National Institute of Standards and Technology



National Voluntary Laboratory Accreditation Program

PROGRAM HANDBOOK

AIRBORNE ASBESTOS ANALYSIS



NISTIR 89-4137 AUGUST 1989



U.S. Department of Commerce National Institute of Standards and Technology Gaithersburg, Maryland 20899

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OPERATIONAL AND TECHNICAL REQUIREMENTS

OF THE

LABORATORY ACCREDITATION PROGRAM

FOR

AIRBORNE ASBESTOS ANALYSIS

NISTIR 89-4137

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I. PROGRAM SUMMARY

This document presents the operational and technical requirements to be fulfilled in order for laboratories to gain accreditation under the National Voluntary Laboratory Accreditation Program (NVLAP) to perform airborne asbestos testing using analytical transmission electron microscopy (ATEM). All steps leading to accreditation are discussed. Technical requirements are explained and the way NVLAP criteria are applied is described.

Laboratory accreditation for airborne asbestos analysis was established by the National Institute of Standards and Technology¹ (NIST) in response to the requirements set forth in Public Law 99-519, the Asbestos Hazard Emergency Response Act of 1986. The purpose of accreditation is to identify and recognize laboratories that produce reliable test data for the services covered.

Test Method Covered:

Environmental Protection Agency's, "Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions", Appendix A to Subpart E, 40 CFR part 763, October 30, 1987 or the current U.S.
Environmental Protection Agency TEM method for the determination of completion of response actions for asbestos.

Technical experts with experience in analysis of

Participation in proficiency testing is required. Testing of precharacterized quality assurance materials sent to the laboratory by NIST or an authorized contractor. Data to be returned to NIST for evaluation. Proficiency testing schedule

airborne asbestos by transmission electron

Period of accreditation:

On-site assessment:

One year

microscopy.

Performed by NVLAP peer assessor, to determine compliance with NVLAP criteria, after initial application and every two years thereafter. Monitoring visits as required.

Assessors:

Proficiency testing:

Fees:

Annual administrative/technical support fee, biannual proficiency testing fee; on-site assessment fee.

Granting Accreditation: Based upon successful on-site assessment, proficiency testing, and technical evaluation of applicable laboratory information.

will be provided in advance.

¹ Formerly the National Bureau of Standards (NBS)

Background

The U.S. Department of Commerce, National Institute of Standards and Technology (NIST) administers the National Voluntary Laboratory Accreditation Program (NVLAP). NVLAP's function is to accredit public and private testing laboratories based on evaluation of their technical qualifications and competence for conducting specific test methods in specified fields of testing. Accreditation is granted on the basis of conformance with criteria published in the Code of Federal Regulations (15 CFR Part 7) as part of the NVLAP procedures (see Appendix A).

This document is intended for information and use by staff of accredited laboratories, those seeking accreditation, other laboratory accreditation systems, and others needing information on the requirements for the NVLAP accreditation under this program. This document is generally included in the NVLAP Application Package along with General Application Forms, Test Method Selection Lists, and other materials needed to apply for or renew accreditation. It presents the administrative and operational procedures and technical requirements of the accreditation program and should be retained and be readily accessible to laboratory personnel.

NVLAP Accreditation

Accreditation is granted only after thorough evaluation of the applicant has demonstrated that all NVLAP criteria have been met. The accreditation is formalized through issuance of a Certificate of Accreditation, Scope of Accreditation and publicized by announcement in various government and private media.

NVLAP accreditation is available to commercial laboratories, manufacturers' in-house laboratories, university laboratories, and Federal, State, and local government laboratories. Foreign-based laboratories may be accredited by NIST if they meet the same requirements as domestic laboratories and pay any additional fees required.

Why NVLAP Accreditation?

A laboratory may wish to be accredited for one or more of the following reasons: legal requirements (such as regulations or codes), contract specifications, or the desire to be recognized as demonstrably competent to meet the needs of its clients.

For accreditation to be meaningful, it must be granted by a clearly credible organization. NVLAP provides an unbiased third party evaluation and recognition of performance as well as expert technical assistance to upgrade laboratory performance when so needed.

Testing Laboratory Defined

NVLAP defines "testing laboratory" as an organization that provides services to

measure, examine, test, calibrate, or otherwise determine the characteristics or performance of materials, products or systems.

Accreditation Defined

NVLAP accreditation signifies recognition of a testing laboratory's competence to perform specific test methods in specified fields of testing. It means that the laboratory's Quality System, staff, facilities, equipment, calibration procedures, test methods and procedures, records, and test reports have all been evaluated and found to meet NVLAP criteria. NVLAP accreditation <u>does not mean</u> a guarantee (certification) of laboratory performance or of product test data; it is solely a finding of laboratory competence.

For further information about NVLAP, or for assistance in understanding and meeting the NVLAP requirements and criteria, please write or call:

NVLAP National Institute of Standards and Technology Bldg 411, A124 Gaithersburg, MD 20899 Phone: (301) 975-4016

III. ADMINISTRATIVE AND OPERATIONAL REQUIREMENTS

Note: Administrative and operational requirements presented here are generally applicable to all NVLAP programs. Technical and proficiency requirements are specifically applicable to this asbestos accreditation program.

