NIST HANDBOOK 150-3
2006 Edition

National Voluntary Laboratory Accreditation Program

BULK ASBESTOS ANALYSIS

Hazel M. Richmond

National Voluntary Laboratory Accreditation Program
Standards Services Division
Technology Services

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U.S. Department of Commerce
Carlos M. Gutierrez, Secretary

National Institute of Standards and Technology
William A. Jeffrey, Director

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Foreword

The NIST Handbook 150 publication series sets forth the procedures, requirements, and guidance for the accreditation of testing and calibration laboratories by the National Voluntary Laboratory Accreditation Program (NVLAP). The series is comprised of the following publications:

- NIST Handbook 150, *NVLAP Procedures and General Requirements*, which contains the general procedures and requirements under which NVLAP operates as an unbiased third-party accreditation body;

- NIST Handbook 150-xx program-specific handbooks, which supplement NIST Handbook 150 by providing additional requirements, guidance, and interpretive information applicable to specific NVLAP laboratory accreditation programs (LAPs).

The program-specific handbooks are not stand-alone documents, but rather are companion documents to NIST Handbook 150. They tailor the general criteria found in NIST Handbook 150 to the specific tests, calibrations, or types of tests or calibrations covered by a LAP.

NIST Handbook 150-3, *NVLAP Bulk Asbestos Analysis*, presents the technical requirements and guidance for the accreditation of laboratories under the NVLAP Bulk Asbestos Analysis LAP. The 2006 edition incorporates changes resulting from the release of the newest editions of ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*, and NIST Handbook 150, as well as editorial improvements. The 2006 edition of NIST Handbook 150-3 supersedes and replaces the 1994 edition.

The handbook was revised with the participation of technical experts in the field of bulk asbestos testing and was approved by NVLAP. The following main changes have been made to this handbook with respect to the previous edition:

- all references to applicable international guides and standards have been updated;

- Laboratory Bulletins (LB-2-1998 and LB-3-1999), previously issued for clarification, have been incorporated into this edition of the handbook;

- on-site assessment checklists and the test method selection list are no longer included in order that they may be provided as separate documents, which may be updated at different intervals than the handbook;

- the body of the handbook has been restructured to conform with internationally accepted rules for the structure and drafting of standards, where appropriate, to promote ease of use and understanding;

- Annex A, *Documents required in the laboratory*, has been added.

This handbook is also available on the NVLAP web site (http://www.nist.gov/nvlap).

Questions or comments concerning this handbook should be submitted to NVLAP, National Institute of Standards and Technology, 100 Bureau Drive, Stop 2140, Gaithersburg, MD, 20899-2140; phone: 301-975-4016; fax: 301-926-2884; e-mail: nvlap@nist.gov.

Introduction

The Asbestos Hazard Emergency Response Act (AHERA) was enacted in October 1986. AHERA requires laboratories that analyze asbestos bulk insulation samples taken from public, or private, elementary or secondary schools, to be accredited by the National Institute of Standards and Technology (formerly the National Bureau of Standards) National Voluntary Laboratory Accreditation Program (NVLAP). NVLAP accredits laboratories for the 1982 procedure, *Interim Method for the Determination of Asbestos in Bulk Insulation Samples*, found in 40 CFR Part 763, Appendix E to Subpart E (formerly Appendix A to Subpart F). AHERA requires that two types of asbestos analysis laboratories be accredited by NVLAP: those performing analysis using polarized light microscopy (PLM) and those performing analysis using transmission electron microscopy (TEM).

To meet the need for an accrediting mechanism in the period before the implementation of the NIST/NVLAP Program, the U. S. Environmental Protection Agency (EPA) Bulk Sample Analysis Quality Assurance Program was continued as the Interim Asbestos Bulk Sample Analysis Quality Assurance Program. This program provided a source of accredited laboratories until NVLAP-accredited laboratories were available. PLM laboratories accredited in the April 1988 round of testing received EPA interim accreditation until January 12, 1989. However, NVLAP did not begin to accredit laboratories until April 1989. To provide a source of accredited laboratories after January 12, 1989, EPA developed a process for extending interim accreditation. Under this process, a laboratory received an extension if it had fully applied to NVLAP by September 30, 1988, and NVLAP had not yet completed its evaluation. EPA interim accreditation was revoked, however, if the laboratory failed NVLAP proficiency testing and/or the on-site assessment.

All extensions granted by EPA expired on October 30, 1989. After this date, PLM laboratories analyzing bulk samples taken from public, or private, elementary or secondary schools, were required to be accredited by NVLAP.

In 1993, the EPA developed an improved method entitled *Method for the Determination of Asbestos in Bulk Building Materials*, EPA/600/R-93/116. The test method provides clarifications and improvements to the 1982 method and is recommended for use as the preferred substitute method. Use of the improved method can provide more precise analytical results at low asbestos concentrations, enhanced analysis of floor tiles that may contain thin asbestos fibers below the limits of resolution of the PLM, and clearer instruction on the analysis of bulk materials, particularly where multiple layers are present. While the improved method is recommended by the EPA to be used in place of the 1982 procedure, and serve as the preferred substitute method, it has not been designated as the new interim method. NVLAP laboratories that use the EPA/600/R-93/116 method must indicate it on their test reports.

