NIST HANDBOOK 150-13
2006 Edition

National Voluntary Laboratory Accreditation Program

AIRBORNE ASBESTOS ANALYSIS

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Contents

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 150-13, *NVLAP Airborne Asbestos Analysis*, presents the technical requirements and guidance for the accreditation of laboratories under the NVLAP Airborne 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 150-13 supersedes and replaces the 1995 edition.

The handbook was revised with the participation of technical experts in the field of airborne 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;

- Lab Bulletin LB-7-2002, previously issued for clarification, has 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 (normative) and Annex B (informative) have 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 purpose of this handbook is to set out procedures and technical requirements for accreditation by NVLAP of laboratories that perform the U.S. Environmental Protection Agency's *Interim Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions*, as found in 40 CFR, Part 763, Appendix A to Subpart E.

The technical requirements expressed in this handbook were originally developed by the Surface and Microanalysis Science Division of the National Institute of Standards and Technology. Special thanks and appreciation are extended to NVLAP Asbestos Program assessors, Thomas Emma, Larry Pierce, and Shu-Chun Su, for their technical assistance.

1 General information

1.1 Scope

1.1.1 NIST Handbook 150-13 specifies technical requirements and provides guidance for the accreditation of laboratories that provide analyses under the NVLAP Airborne Asbestos Analysis Laboratory Accreditation Program (TEM Program). This handbook 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 test method covered by the TEM 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 TEM Program.

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

1.1.4 Any laboratory (including commercial, manufacturer, university, or federal, state, or local government laboratory) that performs the TEM 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 TEM Program. It forms a normative part of this handbook, meaning that it contains provisions to which it is necessary to conform in order to claim compliance with the handbook requirements.

Annex B provides a list of recommended documents that contain information on verified analysis, spot size measurement, k-factor measurement, quality assurance, statistical analysis, and control charts.

1.3 Program description

1.3.1 The purpose of the NVLAP Airborne Asbestos Analysis program is to accredit testing laboratories to provide assurance that they are competent to identify asbestos using transmission electron microscopy (TEM).

1.3.2 Public Law 99-519, *Asbestos Hazard Emergency Response Act of 1986*, referred to as AHERA, requires public and private non-profit primary and secondary schools to inspect their buildings for asbestos-containing building materials. AHERA states that laboratories that analyze TEM asbestos air samples taken from schools are required to be accredited by NVLAP.

1.3.3 NVLAP accredits laboratories to the interim test method developed by the U.S. Environmental Protection Agency, as found in 40 CFR, Part 763, Subpart E, Appendix A, *Interim Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions.*

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 Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions,* 40 CFR, Part 763, Subpart E, Appendix A

1.5 Terms and definitions

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

1.5.1
AEM
Analytical electron microscope.

1.5.2
AHERA
Asbestos Hazard Emergency Response Act of 1986, Public Law 99-519.

1.5.3
analytical sensitivity
Airborne asbestos concentration represented by each fiber counted under the electron microscope and is determined by the air volume collected and the proportion of the filter examined.

1.5.4
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), and 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.

1.5.5
ASTM International
Originally known as the American Society for Testing and Materials.

1.5.6
bias
A systematic error characterized by a consistent (nonrandom) measurement error.

1.5.7
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.

1.5.8
detection limit
The smallest concentration/amount of some component of interest that can be measured using a particular technique with a stated level of confidence.

1.5.9
EDXA
Energy dispersive x-ray analysis.

1.5.10
field blanks
Filters that are processed by removing the cassette cap for not more than 30 seconds before sampling.

1.5.11
filter lot blank
An unopened cassette with filter from the filter supplier.

1.5.12
HEPA filter
High Efficiency Particulate Air filter.

1.5.13
laboratory blank
An unused filter that is exposed in the clean area while a sample set of filters are prepared.

1.5.14
sealed blank
A filter of the same filter lot as those used for sampling. The sealed blank is carried with sample series filters through whole process, but is not opened during sampling operations.

1.5.15
SRM (NIST Standard Reference Material©)
A reference material certified and distributed by the National Institute of Standards and Technology.