LABORATORY CODE NUMBER (LAB CODE)

Each participating laboratory is assigned a four-digit laboratory code number. The code number is used by the NVLAP staff for identification, filing, recordkeeping, and database management. Participants are requested to put their Lab Code number on all correspondence with NVLAP. The Lab Code number is cross-referenced with the laboratory name and location in the NVLAP Directory of Accredited Laboratories.

ACCREDITATION PERIOD

Accreditation is granted for a period specified in the Accreditation Application Package (usually one year). The accreditation period begins on one of four dates: January 1, April 1, July 1, or October 1. Each laboratory retains its assigned accreditation date as long as it remains in the program; its accreditation expires and is renewed on that date.

RENEWAL

Each participating laboratory will be sent a renewal Application Package, well in advance of the expiration date of its accreditation, to allow sufficient time to complete the renewal process. The renewal application contains the same forms used for initial application. The laboratory may use copies of pages of previously submitted applications but must indicate any changes that may have occurred in personnel, equipment, facilities, or the scope of accreditation desired.

The technical requirements and fees for renewal are generally the same as for initial accreditation. The application and fees must be received by NIST prior to expiration of the laboratory's current accreditation to avoid a lapse in accreditation.

PUBLICIZING ACCREDITATION STATUS

BY NVLAP

NVLAP publishes an annual Directory of Accredited Laboratories. The Directory contains the name and address, scope of accreditation, contact person, and the accreditation renewal date for each accredited laboratory. Supplements to the Directory are published quarterly to cover interim accreditation actions including initial accreditations, renewals, suspensions, terminations, and revocations. The Directory is distributed nationally and internationally to manufacturers, suppliers, retailers, professional and trade associations, code groups, and government agencies.

BY LABORATORIES

Accredited laboratories are encouraged, within specified limits, to publicize their accredited status. The major restriction is that advertising must not imply product certification by NIST or the U.S. Government. Laboratories and their clients may not reference their accredited status in consumer media, in product advertising, or on product labels, containers or packaging.

A laboratory may cite its accredited status and use NVLAP logos on reports, stationery, and in business and trade publications provided that it is clearly indicated that it is the laboratory which is accredited and not the test results or the analysts. NVLAP Lab Bulletin No. 3A provides more detailed guidance on how a laboratory may publicize its accredited status and the statements which may be made (see Appendix B).

COMPLIANCE WITH EXISTING LAWS

Accreditation does not relieve the laboratory of the need to observe and comply with existing Federal, State, and local statutes, ordinances, or regulations that may be applicable to its operations, including consumer protection and antitrust laws.

ACCREDITATION PROCESS

Accreditation is granted following successful completion of a process which includes submission of an application and payment of fees by the laboratory, an on-site assessment, resolution of deficiencies identified during the on-site assessment, participation in proficiency testing, technical evaluation, and administrative review. The process is described in the following sections.

APPLICATION AND FEES

An Application Package is sent to a laboratory on request. It includes: General Application Forms, a Fee Calculation Sheet, and this document. The General Application Form must be completed and signed by an authorized representative of the laboratory. The authorized representative is one who can act on behalf of the laboratory and commit it to fulfill the NVLAP requirements. Before completing and signing the application, the authorized representative should review all documents and become totally familiar with NVLAP requirements. Although other laboratory staff may be designated to perform activities such as handling proficiency testing or receiving an assessor, the authorized representative is the only one who can authorize a change in the scope or nature of the application.

In general, the accreditation fee is composed of several parts, some of which are fixed while others depend on the scope of accreditation desired and the

specifics of the program. The total accreditation fee must be paid before accreditation can be granted. The individual parts of the accreditation fee include, <u>as appropriate</u>: an Administrative and Technical Support fee, a Test Method fee, a Proficiency Testing fee (excluding the cost of reference materials), and an On-Site Assessment fee. The fees for this accreditation program are shown in the Fee Calculation Sheet included in the Program Application Package.

The laboratory will be contacted to schedule a mutually acceptable date for the on-site assessment <u>after payment</u> of all required fees. It will also be notified if any additional information must be supplied, and if any applicable proficiency testing requirements must be completed, for the technical evaluation.

APPROVED SIGNATORY

Under NVLAP criteria, an accredited laboratory must have one or more individuals or personnel (approved signatories) in positions designated as having responsibility for signing <u>all test reports endorsed with the NVLAP logo</u>. This is the person(s) to be contacted by NVLAP, laboratory clients, or others if there are questions or problems with the report.

There is no formal requirement for nomination or approval of persons or laboratory positions designated as approved signatories. The laboratory should inform NVLAP of its appointments by completing the appropriate sections in the application for accreditation. Approved signatories should be persons in positions with adequate responsibility or authority within the organization, with adequate and appropriate technical capabilities, and without conflict of interest. The approved signatory may be the authorized representative who is responsible for signing the NVLAP Application Form.