The technical content of this handbook was originally developed by the Surface and Microanalysis Science Division of the National Institute of Standards and Technology and was updated in 1994 to reflect new requirements and information.

For this edition of the handbook, special thanks for their contributions go to NVLAP assessors Peter Cooke, Steve Lerman, Thomas Emma, Charles Knowles, Wade Mullin, Larry Pierce, Shu-Chun Su, and Gerald Wright.

1 General information

1.1 Scope

1.1.1 This handbook specifies the technical requirements and provides guidance for the accreditation of laboratories under the NVLAP Bulk Asbestos Analysis Program (PLM Program). It supplements the NVLAP procedures and general requirements found in NIST Handbook 150, by tailoring the general criteria found in NIST Handbook 150 to the specific tests covered by the PLM Program.

1.1.2 NIST Handbook 150, this handbook, and their respective checklists (see 1.6) constitute the collective body of requirements that must be met by a laboratory seeking NVLAP accreditation for the PLM Program. These requirements include satisfactory performance in selected proficiency testing, as required, and fulfilling the on-site assessment requirements, including resolution of identified nonconformities.

1.1.3 This handbook is intended for information and use by accredited PLM laboratories, assessors conducting on-site assessments, laboratories seeking accreditation, other laboratory accreditation systems, users of laboratory services, and others needing information on the accreditation requirements under the PLM Program.

1.1.4 Any laboratory (including commercial, manufacturer, university, or federal, state, or local government laboratory) that performs the PLM test method may apply for NVLAP accreditation.

1.2 Organization of handbook

The numbering and titles of the first five clauses of this handbook match those of NIST Handbook 150. The primary subclauses in clauses 4 and 5 (e.g., 4.1, 4.2, etc.) are also numbered and titled to correspond with those of NIST Handbook 150, even when there are no requirements additional to those in NIST Handbook 150.

Annex A provides a list of documents that are required to be available in a laboratory accredited under the PLM Program.

1.3 Program description

The purpose of the PLM Program is to accredit testing laboratories to provide assurance that they are competent to analyze bulk samples for asbestos using polarized light microscopy (PLM). Public Law 99-519, *Asbestos Hazard Emergency Response Act of 1986*, referred to as AHERA, requires the National Institute of Standards and Technology (formerly the National Bureau of Standards) to develop an accreditation program for laboratories conducting analyses of bulk samples of asbestos-containing materials.

1.4 References

The following documents are referenced in this handbook. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any

amendments) shall apply within one year of publication or within another time limit specified by regulations or other requirement documents.

— NIST Handbook 150, *NVLAP Procedures and General Requirements*

— U.S. Environmental Protection Agency *Interim Method for the Determination of Asbestos in Bulk Insulation Samples* as found in 40 CFR, Part 763, Subpart E, Appendix E (formerly Subpart F, Appendix A), EPA/600/M4-82-020, 1982, or the current U.S. Environmental Protection Agency method for the analysis of asbestos in building material

— U.S. Environmental Protection Agency *Method for the Determination of Asbestos in Bulk Building Materials* (EPA/600/R-93/116), 1993, R. L. Perkins and B. W. Harvey

— NISTIR 5951, *Guide for the Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1*, Jennifer R. Verkouteren and David L. Duewer

— *Guide to Statistical Analysis of Bulk Asbestos Quality Control Data*, Steve Lerman (available from NVLAP upon request)

1.5 Terms and definitions

For the purposes of this handbook, the terms and definitions given in NIST Handbook 150 apply and the following apply:

1.5.1
asbestos
A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), asbestiform anthophyllite, asbestiform tremolite, and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability; and 5) high heat resistance.

1.5.2
Becke line
A band of light seen at the periphery of a specimen when the refractive indices of the specimen and the mounting medium are different; it is used to determine refractive index.

1.5.3
bias
A systematic error characterized by a consistent (non-random) measurement error.

1.5.4
binder
With reference to a bulk sample, a component added for cohesiveness (e.g., plaster, cement, glue, etc.).

1.5.5
birefringence
The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated using a Michel-Levy chart from the interference colors observed under crossed polars. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below.

Qualitative	*Quantitative (N-n)*
none	0.00 or isotropic
low	$\leq$ 0.010
moderate	0.011-0.050
high	> 0.050

1.5.6
bulk sample
A sample of building material taken for the identification and quantification of asbestos. Bulk building materials may include a wide variety of friable and non-friable materials.

1.5.7
color
The color of a particle or fiber when observed under low-power magnification using top-lighting.

1.5.8
compensator
A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530 nm to 550 nm retardation).

1.5.9
control chart
A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

1.5.10
detection limit
The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

1.5.11
dispersion staining (focal masking)
An optical means of imparting apparent or virtual color to transparent substances by the use of an opaque screen (stop) in the objective back focal plane; it is used to determine refractive indices.