1.5.16
STEM
Scanning transmission electron microscope.

1.5.17
TEM
Analytical transmission electron microscope.

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 150-13 Checklist

The NIST Handbook 150-13 Checklist (also referred to as the TEM Program-Specific Checklist) addresses the requirements specific to airborne asbestos analysis. The checklist items are numbered to correspond to clauses 3, 4, and 5 of this handbook and 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

3.1.1 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.

3.1.2 The NVLAP assessment process consists of a NVLAP review of the application and management system documentation, an on-site assessment, and proficiency testing.

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 An on-site assessment typically takes place over a two- to three-day period. During the assessment the laboratory personnel shall be prepared to conduct 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 and will try to minimize disruption to the normal daily routine 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 also attend the meeting.

3.3.3 The assessor reviews laboratory documentation not provided before the assessment. At least one laboratory staff member shall be available to answer questions. 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 assessor for use during the on-site assessment.

3.3.5 The assessor does not need to access employee information that may be considered sensitive or private such as salary, medical information, or performance reviews for work done outside the scope of the laboratory's accreditation. However, the NVLAP assessor checks job descriptions, resumes, and technical performance reviews. At the discretion of the laboratory, a member of its human resources department may be present during the review of personnel information.

3.3.6 The assessor examines equipment and facilities and observes the demonstration of selected procedures by appropriate personnel assigned to conduct the tests, and interviews those personnel. 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 also provide a proficiency test sample and request a specific demonstration.

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

3.3.8 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.

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

3.4 Proficiency testing

3.4.1 General

Proficiency test materials are chosen to test the laboratory's ability to follow the test method and to achieve proper accuracy, precision, and detection limits. Each laboratory will be sent test materials, data sheets, and instructions for performing the test and reporting the results. The test shall be conducted in accordance with the specific test method using the laboratory's normal operating procedures.

A proficiency test is not required for initial accreditation, but laboratories shall participate in rounds of testing as scheduled by NVLAP.

3.4.2 Types of proficiency testing

Proficiency testing may involve materials or artifacts that must be returned to NVLAP. These materials shall be protected from damage both in the laboratory and during shipment to NVLAP or its designated contractor. Examples of such materials and artifacts are: filters, grids, photographs, and data sheets. These materials may be used to determine testing performance for specific subparts of the test method. Unless otherwise instructed, laboratories may keep the proficiency testing materials for use as in-house instructional materials.

3.4.3 Analysis and reporting

3.4.3.1 All laboratory analysts, including those in sub-facilities (see 4.1.2 and 4.1.3), 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.

3.4.3.2 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 3.4.4.3).

3.4.3.3 Special instructions shall be followed, as they are designed to ensure uniformity in procedures among proficiency testing participants.

3.4.3.4 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 will result in penalties, which may include failing that round.

3.4.3.5 NVLAP will report proficiency test performance to the participants in appropriate documents. The identity and performance of individual laboratories will remain confidential. Any analytical issues indicated by proficiency testing shall be discussed with appropriate laboratory personnel, who shall then

be responsible for developing and implementing plans for resolving the issues. The satisfactory resolution of proficiency testing nonconformities is required for accreditation.

3.4.4 Proficiency testing nonconformities

3.4.4.1 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 failure, detailed, written documentation that includes an analysis of why the laboratory failed the test, and what corrective action(s) it has taken (analyst training, revised procedures, quality assurance activities, etc.) to resolve the analytical problem(s);

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

c) pass the next round of proficiency testing.

3.4.4.2 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 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 on-site assessment 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.

3.4.4.3 Failure to participate in a round of proficiency testing will result in immediate suspension of accreditation. To have 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

4.4.1 The laboratory shall ensure that the contract review includes a determination of the client's need for an AHERA-compliant test.

4.4.2 Non-AHERA work shall meet NVLAP and client requirements.

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 airborne asbestos analysis. [See also NIST Handbook 150, 5.10.2 b) and 5.10.6].