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Laboratory <u>test reports carrying the NVLAP logo need not be signed individually</u> by the approved signatory. Test report forms may be preprinted with the required information. Forms that are electronically or computer generated may have the information printed along with the test results.

TECHNICAL EXPERTS

NVLAP uses Technical Experts (TEs) as assessors and evaluators. They are engineers and scientists currently active in their field, consultants, college professors or retired persons. They are selected on the basis of their professional and academic achievements, experience in the field of testing, management and quality assurance experience, and tact in dealing with people. Their services are generally contracted as required; they are not NVLAP staff members.

<u>Assessors</u> are TEs selected to conduct an on-site assessment of a particular laboratory on the basis of how well their individual experience matches the type of testing to be assessed, as well as absence of conflicts of interest. The laboratory has the right to appeal the assignment of an assessor and may request an alternate. <u>Evaluators</u> are TEs selected to review the record of the laboratory as a whole, including the application, assessment report, deficiencies, corrections to deficiencies, and proficiency test results and, based on this record, to recommend whether or not a laboratory should be accredited. The evaluators are matched to the type of testing being evaluated and are selected to avoid conflicts of interest. The TE who assessed the laboratory does not serve as an evaluator of the laboratory.

ON-SITE ASSESSMENT

Before initial accreditation and periodically thereafter, an on-site assessment of each laboratory is conducted to determine its compliance with the NVLAP criteria. The assessment is conducted by one or more NVLAP assessors selected on the basis of their expertise in the field of testing to be reviewed. Assessors use checklists developed by NVLAP so that each laboratory receives an assessment comparable to that received by others.¹ However, assessors have considerable latitude to make judgments about a laboratory's compliance with the NVLAP criteria, depending on the assessor's experience and the unique circumstances of the laboratory. The laboratory may request a change of assigned assessor based on conflict-of-interest or prior business or professional associations.

Each laboratory will be contacted to arrange a mutually agreeable date for an assessment. The time needed to conduct an assessment varies, but one day is the norm. Every effort is made to conduct an assessment with as little disruption as possible to the normal operations of the laboratory. During the assessment the assessor will carry out the following functions:

- meet with management and supervisory personnel responsible for the laboratory's activities (for which accreditation is being sought) to review the assessment process with the individuals involved and to set the assessment agenda.
- examine the quality assurance system employed by the laboratory. The assessor may select and trace the history of one or more samples from receipt to final issuance of test reports. The assessor will conduct a thorough review of the laboratory's Quality Assurance Manual or equivalent, evaluate the training program, examine notebooks or records pertaining to the samples, check sample identification and tracking procedures, determine whether the appropriate environmental conditions are maintained, and examine copies of completed test reports.
 - review records of periodic internal audits, use of check samples or participation in round robin testing or other similar programs.
- review personnel records including resumes and job descriptions of key

¹ The NVLAP General Operations Checklist and the Specific Operations Checklist for the Airborne Asbestos Program are included as appendices to this Handbook.

personnel, competency evaluations for all staff members who routinely perform the testing for which accreditation is sought, calibration or verification records for apparatus used, test reports, and sample control records.

- observe demonstrations of testing techniques and discuss them with the technical personnel to assure their understanding of the procedures.
- examine major equipment, apparatus, and facilities associated with testing for which the laboratory is seeking accreditation.

At the conclusion of the assessment, the assessor will conduct an exit briefing to discuss his or her observations with responsible laboratory staff and call attention to any deficiencies uncovered. <u>A written summary of any deficiencies</u> <u>discussed will be left at the laboratory</u>. The assessor will forward the assessment forms and a written summary to NIST.

> If deficiencies have been noted, the laboratory must provide NVLAP with documentation or certification by the authorized representative, that the specified deficiencies have been corrected or that specific actions are being taken to correct the deficiencies.

If any deficiencies are noted at laboratories which are currently accredited, such deficiencies must be corrected within 30 days after notification or the laboratory may face possible revocation, suspension, or expiration of its accreditation. Any test equipment that is identified as out of calibration, should not be used until corrective action has been completed. All deficiencies noted for corrective action will be subject to thorough review and verification during subsequent assessments and technical evaluations.

MONITORING VISITS

In addition to regularly scheduled assessments, monitoring visits may be conducted by assessors or by NIST staff at any time during the accreditation period. Monitoring visits may occur for cause or on a random selection basis.

These visits serve to verify reported changes in the laboratory's personnel, facilities, and operations or to explore possible reasons for poor performance in proficiency testing.

The scope of a monitoring visit may range from checking a few designated items to a complete review. Failure to cooperate with NVLAP assessors will be grounds for initiation of adverse accreditation action.

PROFICIENCY TESTING

Proficiency testing is an integral part of the NVLAP accreditation process. Demonstration of appropriate facilities, equipment, personnel, etc., is essential, but may not be sufficient for the evaluation of laboratory competence. The actual determination of test data using special proficiency testing samples provides NVLAP with a means for determining the overall effectiveness of the laboratory.