1.5.12
extinction
The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique (inclined), symmetrical and undulose.

1.5.13
extinction angle
For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

1.5.14
fiber
With reference to asbestiform morphology: a structure consisting of one or more fibrils.

1.5.15
friable
Refers to the cohesiveness of a bulk material, indicating that it may be crumbled or disaggregated by hand pressure.

1.5.16
gravimetry
Any technique in which the concentration of a component is determined by weighing. As used in this document, it refers to the measurement of asbestos-containing residues after sample treatment by ashing, dissolution, etc.

1.5.17
homogeneous
Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

1.5.18
matrix
Nonasbestos, nonbinder components of a bulk material; for example, such components as cellulose, fiberglass, mineral wool, mica, etc.

1.5.19
Michel-Levy scale of retardation colors
A chart plotting the relationship between birefringence, retardation and thickness of anisotropic substances. Any one of the three variables can be determined if the other two are known.

1.5.20
morphology
The structure and shape of a particle. Characterization may be descriptive (platy, rod-like, acicular, etc.) or in terms of dimensions such as length and diameter (see asbestiform).

1.5.21
oblique illumination
Illumination by light that is at an oblique angle to the optical axis.

1.5.22
pleochroism
The change in color or hue of a colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

1.5.23
point counting
A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to nonasbestos sample components.

1.5.24
refractive index (index of refraction)
The ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as *n* and varies with wavelength and temperature.

1.5.25
sign of elongation
Referring to the location of the high and low refractive indices in an elongated anisotropic substance, a specimen is described as positive when the higher refractive index is lengthwise (length slow), and as negative when the lower refractive index is lengthwise (length fast).

1.5.26
trace
The quantity of asbestos that is above the laboratory's detection limit and below its limit of quantification.

1.5.27
visual estimation
An estimation of the concentration of asbestos in a sample as compared to other sample components.

1.6 Program documentation

1.6.1 General

Assessors use NVLAP checklists to ensure that each laboratory receives an assessment comparable to that received by others. Checklists assist assessors in documenting the assessment to the NVLAP requirements found in NIST Handbook 150, this handbook, and the checklists themselves. Checklists contain definitive statements or questions about all aspects of the NVLAP criteria for accreditation, and form part of the On-Site Assessment Report (see NIST Handbook 150). The current version of each checklist is available on the NVLAP web site <http://www.nist.gov/nvlap>.

1.6.2 NIST Handbook 150 Checklist

All NVLAP programs use the NIST Handbook 150 Checklist (formerly called the General Operations Checklist), which contains the requirements published in NIST Handbook 150. The checklist items are numbered to correspond to clauses 4 and 5 and annexes A and B of NIST Handbook 150.

1.6.3 NIST Handbook 150-3 Checklist

The NIST Handbook 150-3 Checklist (also referred to as the Bulk Asbestos Program-Specific Checklist) addresses the requirements specific to bulk asbestos analysis testing given in NIST Handbook 150-3. The checklist may contain requirements expressed at a more detailed level than found in this handbook.

1.6.4 NVLAP Lab Bulletins

NVLAP Lab Bulletins are issued to laboratories and assessors, when needed, to clarify program-specific requirements and to provide information about program additions and changes.

2 LAP establishment, development and implementation

This clause contains no information additional to that provided in NIST Handbook 150, clause 2.

3 Accreditation process

3.1 General

An overview of the laboratory accreditation process is provided in NIST Handbook 150, clause 3, and includes information pertaining to application for accreditation; on-site assessment; proficiency testing; accreditation decision; granting accreditation; renewal of accreditation; changes to scope of accreditation; monitoring visits; and suspension, denial, revocation, and voluntary termination of accreditation.

3.2 Management system review

3.2.1 Prior to applying to NVLAP for accreditation, a laboratory shall have a fully implemented management system.

3.2.2 The NVLAP assessor assigned to conduct the on-site assessment will request a copy of the laboratory's management system manual and relevant documented procedures in advance of the assessment to reduce time at the laboratory.

3.2.3 The assessor will review all relevant management system documentation for conformity with NVLAP requirements, including the requirements of NIST Handbook 150 and this handbook. During this review, the assessor may request additional management system documents and/or records, which will be returned upon request.

3.3 On-site assessment

3.3.1 The purpose of the on-site assessment is to determine if the laboratory is following its documented management system and to assess the competence of the laboratory's delivery of its testing services. The laboratory shall be prepared for conducting test demonstrations, have equipment in good working order, and be ready for examination according to the requirements identified in this handbook, NIST Handbook 150, and the laboratory's management system manual. The assessor will need time and workspace to complete assessment documentation while at the laboratory.

3.3.2 An opening meeting is conducted with the laboratory manager to explain the purpose of the on-site visit and to discuss the schedule for the day(s). At the discretion of the laboratory manager, other staff may attend the meeting.

3.3.3 At least one staff member shall be available to answer questions; however, the assessor may wish to review the documents and records alone. The assessor usually does not ask to remove any laboratory documents or records from the laboratory premises.

3.3.4 The results of proficiency testing shall be made available to the on-site assessor for use during the on-site assessment.