4.5.2 A NVLAP-accredited laboratory shall not represent test data produced at any 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 shall be three years; however, a longer period of retention may be required by the client, by regulation, or by the laboratory's own procedures. The records shall be stored in a logical fashion allowing retrieval within one working day. The records to be maintained include:

a) personnel records including operator characterization;

b) sample custody records;

c) original data collected by the analyst;

d) contamination monitoring data;

e) calibration and verification data;

f) data and results of quality control;

g) facility, equipment and maintenance records;

h) test reports.

NOTE For more information on records, see the NIST Handbook 150-13 Checklist.

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

5.1.1 The laboratory shall maintain and summarize all of the quality assurance activities each month including:

a) contamination checks;

b) measurement uncertainty for each analyst and for the laboratory;

c) interlaboratory and intermicroscope analyses;

d) sampling precision determined by filter reanalysis;

e) calibrations;

f) sample custody;

g) problems, corrective actions and nonconformity corrections.

5.1.2 The laboratory's quality assurance analyses shall represent at least 10 % of the total number of analyses (including all analyses where the AHERA protocol is used). The value of 10 % is a minimum value that applies to a laboratory that has:

a) trained analysts;

b) laboratory calibrations, contamination checks and other management system components statistically characterized and in a state-of-control;

c) a high frequency of analyses.

5.1.3 For laboratories not fitting the criteria in 5.1.2, the quality assurance analyses shall be a higher percentage of the total number of TEM asbestos analyses performed.

5.1.4 The quality assurance checks shall be performed routinely, covering all time periods, sample types, instruments, tasks and personnel. The laboratory shall select samples for quality assurance checks and when possible, the checks on personnel performance shall be conducted without their prior knowledge.

5.1.5 The laboratory management shall foster an environment conducive to quality work.

5.1.6 The most recent documents specified in the NIST Handbook 150-13 Checklist shall be available as references in maintaining the laboratory's management system.

5.2 Personnel

5.2.1 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. Verified asbestos analysis status is required to evaluate the performance of the analyst (see NIST Handbook 150-13 Checklist). The laboratory shall establish and document performance criteria to determine when a new analyst is qualified to work independently.

5.2.2 Technical supervisors shall be qualified to conduct analytical electron microscope (AEM) studies and apply AEM to crystalline materials, and shall be knowledgeable in the field of asbestos analysis, including procedures for sample handling, preparation, analysis, storage, disposal, and contamination monitoring.

5.2.3 Technical supervisors shall be sufficiently familiar with quality control and statistical analysis to present laboratory data in a meaningful way.

5.2.4 An AEM analyst(s) shall follow the test method, including accurately finding and analyzing fibrous materials, measuring the pertinent physical properties on the AEM, drawing proper conclusions from these data, and knowing when and where to obtain aid in the analysis of the samples as prescribed by the quality manual or other established laboratory procedure.

5.2.5 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.

5.2.6 AEM analysts and technical supervisors shall participate in an appropriate form of continuing education, such as formal coursework, in-house education, and scientific or technical meetings, and have access to journals that describe advances in the field of electron microscopy and/or asbestos analysis. The laboratory shall have a written description of its criteria for successful completion of its training program.

5.2.7 The laboratory will be responsible for demonstrating staff competence to analyze asbestos samples following the practice outlined in its quality documentation.

5.2.8 Staff members involved in the analysis of samples will be responsible for demonstrating competence, as required, during an on-site assessment. In particular, analysts shall be able to demonstrate the ability to use and interpret results from the AEM in imaging, diffraction, and x-ray analysis modes, to identify the various types of asbestos, and to differentiate asbestos from non-asbestos fibers.

5.2.9 All personnel shall be instructed in contamination detection and prevention.

5.3 Accommodation and environmental conditions

5.3.1 Laboratory facilities

5.3.1.1 The laboratory shall have the following facilities necessary to analyze airborne asbestos under safe working conditions:

a) a clean room or clean areas for sample preparation and handling that is separate from bulk asbestos, and is equipped with class 100 (or cleaner) HEPA-filtered air under positive pressure and an exhaust hood for the safe use of filter dissolution reagents;

b) an electron microscopy facility;

c) a room or area for filter and grid storage separate from bulk asbestos or other sources of contamination.