Proficiency testing is a process for checking actual laboratory testing performance, usually by means of interlaboratory comparisons. Each accreditation program has unique proficiency testing requirements. The data are analyzed by NVLAP and summary reports of the results are sent to the participants.

For many test methods, proficiency testing results are good indicators of a laboratory's testing capability. Information obtained from proficiency testing helps to identify problems in a laboratory. If problems are found, NVLAP staff members work with the laboratory staff to solve them. If problems with the test method are suspected, NVLAP provides information to the appropriate standards writing bodies.

The specific proficiency testing requirements for this Program are included in Section V of this document.

TECHNICAL EVALUATION

After a laboratory has completed all the technical requirements of a Program and is ready for an accreditation action, a final technical evaluation is conducted by experts chosen for their experience and knowledge of the pertinent test methods. They review records on an applicant laboratory and base their evaluation on:

- information provided on the application;
- on-site assessment reports;
- actions taken by the laboratory to correct deficiencies;
- results of proficiency testing; and
- information from any monitoring visits of the laboratory.

If the technical evaluation reveals additional deficiencies, written notification describing them will be made to the laboratory. The laboratory must respond within 30 days of such notification and provide documentation or certification by the authorized representative that the specified deficiencies have been corrected. Clarification of some issues may be requested by telephone. <u>All deficiencies must be corrected before accreditation can be</u> granted or renewed.

ADMINISTRATIVE REVIEW

After the technical evaluation has been completed, the NVLAP staff prepares an administrative recommendation whether the laboratory should be granted or denied accreditation. This recommendation is based on a review of the technical evaluation and other records to ensure that all NVLAP technical, financial and administrative obligations have been satisfied.

ACCREDITATION ACTIONS

Based on the technical findings pertaining to the laboratory's compliance with the NVLAP criteria, the Associate Director for Industry and Standards (ADIS), acting for the Director of NIST, makes one of the following decisions:

- Accreditation The recommendation forms the basis for granting accreditation and a Certificate of Accreditation is issued to the laboratory.
- <u>Denial</u> The laboratory is notified of the intent to deny accreditation and the reason(s) therefor.
- <u>Suspension</u> If a laboratory is found to have violated the terms of its accreditation, the laboratory will be notified of the reasons for and conditions of the suspension and the action(s) that the laboratory must take to have accreditation reinstated.
- Revocation If a laboratory is found to have violated the terms of its accreditation, the laboratory is notified of the intent to revoke accreditation and the reasons therefor. The laboratory may be given the option of voluntarily terminating accreditation. If accreditation is revoked, the laboratory must return its Certificate of Accreditation and must cease using the NVLAP logo on any of its reports, correspondence, or advertising.

If denial or revocation has been proposed, the laboratory may request a hearing, under United States Code 5 U.S.C. 556, within 30 days of the date of receipt of the notification. If a hearing is not requested, the action becomes final upon the expiration of that 30-day period.

After a participant's accreditation has been terminated, whether voluntarily or through adverse action, the accreditation certificate must be returned to NVLAP. If a laboratory elects not to renew or voluntarily chooses to terminate its accreditation at any time, the notification of such intention should be forwarded to NIST in writing.

IV. TECHNICAL REQUIREMENTS

The <u>Criteria for accreditation</u>, Section 7.33 of the NVLAP Procedures (see Appendix) provides the basis for the technical evaluation of a laboratory. This section provides interpretive comments and additional general information to make the criteria specifically applicable to the Airborne Asbestos Laboratory Accreditation Program. The Specific Operations Checklist (Appendix F) provides further details regarding the program technical requirements.

Scope of the Program

The NVLAP Airborne Asbestos Program offers accreditation to laboratories that perform analytical transmission electron microscopy (ATEM) using the following test method:

Environmental Protection Agency's, "Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions", Appendix A to subpart E, 40 CFR part 763, October 30 1987, or the current U.S. Environmental Protection Agency TEM method for the determination of completion of response actions for asbestos.

Comments on QUALITY SYSTEM (see Procedures Sec. 7.33a)

The Quality System requirements are designed to promote a laboratory practice that ensures technical integrity of the analyses and adherence to quality assurance standards. It also requires that a Quality Assurance Manual be produced that documents the laboratory practice and the specific steps taken to ensure quality assurance. The requirements state that quality assurance standards for each function in the laboratory must be outlined. In the analysis of asbestos in air by ATEM, those areas that must be specifically addressed are: 1) sample custody, 2) contamination, 3) instrument calibration, 4) analyst characterization, and 5) laboratory characterization.

The Quality System must show that appropriate log-in procedures are followed to document the condition of the samples upon receipt. The laboratory may reject the samples if rejection criteria are met. If the samples are accepted, the laboratory is responsible for documenting sample custody, and for tracking the samples through the analysis procedure.