3.3.5 The assessor physically examines equipment and facilities, and observes and interviews personnel who demonstrate selected procedures. The demonstrations requested may be selective or all-inclusive, and shall include sample test material(s), preparation devices, establishment of test conditions and the setup/use of major equipment. The assessor may provide a proficiency test sample and request a specific demonstration.

3.3.6 The assessor completes an On-Site Assessment Report, which clearly lists all nonconformities and comments (both positive and negative).

3.3.7 The assessor conducts a closing meeting, attended by appropriate laboratory personnel, to discuss the findings, including comments and nonconformities. The first page of the report shall be signed by the assessor and the laboratory's Authorized Representative to acknowledge the discussion, but does not necessarily indicate agreement; appeals may be made through NVLAP.

3.3.8 The information obtained by the assessor is held in the strictest confidence.

3.4 Proficiency testing

3.4.1 NIST Handbook 150 defines proficiency testing and describes how it is included in the accreditation process.

3.4.2 Proficiency testing is required for all laboratories before initial accreditation can be granted and for maintaining accreditation in the bulk asbestos program.

3.4.3 The proficiency testing materials, in general, are representative examples of bulk insulation materials or their derivatives. The goal of the program is to provide material that will test the laboratory's ability to follow the test method and to achieve the proper accuracy, precision, and detection limits.

3.4.4 The proficiency test contractor will send participating laboratories test samples, data sheets, and instructions for performing the test and reporting the results. The test shall be conducted in accordance with EPA/600/M4-82-020, 1982, or the current U.S. Environmental Protection Agency method for the analysis of asbestos in building material, using the laboratory's normal operating procedures.

3.4.5 Proficiency testing shall not be contracted out to another laboratory.

NOTE A laboratory that subcontracts proficiency testing to another laboratory will be immediately suspended for not participating in the test round and risks revocation of its accreditation. (See also 3.4.13.)

3.4.6 Special instructions shall also be followed as they are designed to ensure uniformity in procedures among participants. Completed data sheets shall be returned to NVLAP or its designated contractor for analysis by the date specified on the data sheets. Failure to return the proficiency testing data sheets by the deadline date will result in penalties, which may include failing that round (see 3.4.11).

3.4.7 Proficiency testing may involve materials or artifacts that shall be returned to NVLAP for use by other participants. These materials shall be protected from damage both in the laboratory and during shipment back to NVLAP, or its designated contractor. Examples of such materials and artifacts are: permanently mounted slides, photographs, glasses, and special optical materials. These materials may be used to determine testing performance for specific subparts of the test method. Unless otherwise noted, laboratories should use proficiency test materials for in-house instructional materials.

3.4.8 Occasionally, laboratories may be sent blind samples to test their proficiency under normal conditions. The results of any blind testing will be used to determine a laboratory's continued compliance with NVLAP requirements.

3.4.9 All analysts, including those in sub-facilities, shall participate in proficiency testing. Each analyst shall separately analyze, record, and report test results. The laboratory shall report one result to NVLAP. The test results shall be used for inter-analyst comparisons and entered into the management system records.

3.4.10 NVLAP will report proficiency test performance to participants in appropriate documents. The identity and performance of individual laboratories will remain confidential. Any problems indicated by proficiency testing shall be discussed with appropriate laboratory personnel, who shall then be responsible for developing and implementing plans for resolution. The satisfactory resolution of proficiency testing nonconformities is required.

3.4.11 If an accredited laboratory fails a round of proficiency testing, it shall do the following to maintain its accreditation:

a) provide to NVLAP within 30 days of notification of the failure, detailed, written documentation that includes an analysis of why the laboratory failed, and what corrective action(s) it has taken (analyst training, revised procedures, quality assurance activities, etc.) to resolve its analytical problem(s) to avoid similar errors in the future;

b) provide documentation to show that the corrective action(s) has been effectively implemented;

c) pass the next round of proficiency testing.

3.4.12 If an accredited laboratory fails any two rounds of proficiency testing within a set of three consecutive rounds, its accreditation will be immediately suspended. To regain accreditation, the laboratory may undergo a complete on-site assessment and proficiency test to determine the cause of the nonconformities, and within 30 days provide documentation to NVLAP to demonstrate that effective corrective actions have been implemented. Failure to perform satisfactorily at the on-site assessment will result in continued suspension of the accreditation.

The full cost of any reassessment shall be paid in advance. NVLAP staff will make every effort to expedite these extraordinary assessments to give a laboratory every reasonable opportunity to demonstrate competence to perform the test method and regain accreditation.

3.4.13 Failure to participate in a round of proficiency testing will result in the immediate suspension of accreditation. To have the accreditation reinstated, the laboratory shall pass the next regularly scheduled round of proficiency testing.

4 Management requirements for accreditation

4.1 Organization

4.1.1 The laboratory shall be organized so that staff members are not subjected to undue pressure or inducement that might influence their judgment or the results of their work.