5.3.1.2 Safe working conditions shall be maintained, including the safe handling and storage of asbestos and filter-dissolving reagents, such as chloroform, dimethyl formamide, acetone, acetic acid, etc.

5.3.2 Contamination control

5.3.2.1 Preventing contamination is critical in the analysis of asbestos in air, which typically involves analyzing nano- or pico-gram quantities of asbestos. No statistically significant contamination of work areas, personnel, instrumentation, or materials used for preparation or analysis of air filter samples shall be tolerated. Laboratories shall have written procedures for the prevention, monitoring, and control of contamination of filters and grids, and for the systematic checking for possible sources of contamination, if contamination is detected. The procedures shall include the checking of all work areas, instrumentation, and materials used in the preparation and analysis of air filter samples.

Work areas, personnel, instrumentation, and materials used for the preparation and analysis of bulk materials that contain asbestos are a potential source of contamination. The probability of cross-contamination requires that work areas, personnel, instrumentation, and materials be kept completely separate from those used for the preparation and analysis of air filter samples. Personnel who have worked with bulk samples shall not subsequently be allowed to work with air filter samples on the same

day. It is acceptable, however, for personnel to work on bulk samples after having worked with air samples. All reagents shall be checked for asbestos contamination prior to use for sample preparation.

5.3.2.2 The management system shall outline the frequency and timing of asbestos contamination checks of blank materials, which commonly consist of filters that have not been used for sampling, but can also include carbon film grids, or other items appropriate for detecting asbestos contamination. Filter blanks include filter lot blanks, sealed blanks, field blanks and laboratory blanks.

5.3.2.3 The laboratory blanks shall be obtained from a lot of filters that has been shown not to be contaminated.

5.3.2.4 Laboratory blanks shall be analyzed on a routine basis, but with increased frequency if contamination is discovered and corrected. At a minimum:

a) one laboratory blank shall be prepared for every sample set from a site or for 10 % of the samples (whichever is greater);

b) a laboratory blank shall be analyzed after the analysis of 25 samples, or when the average count for a full set of filters is found to be above 70 structures/mm^2;

c) a laboratory blank shall be prepared and analyzed after cleaning or servicing the clean room or clean area;

d) field and sealed blanks shall be prepared with each set of samples and shall be analyzed when the full indoor/outdoor analysis is performed.

5.4 Test and calibration methods and method validation

The NVLAP requirement for grid square overloading criteria is 10 % coverage. Laboratories are required to have written procedures for examining a grid square and for counting and analyzing particles. Among the rejection criteria are particle loading exceeding > 10 % by area and uneven particle loading. For NVLAP TEM laboratories, the 10 % criterion shall be written into their procedures, and the laboratories will be assessed to the 10 % requirement.

The NVLAP requirement was written after careful study of AHERA. In the case of the criterion for acceptance/rejection of loaded grid squares, the technical experts at NIST decided that 25 % coverage would allow asbestos structures to be obscured, so a lower coverage of 10 % was adopted as the NVLAP requirement.

5.5 Equipment

The laboratory shall be equipped with a clean room or area, low temperature plasma asher, electron microscope, specific document references, etc., which are specified in-depth in the NIST Handbook 150-13 Checklist.

5.6 Measurement traceability

The laboratory shall have specific procedures in its management system documentation for measurement traceability. See the NIST Handbook 150-13 Checklist for specific requirements.

It is highly recommended that NIST Standard Reference Materials, or other appropriate reference materials for asbestos, be used to achieve traceability.

5.7 Sampling

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

5.8 Handling of test and calibration items

5.8.1 The laboratory shall have a sample log-in system that includes the following documentation:

a) date of receipt;

b) identity of the client;

c) unique identification for the sample;

d) air volume pulled through the sample;

e) filter pore size;

f) condition of the samples;

g) acceptance or rejection of the samples.

5.8.2 The laboratory shall have written criteria for acceptance or rejection of filter cassettes.

5.8.3 The laboratory shall have a documented chain-of-custody system by which the following is recorded:

a) location of sample;

b) a listing of personnel who have handled or worked with the sample;

c) a listing of what has been done to the sample.

