other Measures

a) Friable Materials. Accuracy cannot be determined by re-analysis of routine field samples. At least one (1) out of one hundred (100) samples shall be a standard or reference sample that has been routinely resubmitted to determine the analyst's precision and accuracy as developed by the laboratory in accordance with quality control requirements. A set of these samples may be accumulated from proficiency testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk materials. At least half of the reference samples submitted for this QC shall contain between 1 and 10% asbestos.

b) Non-Friable Materials. At least one (1) out of one hundred (100) samples shall be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

7.4.7 Standards and Reagents

a) The QC program shall establish and maintain provisions for asbestos standards.

b) Reference standards that are used in an asbestos laboratory shall be obtained from NIST, EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

c) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

d) All reagents used shall be analytical reagent grade or better.

e) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from "look-alikes"; e.g., fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

7.5 Constant and Consistent Test Conditions for Sample Management

7.5.1 Samples shall be transported to the laboratory as soon as possible after collection. Date and time of sampling shall be noted on submittal forms and appropriately documented by the laboratory. The names of the collectors with their signatures and the site shall be included on the chain-of-custody forms. No preservatives are required during sampling.

7.5.2 The laboratory shall establish and adhere to written procedures to minimize the possibility of cross contamination between samples.

7.5.3 Refer to the specific method of analysis for additional requirements.