4.1.2 A laboratory operating as a sub-facility shall be technically dependent on the main facility. The main facility shall provide technical management and supervision in accordance with the following requirements.

a) The nature, scope, and frequency of on-site quality assurance reviews by the main facility quality manager shall be clearly defined in the management system and be appropriate for the nature and scope of work performed by the sub-facility.

b) Copies of all permanent quality assurance and personnel records shall be retained at the main facility.

c) Quality assurance data from each sub-facility shall be frequently and routinely compared both to the main facility's data and data from other sub-facilities.

d) Records of such comparisons shall be retained in quality assurance records along with actions taken to evaluate and resolve nonconformities.

4.1.3 NVLAP accreditation of a laboratory main facility does not extend to accreditation of sub-facilities unless the sub-facilities have been evaluated separately. These facilities are uniquely identified in the NVLAP accreditation documents.

4.2 Management system

There are no requirements additional to those set forth in NIST Handbook 150.

4.3 Document control

There are no requirements additional to those set forth in NIST Handbook 150.

4.4 Review of requests, tenders and contracts

There are no requirements additional to those set forth in NIST Handbook 150.

4.5 Subcontracting of tests and calibrations

4.5.1 A laboratory that subcontracts its AHERA work shall ensure that the subcontracted laboratory is accredited by NVLAP for bulk asbestos analysis. [See also NIST Handbook 150, 5.10.2 b), 5.10.6, and Annex A, A.1 i).]

4.5.2 A NVLAP-accredited laboratory shall not represent test data produced at a non-accredited sub-facility as having been produced by an accredited laboratory.

4.6 Purchasing services and supplies

There are no requirements additional to those set forth in NIST Handbook 150.

4.7 Service to the customer

There are no requirements additional to those set forth in NIST Handbook 150.

4.8 Complaints

There are no requirements additional to those set forth in NIST Handbook 150.

4.9 Control of nonconforming testing and/or calibration work

There are no requirements additional to those set forth in NIST Handbook 150.

4.10 Improvement

There are no requirements additional to those set forth in NIST Handbook 150.

4.11 Corrective action

There are no requirements additional to those set forth in NIST Handbook 150.

4.12 Preventive action

There are no requirements additional to those set forth in NIST Handbook 150.

4.13 Control of records

The period of retention of records shall be three years, unless a longer period is required by the client, regulation, or the laboratory's own procedures. The records to be maintained shall include:

a) sample custody records;

b) original data collected, signed (or initialed), and dated by analyst;

c) contamination monitoring data;

d) calibration and verification data;

e) data and results of quality control;

f) equipment and maintenance records;

g) test reports;

h) records of proficiency testing results for each analyst.

4.14 Internal audits

4.14.1 The internal audit shall cover compliance with NVLAP, laboratory management system, regulatory, contractual, and testing requirements.

4.14.2 An applicant laboratory shall conduct at least one complete internal audit prior to the first on-site assessment. The records will be reviewed by the NVLAP assessor before or during the on-site assessment visit.

4.14.3 For accredited laboratories, internal audit reports conducted since the previous on-site assessment shall be made available for review.

4.14.4 Internal audits are separate and distinct from management reviews (see 4.15).

4.15 Management reviews

4.15.1 Periodic reviews of the management system shall reflect adherence to NVLAP requirements and the laboratory's quality objectives.

4.15.2 Management reviews shall review all nonconformities and may reflect positive aspects of the management system.

4.15.3 An applicant laboratory shall perform at least one complete management review prior to the first on-site assessment. The records will be reviewed by the NVLAP assessor before or during the on-site assessment visit.

4.15.4 The report of the management review shall be available during the NVLAP on-site assessment.

5 Technical requirements for accreditation

5.1 General

There are no requirements additional to those set forth in NIST Handbook 150.

5.2 Personnel

5.2.1 The laboratory shall have a detailed, documented description of its training program for new and current staff members. The analytical results obtained by new staff members shall be checked by an

analyst whose performance has been demonstrated to be acceptable, or by using an independent technique, until the new staff member demonstrates the required level of performance. The laboratory shall establish and document performance criteria to determine when a new analyst is qualified to work independently. Reference documents, texts, journals, and current scientific and industry periodicals shall be made available to all analysts to keep their knowledge up to date and shall include, as a minimum, the documents listed in Annex A. An ongoing process of practice, training and professional development is essential to the maintenance and improvement of analyst expertise.

5.2.2 A laboratory analyst shall be able to obtain enough information from the laboratory's quality documentation to perform analyses in the absence of the laboratory manager. Specific evidence that all staff members have been trained for their role in the quality assurance program is required.

5.2.3 All analysts and technical supervisors shall understand polarized light microscopy and its application to crystalline materials. They shall understand what measurements are possible with the PLM, how they are performed, and how to form conclusions about the identity of a component from the measurements. They shall be able to measure all optical properties required for the identification of regulated asbestos types, and in particular, the index of refraction by the immersion method. These measurements shall be satisfactorily performed, upon request, during an on-site visit by a NVLAP assessor. Knowledge of several techniques of refractive index measurement (e.g., Becke line, dispersion staining, and oblique illumination), in addition to other analytical methods (e.g., x-ray diffraction, transmission electron microscopy, and scanning electron microscopy) are also helpful.

