ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1, MODULE 3

ASBESTOS TESTING

Draft Interim Standard

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

PREFACE

This document is a **Draft Interim Standard** prepared by TNI and is the result of many hours of effort by those volunteers on the TNI Expert Committees. The TNI Board of Directors wishes to thank all of these volunteers for their efforts.

Any TNI member may provide a vote on this Standard to TNI before July 31, 2007.

The TNI Expert Committees will consider all affirmative and negative comments associated with the vote in a public meeting on August 20-24, 2007. After resolving all negative comments, including making changes to the Standard as appropriate, this document will become an Interim Standard to be voted on once again by the TNI membership. After that vote is final and any appeals are considered, the document will be released as a Final Standard for other organizations to use as appropriate

More details about this process can be found in *Policies Governing Standards Development,* published on the TNI Web site.

VOLUME 1, MODULE 3

Asbestos Testing

Table of Contents

VOLUME 1, MODULE 3

Asbestos Testing

1.0 ASBESTOS TESTING

1.1 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.

1.2 Scope

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

1.3 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.

1.3.1 Additional Terms and Definitions

Reserved

1.3.2 Exclusions and Exceptions

Reserved

1.4 Method Selection

A standard method is a test method issued by an organization generally recognized as competent to do so. When a laboratory is required to analyze a parameter by a specified method due to a regulatory requirement, the parameter/method combination is recognized as a standard method. If there is not a regulatory requirement for the parameter/method combination, the parameter/method combination is recognized as a standard method if it can be analyzed by another similar standard method of the same matrix and technology.

The inclusion of the parameter in the method shall meet all required calibration requirements of the method and the quality control requirements of the method to which the parameter is being added. If no QC exists in the method, the laboratory shall adhere to the requirements outlined in the similar method. A method that meets the above requirement shall be identified in such a way that there is no confusion that the method has been modified.

When it is necessary to use methods not covered by standard 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.

1.5 Method Validation

Validation is the confirmation, by examination and objective evidence, that the particular requirements for a specific intended use are fulfilled. The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

Laboratories shall participate in Proficiency Testing programs. The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.

1.6 Demonstration of Capability

1.6.1 General

Prior to acceptance and institution of any method for data reporting, satisfactory demonstration of method capability is required (see Section 1.6.2).

Thereafter, ongoing demonstration of method performance (section 1.6.3), as per the quality control requirements in Section 1.7.3 (such as laboratory control samples), is required.

In cases where a laboratory analyzes samples using a method that has been in use by the laboratory for at least one year prior to applying for accreditation, and there have been no significant changes in instrument type, personnel or method, the continuing demonstration of method performance and the analyst's documentation of continued proficiency shall be acceptable. The laboratory shall have records on file to demonstrate that a demonstration of capability is not required.

For the initial demonstration of capability, appropriate records as discussed in Section 1.6.2 shall be completed and retained by the laboratory to be made available upon request. Data to support the continuing demonstration of capability shall be retained by the laboratory.

A demonstration of capability shall be completed each time there is a change in instrument type, personnel, or method.

1.6.2 Demonstration of Capability

A demonstration of capability (DOC) shall be conducted prior to using any test method and at any time there is a change in instrument type, personnel or test method or any time that a method has not been performed by the laboratory or analyst in a twelve-month period.

All initial demonstrations shall be documented. All data applicable to the demonstration shall be retained and available at the laboratory.

1.6.2.1 Initial Demonstration of Capability Documentation

a) The laboratory shall document each initial demonstration of capability in a manner such that the following information is readily available for each affected employee:

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

   ii. matrix;

   iii. analyte(s), class of analyte(s), or measured parameter(s);

   iv. identification of test method(s) performed;

v. identification of laboratory-specific standard operating procedure (SOP) used for analysis, including revision number;

vi. date(s) of analysis; and

vii. summary of analyses, including information outlined in 1.6.2.2.d.

1.6.2.2 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) A quality control sample shall either be obtained from an outside source or be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) 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 test method) sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration of one to four times the limit of quantitation.

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

d) 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 parameter 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.

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

f) When one or more of the tested parameters 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 parameters of interest beginning with c) above.

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

g) 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 c).

h) When an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited test method, an initial demonstration shall be performed for that analyte.

1.6.3 Ongoing Demonstration of Capability

1.6.3.1 The laboratory shall have a documented procedure describing ongoing demonstration of capability. The analyst(s) shall demonstrate ongoing capability by meeting the quality control requirements of the method, laboratory SOP, client specifications, and/or this Standard. It is the responsibility of the laboratory to document that other approaches to Ongoing Demonstration of Capability are adequate.