Contamination is a critical factor in the analysis of asbestos. The analysis of asbestos in air typically involves analyzing nanogram quantities of asbestos and therefore a class 100 clean room or bench [1] is required. The preparation and analysis of trace quantities of asbestos on air filters is not compatible with the preparation and analysis of bulk materials containing potentially major amounts of asbestos. The probability of cross contamination requires that these materials and techniques be kept completely separate. The Quality System must outline the frequency and timing of checks for contamination of any laboratory equipment or supplies which are used in the analysis of asbestos. These include, but are not restricted to, laboratory filter blanks, field blanks, sealed filter blanks and blanks relative to other materials and locations including solvents, low temperature ashers, instruments (microscopes), Jaffe wick materials, evaporators, tweezers, specimen storage boxes, TEM specimen holders, etc. as needed.

When contamination above acceptable levels is found, it is corrected or analysis cannot be performed for AHERA clearance. Both the initial contamination problem and the corrective measures are entered into the Quality System records on contamination control.

The Quality System must address the qualifications, duties, and methods used to determine the proficiency of each staff member. Summaries of staff performance and problems are entered into the Quality System records. The Quality System must outline the frequency and timing of calibration of the transmission electron microscope(s), the energy dispersive x-ray detector(s), multichannel analyzer(s), and low temperature asher(s). The specific items which must be calibrated are listed under <u>Comments on CALIBRATION</u>. Summaries of calibration

results and problems are entered into the Quality System records on calibration.

A summary of the capabilities of the technical facility including information on the equipment is present in the Quality System records. Any additions to the laboratory facilities or changes in the condition of equipment are noted in the Quality System records on Facilities and Equipment.

The Quality System details the ATEM test method as it is applied in the laboratory. Summaries of changes and problems are entered into the Quality System records on the Test Method.

The laboratory records are defined in the Quality System and are updated and reviewed at least annually. Summaries of changes and problems are entered in the Quality System records.

The Quality System includes the laboratory requirements for test reports and verification of reporting results.

<u>Comments on STAFF</u> (See Procedures Sec. 7.33b)

A laboratory requesting accreditation must maintain a complete listing of its staff. The duties for each position must be outlined and the staff member(s) assigned to the position must be identified. Each staff member must have a resume, and the results of any Quality System tests must be readily available. As stated in Sec. 7.33b, item 3, the laboratory must have a description of its training program for ensuring that new or untrained staff are able to perform tests properly and uniformly to the requisite degree of precision and accuracy.

The personnel positions necessary for analysis of asbestos in air by ATEM cover activities necessary to perform the following required procedures: sample receipt/custody, sample preparation, sample analysis, data analysis and reporting, quality assurance, and laboratory supervision. All personnel must be qualified to perform the laboratory procedures for which they are responsible. The person in charge of sample custody is responsible for such things as the documented security, tracking and storing of samples. The laboratory technical supervisor(s) must understand the principles and practice of transmission electron microscopy and must be qualified to conduct analytical transmission electron microscopy and its application to crystalline materials, including the measurement and interpretation of electron diffraction patterns, and the interpretation of energy dispersive x-ray spectra.

The analyst(s) must be capable of following the analysis method, including accurately finding and analyzing fibrous materials, measuring the pertinent physical properties on the ATEM, 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 System Manual or other established laboratory procedure. ATEM analysts must attain an average accuracy of better than 80% true positives (≤20% false negatives) and less than 10% false positives on quality control samples.

The responsibility of the quality assurance supervisor is to define and maintain the laboratory Quality System and the associated Quality Assurance Manual. The technical laboratory director (NVLAP Approved Signatory) is responsible for all analyses, and must review all official laboratory reports and quality assurance summaries.

Because of the analytical method used for the analysis of asbestos in air, it is necessary to establish the technical capabilities of the individual analysts. The Quality System must provide for routine checks of ATEM operators which include intraoperator tests (checking the analyst against himself), interoperator tests (checking the operator against other analysts) and tests on standards (Standard Reference Materials [SRM's], Reference Materials [RM's], Proficiency Testing Materials, and internal laboratory standards). The intraoperator tests include training and testing with standards and duplicate analyses. The interoperator tests require multiple operators to perform analyses on the same samples.

Verified Asbestos Analysis is currently the only definitive way to compare results among analysts and check for the accuracy of an analyst on an unknown sample [2]. Verified Asbestos Analysis consists of multiple operators independently analyzing a grid square and comparing results. This should include, if possible, at least one highly qualified analyst with a proven average accuracy of greater than 85% true positives, and less than 5% false positives. This requires that beam currents be low enough that at least two consecutive analysts can observe electron diffraction patterns from the same fiber.

Laboratories may find it advantageous to use up to 4 operators on one analysis to characterize operator performance initially. Multiple analysts from different laboratories can be used, but since the grid is moved between analyses, grid relocation and reorientation becomes necessary and can make cross comparison difficult. Grid squares are counted in the same manner (same grid orientation, same starting point, same initial traverse direction, same traverse pattern) and the size, identification data, location and/or drawing of the structures are recorded. This data is compared for the multiple independent analyses and any questionable structures are reanalyzed and confirmed.