5.3 Accommodation and environmental conditions

5.3.1 The laboratory shall have the proper facilities to perform asbestos analysis, to store asbestos in accordance with federal, state and local laws, and to maintain sample integrity.

5.3.2 The workspace shall be monitored for asbestos contamination on a routine basis. Laboratory blanks, using asbestos-free materials, shall be prepared and analyzed with sufficient frequency to detect contamination of laboratory equipment or supplies, including, but not restricted to, glass slides, cover slips, refractive index liquids, sampling instruments, analytical instruments (microscopes), workstations, and cleaning fluids. There shall be written procedures for dealing with any contamination.

5.3.3 Safe working conditions shall be maintained while handling bulk asbestos.

5.4 Test and calibration methods and method validation

5.4.1 General

The laboratory's accreditation is limited to the test method contained in the U.S. EPA Interim Method, EPA/600/M4-82-020, 1982 (see 1.4), or the current EPA method for the analysis of asbestos in building material. The laboratory shall have written procedures that describe how the method is implemented in the laboratory and shall conform in all respects with the test method except when a departure becomes necessary for technical reasons. When a departure is necessary, it shall be stated on the laboratory's test report.

5.4.2 Selection of methods

EPA/600/R-93/116, 1993 (see 1.4), provides clarifications and improvements to the 1982 EPA Interim Method. Specifically, use of the improved method can provide more precise analytical results, especially at low asbestos concentrations, enhanced analysis of floor tiles that may contain thin asbestos fibers below the limits of resolution of the PLM, and clearer instruction on the analysis of bulk materials, particularly where multiple layers are present.

5.4.3 Validation of methods

The laboratory shall have data to demonstrate that departures from the test method do not detract from the expected precision and/or accuracy of the measurement. Laboratories utilizing departures from a test method shall have written procedures detailing how the analysis is conducted. These procedures shall include criteria to determine when such departures are warranted.

5.4.4 Estimation of uncertainty of measurement

5.4.4.1 The detection limit for asbestos is a function of the analyst's capabilities, the laboratory's reliable blank level, and the visibility of asbestos in the sample (a function of microscopic image, contrast and resolution, asbestos fiber size, sample matrix, slide preparation, etc.). The level of quantification may be defined as the concentration at which a statistical uncertainty is determined for the quantity of asbestos reported. The uncertainty may be determined by analysis of standard materials and is a function of the same parameters listed for the limit of detection. If a method other than the point count is used for quantification, the level of quantification for the method shall be determined and documented by the laboratory in order to report the presence of a trace of asbestos.

5.4.4.2 The PLM technique is dependent on the amount of asbestos in the sample and the characteristics of the matrix. Laboratories should recognize this and incorporate provisions for addressing it into their management system. An example of the variability that can be expected with concentration is given in EPA/600/R-93/116, 1993. NVLAP proficiency testing materials, or typical manufactured standards, give the concentration of asbestos in units of weight percent; however, the PLM technique yields results in units of relative area. The conversion of weight percent to area percent requires knowledge of the density and relative grain size of the components of the sample—factors that are often not easily determined. However, the difference between weight percent and area percent is often obscured by the semi-quantitative nature of the PLM technique; for many samples there is no significant difference between weight and area percent results. The exceptions to this statement occur when the sample contains components (such as organic materials) that have a very different density than the asbestos minerals or when there is a large disparity in relative grain size. It is recommended that laboratories use weight percent and point-counted standards when training an analyst for quantification of asbestos by the PLM technique.

5.4.4.3 There shall be an error rate of less than 1 % on qualitative analysis. The laboratory shall monitor the error rate and use it as a guide to determine when an analyst requires additional training to ensure the integrity of measurements. Samples that present particular difficulty shall be identified and specific procedures shall be described for analyzing them, which may include sample rejection or the use of additional analytical or sample preparation techniques.

5.4.5 Control of data

Computerized systems that provide default optical measurements do not meet NVLAP requirements. Laboratories shall have documentation to show that analysts are measuring optical data by PLM examination.

5.5 Equipment

5.5.1 The laboratory shall have the following equipment and supplies for the performance of the tests and measurements required for bulk asbestos analysis:

a) polarized light microscope with the following characteristics: binocular or monocular; one of the oculars shall have a cross hair reticle or functional equivalent that does not rotate during normal operation of the microscope; one of the oculars shall have a magnification of at least 8 X; and objectives low (≥ 5 X and ≤ 15 X), medium (> 15 X and < 40 X), and high (≥ 40 X), or similar magnifications; light source; 360º rotatable stage; substage condenser with iris diaphragm; polarizer and analyzer that can be placed at 90º to one another; accessory slot for wave plates and compensators; wave retardation plate (approximately 550 nm retardation);

b) low-power binocular microscope or stereomicroscope that will not move during normal operation, approximately 10 X to 45 X, with light source;

c) sampling utensils (tweezers, razors, knives, forceps, probe needles, pliers, etc.);

d) sample containers (glassine paper, glass plates, ceramic bowls, petri dishes, etc.);

e) microscope slides and cover slips;

f) refractive index liquids: 1.490 - 1.720, in increments of ≤ 0.005 and calibrated with an accuracy of ± 0.004;

g) mortar and pestle;

h) thermometer that will allow the laboratory to meet NVLAP requirements (a laboratory thermometer from a reputable scientific supply company is acceptable);

i) calibrated refractive index solids or refractometer (or access to) for the calibration of refractive index liquids.