1.6.3.2 For asbestos, this ongoing demonstration may be one of the following:

a) acceptable performance of a blind sample (single blind to the analyst). Note: successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5030/8260) would only require documentation for one of the tests;

b) another demonstration of capability;

c) at least four consecutive laboratory control samples 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 4 consecutive passing laboratory control samples (LCS) for each method for each analyst each year;

d) a documented process of analyst review using quality control (QC) samples can serve as the annual ongoing demonstration of capability. QC samples can be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary; or

e) if a) through d) are not technically feasible, then analysis of real-world samples with results within predefined acceptance criteria (as defined by the laboratory or method) shall be performed.

1.7 Technical Requirements

1.7.1 Calibration

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.

1.7.1.1 Transmission Electron Microscopy

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

1.7.1.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) shall be performed under the same analytical conditions used for routine asbestos analysis and must be recorded in a notebook and include date and analyst's signature. 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 logbook 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 or camera film. This must also allow accurate (<5% variation) measurement of zone axis SAED patterns on permanent media (e.g., film). Calibrations shall be performed monthly 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 250 nm 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 >1 µm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 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.

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 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. 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.

f) Low Temperature Asher. The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-celluloseester (MCE) filters. Calibration data shall be displayed on control charts that show trends over time.

g) Grid Openings. The magnification of the grid opening measurement system shall be calibrated using an appropriate standard at a frequency of 20 openings/20 grids/lot of 1000 or 1 opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

1.7.1.1.2 Air

All calibrations shall be performed in accordance with Section 1.7.1.1.1, with the exception of magnification. 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 15,000 to 20,000x. A logbook shall be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

1.7.1.1.3 Bulk Samples

All calibrations shall be performed in accordance with Section 1.7.1.1.1.

1.7.1.2 Phase Contrast Microscopy

1.7.1.2.1 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.

1.7.1.2.2 The phase-shift detection limit of the microscope shall be checked monthly or after modification or relocation using an HSE/NPL phase-contrast test slide for each analyst/microscope combination. This procedure assures that the minimum detectable fiber diameter (<ca. 0.25µm) for this microscope is achieved.

1.7.1.2.3 Prior to ordering the Walton-Beckett graticule, calibration, in accordance with NIOSH 7400, Issue 2, 15 August 1994, 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 must 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 also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 mm2 to 0.00817 mm2. The actual field area shall be documented and used.

1.7.1.3 Polarized Light Microscopy

1.7.1.3.1 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.

1.7.1.3.2 Refractive Index Liquids. Series of n_D = 1.49 through 1.72 in intervals less than or equal to 0.005. Refractive index liquids for dispersion staining, high-dispersion series 1.550, 1.605, 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.

1.7.2 Quality Control

1.7.2.1 Negative Controls

1.7.2.1.1 Transmission Electron Microscopy

a) Water and Wastewater

i. Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each twenty-four shall be tested for background level. When using glass bottles, four bottles from each twenty-four shall be tested. An acceptable bottle blank level is defined as < 0.01 million fibers per liter (MFL) > 10 µm.

ii. A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be > 10 mL for a 25-mm diameter filter and > 50 mL for a 47-mm diameter filter.

b) Air

i. A blank filter shall be prepared with each set of samples. A 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 blank filter shall be analyzed for each twenty samples analyzed.

ii. Maximum contamination on a single blank filter shall be no more than 53 structures/mm2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm2.

c) Bulk Samples

i. 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 should have a set of reference asbestos materials from which a set of reference diffraction and x-ray spectra have been developed.

1.7.2.1.2 Phase Contrast Microscopy

At least two 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 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 should be unknown to the counter until all counts have been completed. If a field blank yields greater than seven fibers per one-hundred graticule fields, report possible contamination of the samples.

1.7.2.1.3 Polarized Light Microscopy

a) Friable Materials. At least one blank slide shall be prepared daily or with every fifty 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 oil (nD should reflect usage of various nD's) 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 must be made after every twenty uses of each piece of homogenization equipment. An isotropic verified non-ACM must 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 non-ACM non-friable material shall be prepared and analyzed with every twenty samples analyzed. This non-ACM must go through the full preparation and analysis regimen for the type of analysis being performed.

1.7.3 Test Variability/Reproducibility

1.7.3.1 Transmission Electron Microscopy

Quality assurance analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random and samples of special interest may be included in the selection of samples for quality assurance analyses. When possible, the checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed prior to internal or external audits. It is recommended that a laboratory initially be at 100% quality control (all samples re-analyzed). The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%.

1.7.3.1.1 Water and Wastewater

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 QC 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. 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 per one-hundred samples.

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 per one-hundred samples.

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 sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of 1 per 20 samples. Qualified analysts shall maintain an average of ≥ 80% true positives, ≤ 20% false negatives, and ≤ 10% false positives.