The design, implementation, and evaluation of the operator tests is the responsibility of the laboratory. An example of an operator characterization system follows: 1) standardized results reporting forms are submitted for each analysis, 2) the forms contain fields for sample/grid/square identification, operator, instrument parameters, and for each fibrous structure: morphology type and size, electron diffraction measurements, x-ray measurements, structure type, (for verified analysis the location or drawing of each structure is recorded) 3) the data is tabulated manually or by computer, 4) comparisons are made between operators, and 5) specific deficiencies for each operator are determined. The results of these tests must be appended to the information on the operator which is included in the Quality Assurance Manual (or other location as referenced in the Quality Assurance Manual). The results of tests using proficiency testing materials must also be appended to the analyst's Quality System data file.

Quality System checks should comprise a minimum of 10 percent of the sample analyses. It is the responsibility of the laboratory to schedule the timing and frequency of Quality System checks such that failures of the system can be detected and corrected. New or part-time analysts require a greater percentage of Quality System analyses. It has been noted in the past that the analyst's performance can change (for better or worse) as a function of time and sample. A summary of all Quality System checks performed in the laboratory should be entered into the Quality System records on at least a monthly basis to characterize the laboratory. The summary includes the performance of each operator and subfacility and the performance of the laboratory as a whole.

Comments on FACILITIES AND EQUIPMENT (See Procedures Sec. 7.33c)

The laboratory must have proper facilities for handling, preparation, analysis, and storage of air filters for asbestos analysis and for maintaining safe working conditions while handling bulk asbestos. The list of required facilities and equipment follows. A few items, as marked, are optional.

- NIST or NIST traceable standards for the major asbestos types (SRM 1866), air filter (SRM 1876, optional: RM 8410, RM 8411) and thin film AEM (SRM 2063) detector calibration materials.
- Note: SRM 1866 contains bulk asbestos and therefore, precautions need to be taken against contaminating the filter preparation area with these specimens.
 - Class 100 clean area or bench
 - Exhaust fume hood or equivalent for volatile solvents
 - Filter handling utensils (razor knives, forceps, probe needles, microscope slides, indexed TEM grids, etc.)
 - Filter preparation equipment including modified Jaffe wick equivalent and/or condensation washer
 - The air filter analysis facilities are separate from bulk asbestos (and any other potentially contaminating) facilities
 - Carbon evaporator (and supplies)
 - Gold coating capability
 - Low temperature asher
 - Instrumentation for recording images and electron diffraction patterns (using electron micrographs or other suitable media)
 - Filter and grid storage facility

Transmission electron microscope with the following under routine asbestos analysis conditions:

- Capability of operation at a voltage between 80-120 kV
- Capability of producing an electron diffraction pattern of single fibers of chrysotile that are \geq .5 microns in size
- Capability of displaying and resolving the hollow tube of chrysotile
- Fluorescent screen with calibrated gradations for fiber size and electron diffraction measurement or other similar measurement system
- Mechanical stage with linear, reproducible movements along two perpendicular directions
- Capability of producing a spot at crossover that is \leq 250 nm
- Energy dispersive x-ray spectrometer and multichannel analyzer capability

Comments on SUBFACILITIES (See Procedures Sec. 7.33c)

Main (laboratory) facilities and subfacilities are defined in NVLAP POLICY GUIDE 10, which is included in this Handbook as APPENDIX D. To qualify as a subfacility, a laboratory <u>must be technically dependent</u> on the main facility; technical management and supervision must be provided by the main facility. Quality assurance activities of the subfacility must be directed by the main facility. The nature, scope, and frequency of on-site quality assurance reviews by the main facility Quality Assurance Manager must be clearly defined in the Quality Assurance manual and appropriate for the nature and scope of work performed by the subfacility. Copies of all permanent quality assurance and personnel records must be retained at the main facility. Quality assurancedata from each subfacility must be regularly compared both to the main facility's data and data from other subfacilities. Records of such comparisons must be retained in quality assurance records along with actions taken to evaluate and resolve differences.

Comments on CALIBRATION (See Procedures Sec. 7.33d)

Calibration is important to show the validity of data collected during the analysis of asbestos. Calibration data on known reference samples give a data base from which unknown samples can be analyzed, interoperator and interlaboratory results can be compared, and ideal minimum analytical error can be estimated.

The transmission electron microscope will be calibrated according to the frequency specified in the Quality Assurance Manual. All items required in the equipment list must be checked to ensure that they are in working order. A list of the required calibrations follows.

- Transmission Electron Microscope electron beam alignment
- X-ray detector/multichannel analyzer calibration of eV/channel, resolution at Mn Kα, and relative sensitivity factors for Na, Mg, Al, Si, Ca, Fe and other elements as needed. This calibration includes measurements to show that the system has sufficient sensitivity to determine the presence of Na in standard crocidolite and Mg and Si on a single fibril of chrysotile under routine analytical conditions.
- Electron diffraction camera constant calibration for both the phosphor screen and the electron micrograph
- Magnification calibration for both the phosphor screen and the recording media for images and diffraction patterns (electron micrographs or other suitable media)
- Beam dose calibration to allow observation of chrysotile diffraction patterns for 15 seconds or longer
- Analytical TEM spot size is 250 nm or smaller (spot should be properly astigmated before measurement to ensure that the spot is approximately round)

An x-ray detector's performance on an ATEM can change dramatically as a function of time, often due to damage to the silicon detector or from oil and/or ice contamination of the detector and/or detector window. These problems can cause peak broadening across the spectrum and reduced sensitivity at lower x-ray energies. Since these changes in sensitivity are a function of x-ray energy, they can affect the identification criteria for asbestos. The best way to calibrate the detector and check for these effects is with a reference material of known thickness and composition that yields both low and medium to high energy x-rays, such as found in SRM 2063. The variation in k-factors over time is monitored.