5.5.2 In addition to the equipment and supplies listed in 5.5.1, a biohazard hood of Class I or better, or a re-circulating hood with a HEPA filter (glove box) is required for the safe and non-contaminating handling of bulk asbestos materials in the laboratory. For this program, the purpose of the hood is to protect the laboratory environment from contamination with the potentially large quantities of asbestos handled during routine preparation and macroscopic examination of building materials. A Class I hood is a ventilated cabinet for personnel and environmental protection, with an inward airflow away from the operator. The cabinet exhaust air shall be treated to protect the environment before it is discharged to the outside atmosphere or shall exhaust HEPA-filtered air back into the laboratory. A minimum inward velocity of air into the hood opening of 38 X 10^{-2} m/s (75 fpm) is recommended.

5.5.3 The following equipment and supplies are optional for the laboratory:

a) dispersion staining objective or functional equivalent;

b) high dispersion refractive index liquids;

c) blue "daylight" filter for white light balance;

d) monochromatic (≈589 nm) filter or functional equivalent;

e) mechanical stage and/or point-counting stage;

f) mill and/or blender;

g) analytical balance (readability of 1 mg or better);

h) muffle furnace (temperature range to 500 °C or higher, stable to ± 10 °C or better)*;

i) drying oven*;

j) hydrochloric acid (recommend 10 % by volume)*;

k) filtration supplies.

NOTE It is recommended that the items marked by an asterisk (*) be performed in an exhaust hood.

5.5.4 The periodic cleaning and maintenance of all microscopes by qualified professional technicians is recommended. Routine maintenance of the PLM shall be performed, including:

a) alignment of the light source and substage assembly to provide proper illumination;

b) centering of objectives;

c) focusing of the cross hair reticle in the field of view;

d) testing for coincidence, between the cross hair reticle and the privileged direction of the polarizer and analyzer;

e) determining whether the polarizer and analyzer are normal to one another.

5.6 Measurement traceability

5.6.1 The use of standards, such as NIST Standard Reference Materials, other reference materials, and samples that have been well-characterized by intra- and inter-laboratory testing, such as samples from past NVLAP proficiency test rounds, AIHA, or other proficiency testing programs, and alternate methods of analysis are the best ways to ensure measurement accuracy. Specific preparation techniques may be used for samples to be used as standards, such as those found in Appendix C of EPA/600/R-93/116, 1993.

NOTE It is highly recommended that NIST Standard Reference Materials for asbestos, when available, are purchased to achieve traceability.

5.6.2 Assistance for the calculation of accuracy, precision and required quantification leading to the generation of control charts can be found in many analytical chemistry texts. Laboratories may refer to the following documents that are available for statistical techniques for bulk asbestos analysis using PLM: EPA/600/R-93/116, 1993, NISTIR 5951, and *Guide to Statistical Analysis of Bulk Asbestos Quality Control Data* (see 1.4).

5.6.3 Precision and accuracy of analyses shall be determined on the types of samples received for analysis.

Precision is the ability to repeat a measurement, and can be determined by comparing the results of a single sample by multiple analysts and/or from multiple analyses by the same analyst.

Accuracy describes the correctness of a measurement and can be determined using materials of known concentration, such as past NVLAP proficiency testing materials, prepared standards (in-house or purchased), or materials analyzed by an independent technique. Appendix C of EPA/600/R-93/116, 1993, describes how bulk asbestos samples can be prepared for use as training standards.

5.6.4 For qualitative analyses, the laboratory shall determine whether multiple analyses of the same sample, either by the same or different analysts, and/or by different analytical techniques, yield the same results (asbestos present or not, and type of asbestos) and whether they are correct.

5.6.5 For quantitative analyses, the laboratory shall determine an average value for the concentration of asbestos in the sample with an associated measurement uncertainty. The uncertainty can be determined from several analyses conducted on known quality assurance standards. The precision in the quantitative analysis of a sample by a single analyst is typically defined by the variation observed among multiple slides prepared from the sample or through blind reanalysis of a particular sample. Quantitative results should be represented in the form of tabulated numeric values for each analyst on the various materials.

5.6.6 Control charts documenting precision, accuracy and error rates shall be prepared to assist laboratory personnel in characterizing the overall laboratory and individual performance. Analyst precision and accuracy data shall be documented and reflected in the required monthly summaries. These summaries should be filed appropriately and will undergo evaluation during NVLAP on-site visits. All procedures for such measurements shall be documented in the management system documentation.

5.7 Sampling

Specific sampling procedures shall be documented where appropriate. This is especially important for samples containing multiple layers.