1.7.3.1.2 Air

a) All analyses shall be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings.

b) The laboratory and TEM analysts shall obtain mean analytical results on NIST SRM 1876b 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 1.7.3.1.1.c, Verified Analysis, below. SRM 1876b 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 standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

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 per two-hundred grid square analyses for clients.

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

i. Replicate. A second, independent analysis shall be performed in accordance with Section 1.7.3.1.1.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 per one-hundred samples.

iii. Verified Analyses. A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section 1.7.3.1.1.c.

1.7.3.1.3 Bulk Samples

Because bulk samples with low (< 10%) asbestos content are the most problematic, a laboratory's quality control program should focus on such samples. At least 30% of a laboratory's QC analyses shall be performed on samples containing from 1% to 10% asbestos.

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

b) Inter-Analyst Precision. At least one out of fifteen samples must 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 other laboratory. Samples shall be sent to this other lab at least four 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.

1.7.3.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision. Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance (QA) program that includes participation of at least two other independent laboratories. Each laboratory shall participate in round robin testing at least once every six 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.

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 entire range routinely analyzed by the laboratory. 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. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

1.7.3.3 Polarized Light Microscopy

Refer to Section 1.7.3.1.3

1.7.4 Other Quality Control Measures

1.7.4.1 Transmission Electron Microscopy

a) Water and Wastewater

i. Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between one and twenty structures (> 10µm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of one per one-hundred samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

ii. NIST SRM 1876b 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 asbestos types in accordance with Section 1.7.4.1.a)i.

ii. NIST SRM 1876b shall be analyzed annually.

c) Bulk Samples

All analysts shall be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six asbestos types listed are available from NIST (SRMs 1866 and 1867).

1.7.4.2 Phase Contrast Microscopy

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 should 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 should 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.

b) It shall not be necessary to use this statistic on blank recounts.

c) 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).

1.7.4.3 Polarized Light Microscopy

a) Friable Materials. Because accuracy cannot be determined by re-analysis of routine field samples, at least one out of one-hundred samples shall be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should 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 out of one-hundred samples shall be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

1.7.5 Analytical Sensitivity

1.7.5.1 Transmission Electron Microscopy

1.7.5.1.1 Water and Wastewater

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).

1.7.5.1.2 Air

An analytical sensitivity of 0.005 structures/cm2 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.

1.7.5.1.3 Bulk Samples

a) The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001%.

b) There should be an error rate of less than 1% on the qualitative analysis for samples that contain 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.

1.7.5.2 Phase Contrast Microscopy

The normal quantitative working range of the test 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/ cm^2 will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/cm^2 for atmospheres free of interferences.

1.7.5.3 Polarized Light Microscopy

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

1.7.6 Quality of Standards and Reagents

1.7.6.1 Transmission Electron Microscopy

a) The quality control 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 at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

1.7.6.2 Phase Contrast Microscopy

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 such as PAT and AAR samples available from the AIHA may be utilized as reference samples (refer to Section D.6.2.2 b) to standardize the optical system and analyst. All other testing reagents and devices (HSE/NPL test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (refer to NIOSH 7400, Issue 2, 15 August 1994).

1.7.6.3 Polarized Light Microscopy

Refer to Section 1.7.6.1.

1.7.7 Data Acceptance/Rejection Criteria

1.7.7.1 Transmission Electron Microscopy

1.7.7.1.1 Water and Wastewater

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.

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.

1.7.7.1.2 Air

a) The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized, (e.g., AHERA).

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.

1.7.7.1.3 Bulk Samples

a) The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993).

b) 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.

1.7.7.2 Phase Contrast Microscopy

1.7.7.2.1 Airborne fiber concentration in a given sample shall be calculated in accordance with NIOSH 7400, Issue 2,15 August 1994, Sections 20 and 21.

1.7.7.2.2 Measurement Uncertainties. The laboratory shall calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results. (NIOSH 7400, Issue 2, 15 August 1994)

1.7.7.2.3 Fiber counts above 1300 fibers/mm2 and fiber counts from samples with >50% of the filter area covered with particulate shall be reported as “uncountable” or “probably biased.” Other fiber counts outside the 100-1300 fibers/mm2 range shall be reported as having “greater than optimal variability” and as being “probably biased.”

1.7.7.3 Polarized Light Microscopy

1.7.7.3.1 The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993).

1.7.7.3.2 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 should be generated for different concentrations of asbestos.

1.7.8 Constant and Consistent Test Conditions Sample and Sampling Requirements

1.7.8.1 Samples shall be transported to the laboratory as soon as possible after collection. Date and time of sampling shall be noted on submittal forms. 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.

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

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