Records of all calibration results, calibration problems, and corrective measures should be recorded and summarized in the Quality System records.

Comments on TEST METHODS AND PROCEDURES (See Procedures Sec. 7.33e)

The laboratory must use the method specified in [3] for the analytical transmission electron microscope analysis of asbestos on air filters. The laboratory must have detailed descriptions of the sample handling, analysis, and reporting procedures as performed in their laboratory. As stated in the comments on Quality System, the laboratory is responsible for tracking sample custody through the receipt and analysis process. Subsamples and TEM grids should be uniquely identified to prevent any mishandling or confusion.

A recommended method for monitoring contamination problems is the use of laboratory blanks. Laboratory blanks, prepared following standard lab practice but using a filter material which contains asbestos well below an acceptable blank level (see checklist), must be analyzed routinely and with increased frequency after actual contamination is discovered and corrected. A minimum frequency of one blank preparation for every filter series (from one site) is required. The frequency of blank analyses must be sufficient to show the validity of any analysis found to be statistically above the laboratory blank level. The method for contamination control must be outlined in the Quality Assurance Manual. Records of all blank analyses and contamination problems and corrective measures should be recorded and summarized in the Quality System records.

During the ATEM analysis of asbestos air filter materials, it has been determined that one of the most common causes of low results or false negatives occurs when the operator fails to find or observe a fiber. This is probably due to the operator missing a whole or partial traverse of the grid square. Another source of error is found in the individual operator's interpretation of asbestos structure counting rules as it is applied to complex structures. The magnitude of these types of errors can be determined using Verified Asbestos Analysis. The verified counting should also increase the intralaboratory precision over time, as all analysts within a laboratory start to count structures in a more uniform manner.

To document the positive identification of asbestos in a sample, the analyst(s) records the following physical properties (as required in the method): morphology data, electron diffraction data, energy dispersive x-ray analysis data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos are recorded. This information must be present in the sample analysis records. The analyst must sign or initial and date each analysis record.

The identification data is collected to prevent or limit false negative and

false positive asbestos structure counts. Fibers are identified by measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might be confused with asbestos.

To show that the laboratory methods are not resulting in false negatives due to misidentification of asbestos as nonasbestos, data must be collected demonstrating that the combination of the laboratory's analytical criteria and instrumental/operator analysis conditions for asbestos identification will correctly classify at least 90% of asbestos structures (both bundles and individual fibrils) in known standard materials (such as filter or grid materials prepared from the bulk asbestos SRM 1866).

The laboratory will be responsible for demonstrating its competence to analyze asbestos samples following the practice outlined in its Quality System manual. Any staff member involved in the analysis of samples will be responsible for demonstrating their competence as required during an on-site visit. In particular, analysts should be able to demonstrate their ability to use and interpret the results from the ATEM in imaging, diffraction, and x-ray analysis modes and identify the various types of asbestos and differentiate asbestos from nonasbestos fibers.

Comments on RECORDS (See Procedures Sec. 7.33f)

A laboratory must maintain a record-keeping system that allows for rapid and easy retrieval of records that contain complete information on the subject. Records containing complete information on the following items are required:

- logging and tracking of samples
- original data collected by the analyst
- contamination checks
- calibrations
- operator characterization
- laboratory characterization
- reports of analysis

This data may exist in different record locations; i.e. Quality Assurance Manual, log book, analysis lab book, calibration lab book, etc., but they must be easily retrievable and their location documented in the Quality Assurance Manual.

Comments on TEST REPORTS (See Procedures Sec. 7.33g)

The following information should be reported for each sample: laboratory identification, area of filter analyzed, volume of air sampled (with reference to sampling data sheet), analytical sensitivity for the analysis, number of total asbestos structures and number of structures by asbestos type, concentration in asbestos structures per square millimeter of filter and asbestos structures per cubic centimeter of air for total asbestos structures and with data broken down by size (≥ 5 and ≥ 0.5 to $< 5 \ \mu$ m) and by asbestos type, statement of analytical error, including laboratory-analyst accuracy/precision and sample variability (this requirement is delayed, pending the acquisition of data from the laboratories), copy of TEM analysis data record, and signature of the Approved Signatory.

A description of any nonstandard preparation and analysis procedures is reported and statements are included about how these procedures may affect the validity of the results. The following additional information must be supplied if asbestos abatement clearance is determined by the laboratory: calculation formula(e), all calculation variables and constants, and all calculation results.