5.8 Handling of test and calibration items

5.8.1 It is recommended that sample receipt and handling during log-in be conducted in a HEPA-filtered, Class I biohazard hood, or a glove box with continuous airflow (negative pressure) (see 5.5.2).

5.8.2 Samples shall be held for at least 30 days unless client specifications or regulations state otherwise. It is recommended that clients be notified of the sample disposal policies of the laboratory.

5.8.3 Because of the legal issues surrounding asbestos analysis, laboratories may consider keeping samples indefinitely or returning the samples to the client with a signed chain-of-custody form.

5.8.4 Samples shall be stored so that sample identification is properly maintained and samples can be readily retrieved.

5.9 Assuring the quality of test and calibration results

5.9.1 Analyst proficiency is the key to providing reliable data. All analysts shall be tested routinely to evaluate their performance. Test results shall be recorded in the personnel folder, or equivalent, of each staff member, and be available during NVLAP assessments. Testing techniques may include, but not be limited to, reanalysis of materials, intra- and inter-laboratory comparisons, analysis of standards, reference materials, NVLAP proficiency testing materials, and blind testing.

5.9.2 Testing shall be frequent enough to ensure quality analyses. Test specimens shall include asbestos-containing and look-alike materials routinely examined by the analysts, and those not often encountered. Problems shall be discussed with the analyst, and corrected according to documented procedures. Subsequent quality assurance tests shall determine whether the problem has been corrected and the laboratory shall ensure the quality of analyses while the problem is being corrected. All corrective actions shall be documented in monthly quality assurance summaries, periodic laboratory audits, and individual analysts' files. Historical data has shown that visual estimation tends to have a high bias. Laboratories shall monitor precision data to determine if there is a problem in this area.

5.9.3 Additional information and techniques are found in EPA/600/R-93/116, 1993.

5.10 Reporting the results

5.10.1 To document the positive identification of asbestos in a sample, the analyst shall record the average optical properties for the population of each asbestos type, including morphology, color and pleochroism, indices of refraction (nD), birefringence, extinction characteristics, sign of elongation, and any other distinguishing characteristics observed. For chrysotile and amosite, refractive indices shall be determined parallel and perpendicular to elongation with an accuracy of ± 0.005. For anthophyllite, tremolite, and actinolite, α and γ, instead of refractive indices parallel and perpendicular to elongation, and shall be measured if the population of fibers display biaxial optics; otherwise the requirements for chrysotile and amosite apply and shall be determined with an accuracy of ± 0.005. For crocidolite, the refractive indices shall be determined parallel and perpendicular to elongation with an accuracy of approximately ± 0.01.

5.10.2 If original data are entered using a computer, analysts shall type the determined refractive indices into the corresponding fields. A set of fixed default values of refractive indices for respective asbestos types automatically generated by the computer is not acceptable (see 5.4.5).

5.10.3 When reporting the percentage of asbestos in a sample, the following shall be considered:

a) The asbestos phase(s) shall be positively identified by PLM and the optical parameters recorded before stating that asbestos is present in any quantity, including trace.

b) The reported quantity of asbestos, including trace, should be consistent on the slide mounts used for quantification. This assumes that the sample is reasonably homogeneous or has been

homogenized to ensure that each sub-sample is representative of the composition of the total sample.

c) A point count or equivalent method is required for quantification. If asbestos is counted during the point count, the percentage should be reported; if asbestos is consistently observed but not counted, trace should be reported; if no consistent quantity of asbestos is observed, zero concentration should be reported.

5.10.4 In addition to the test report requirements found in NIST Handbook 150, the following information shall be reported for each sample:

a) the presence or absence of asbestos and the identification of each asbestos type;

b) the estimate of the average area percentage (or weight percentage) for each type of asbestos present;

c) the identity and area percentage of other fibrous and matrix materials, if known;

d) both color and macroscopic description (and any other information that serves to identify and describe the sample).

5.10.5 A laboratory using analytical techniques in addition to PLM that yield results in terms of weight percent (e.g., gravimetry or x-ray diffraction) may report results in weight percent.

6 Additional requirements

There are no additional requirements beyond NIST Handbook 150 and its associated normative annexes, and any other normative references previously cited in this handbook.

Annex A
(normative)

Documents required in the laboratory

The following documents shall be available at the laboratory:

a) NIST Handbook 150, *NVLAP Procedures and General Requirements*;

b) NIST Handbook 150-3, *NVLAP Bulk Asbestos Analysis*;

c) *Asbestos-containing Materials in Schools, Final Rule and Notice*, as found in the Federal Register, Volume 52, No. 210, pages 41826 - 41846;

d) U.S. Environmental Protection Agency *Method for the Determination of Asbestos in Bulk Building Materials* (EPA/600/R-93/116), 1993, R. L. Perkins and B. W. Harvey;

e) NIST Technical Note 1297, 1994 Edition, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, Barry N. Taylor and Christ E. Kuyatt (available from NVLAP);

f) reference text(s) on optical mineralogy and crystallography;

g) general references text(s) on statistics and quality assurance.