5.8.4 The laboratory shall:

a) store the unused portions of filters in their cassettes for at least 30 days;

b) store all prepared grids (even in not analyzed) for at least three years;

c) store the filters and grids in a logical fashion so that specified samples can be retrieved in a timely manner.

5.9 Assuring the quality of test and calibration results

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

5.10 Reporting the results

5.10.1 Test reports shall include the following information for each sample set:

a) the area of the filter analyzed;

b) volume of air sampled (with reference to sampling data sheet);

c) analytical sensitivity used for the analysis;

d) number of total asbestos structures and number of structures by asbestos type (chrysotile, grunerite, riebeckite, anthophyllite, tremolite, or actinolite);

e) concentration in asbestos structures/mm^2 of filter and asbestos structures/cm^3 of air for total asbestos structures, and with data broken down by size ($\geq$ 0.5 μm to < 5 μm), and by asbestos type;

f) statement of analytical uncertainty;

g) micrograph number of any recorded diffraction patterns;

h) copy of AEM analysis data record with analyst's signature or initials;

i) descriptions of any departures from the test method.

5.10.2 The following additional information shall be supplied if asbestos abatement clearance is determined to be necessary:

a) calculation formulas;

b) all calculation variables and constants;

c) all calculation results.

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 150-13, *NVLAP Airborne Asbestos Analysis*;

c) U. S. Environmental Protection Agency's (EPA), *Interim Transmission Electron Microscopy Analytical Methods–Mandatory and Nonmandatory–and Mandatory Section to Determine Completion of Response Actions*, Appendix A to Subpart B, 40 CFR Part 763, October 1987, or the current U.S. EPA AEM method for the determination of completion of response actions;

d) *Asbestos-Containing Materials in Schools*; Final Rule and Notice, 40 CFR, Part 763, Subpart E;

e) General references on analytical electron microscopy, transmission electron microscopy, asbestos analysis, and crystallography;

f) AEM manufacturer's operations manual;

g) Multichannel analyzer manufacturer's operations manual.

Annex B
(informative)

Recommended documents

It is recommended that the following documents be available in the laboratory for additional information on the topics listed below.

a) **For verified analysis:**

— E. B. Steel and J. A. Small, "Accuracy of Transmission Electron Microscopy for the Analysis of Asbestos in Ambient Environments," *Analytical Chemistry*, Vol. 57, 1985, pp. 209-213.

— S. Turner and E. B. Steel, "Analysis of Transmission Electron Microscopy Analysis of Asbestos on Filters: Interlaboratory Study," *Analytical Chemistry*, Vol. 63, 1991, pp. 868-872.

— S. Turner and E. B. Steel, NISTIR 5351, A*irborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0*, 1994.

b) **For spot size measurement:**

— D. B. Williams, *Practical Analytical Electron Microscopy in Material Science*, Philips Electronics Instruments, Inc., Mahwah, NJ, 1984, pp. 34-35 (for TEM and STEM mode).

— D. B. Williams, "Standardized Definitions of X-ray Analysis Performance Criteria in the AEM," in A. D. Romig and W. F. Chambers, (ed.) *Microbeam Analysis 1986*, San Francisco Press, San Francisco, CA, 1986, pp. 443-448 (for TEM mode).

— J. I. Goldstein, et al., *Scanning Electron Microscopy in Materials Science*, Plenum Press, New York, 1981, p. 48 (for STEM mode).

c) **For k-factor measurement:**

— D. C. Joy, A. D. Romig, J. I. Goldstein, *Introduction to Analytical Electron Microscopy*, Plenum Press, New York, 1986.

— D. B. Williams, *Practical Analytical Electron Microscopy in Materials Science*, Philips Electronics Instruments, Inc., Mahwah, NJ, 1984.

d) **For quality assurance:**

— J. K. Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, MI, 1987.

e) **For statistical analysis:**

— M. G. Natrella, *Experimental Statistics*, John Wiley & Sons, New York, 1966.

f) **For control charts:**

— *Manual on Presentation of Data and Control Chart Analysis*, ASTM International, Philadelphia, 2002.

— Reference data on the crystallography and chemical composition of minerals that analytically interfere with the regulated asbestos minerals.