REFERENCES

- [1] Federal Standard 209b (or most recent version), "Federal Standard Clean Room and Work Station Requirements, Controlled Environment", April 24, 1973. Single copies of this Standard are available from General Services Administration, Business Service Centers in Boston, New York, Atlanta, Chicago, Kansas City (MO), Fort Worth, Denver, San Francisco, Los Angeles, and Seattle.
- [2] Steel, E.B. and J.A. Small, Anal. Chem., <u>57</u>, 209-213, 1985.
- [3] Environmental Protection Agency's, "Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions", Appendix A to Subpart E, 40 CFR part 763, October 30, 1987 or the current U.S. Environmental Protection Agency TEM method for the determination of completion of response actions for asbestos.

V. PROFICIENCY TESTING

Proficiency testing is an integral requirement of the NVLAP evaluation process. The proficiency testing program may be conducted entirely by NVLAP, or by a NIST approved contract laboratory for a portion of the program. The proficiency testing materials are expected to be challenging, but representative, examples of air filter samples or their derivatives. They will, in particular, test the laboratories' ability to follow the method and achieve the proper accuracy, precision, and detection limits.

Each laboratory will be sent test samples, data sheets, and an information package containing specific instructions for performing the test and reporting the results. The test should be conducted in accordance with the applicable test method; special NVLAP instructions for preparation and analysis mustalso be followed. The special instructions are designed to ensure uniformity in procedures among participants. Completed data sheets must be returned to NIST or its appointed contractor by the date specified on the sheets.

All analysts (including those in subfacilities) must participate in proficiency testing. Each analyst must separately analyze, record, and report test results and keep their results confidential until all analysts have completed their analyses. A single result is to be reported back to NVLAP by the laboratory. The test results are to be used for interanalyst comparisons and entered into the Quality System records. All proficiency testing materials must be analyzed in-house if the results are reported to NIST; they may not be contracted out to another laboratory.

Unless specifically noted, the laboratories may keep proficiency testing materials for use as in-house instructional materials. However, on occasion, some of the materials used in proficiency testing may be rotated among labs. After testing, they must be returned to NIST for use by other participants. The testing materials must be protected from harm and damage both in the laboratory and during shipment back to NIST. Examples of such materials are TEM grid preparations and photographs. These materials may be used to determine testing performance for specific parts of the test method.

The results of the proficiency testing program will be reported to the participants and in appropriate documents and reports. The identities and performance of individual laboratories will remain confidential. The results of proficiency testing will be made available to on-site assessors for use during laboratory visits. If problems are indicated by proficiency testing, they will be discussed with appropriate laboratory personnel, who will then be responsible for developing and implementing plans for resolving the problems. Participation in proficiency testing is required for initial accreditation and continued accreditation.

VI. ON-SITE ASSESSMENT

Before accreditation can be granted, the laboratory must undergo a successful on-site assessment, or resolve any departures from the NVLAP criteria noted during an assessment.

A NVLAP assessor will arrange with the laboratory in advance for the on-site assessment. The laboratory should be in good order and prepared to demonstrate testing. The assessor will try to minimize disruption to the normal working routine. All observations are held in strictest confidence by NVLAP.

The assessor will use NVLAP checklists containing specific questions about all aspects of the visit. The checklists, based on NVLAP criteria for accreditation, serve to ensure a complete assessment and that all assessors cover the same items at each laboratory.

The laboratory will be responsible for demonstrating its competence to analyze airborne asbestos samples following the practice outlined in its Quality Assurance Manual. Staff members involved in the analysis of samples will be asked to demonstrate their competence, as required, during an on-site visit.

Both the central laboratories and the subfacilities must be included in the assessment if those subfacilities are to be included in the accreditation. An assessment of a central laboratory will normally take two days. Assessments of subfacilities may require one or more additional days depending on their number and location.

The agenda for a typical on-site visit is given below.

- 1. Assessor conducts an entry briefing with laboratory manager to explain the purpose of the on-site visit and to discuss the schedule for the day. At the discretion of the laboratory manager, other staff may attend the briefing.
- 2. Assessor reviews equipment calibration and maintenance records, record keeping procedures, Quality System manual(s), laboratory test reports, and personnel competency records. Although a staff member is available to answer questions, the assessor may wish to review the documents alone. The assessor does not usually ask to take any laboratory documents with him when they leave the laboratory.
- 3. Assessor observes the demonstration of selected procedures and interviews the personnel. The demonstrations should include specimen preparation and the use of all major equipment. The assessor may give the laboratory materials to prepare and/or analyze.
- 4. Assessor physically examines equipment and facilities.
- 5. Assessor examines subfacilities. Laboratory personnel should be available to provide transportation and to accompany the assessor. If the laboratory maintains more than one similar mobile or satellite laboratory, the number to be visited will be based on the number and location of the facilities. The central laboratory must demonstrate that all sites are operated and equipped in the same manner as described in the Quality Assurance Manual.
- 6. The assessor needs time during the day to complete NVLAP paperwork.
- 7. An exit briefing is held with the laboratory manager and staff to discuss the assessor's findings. Deficiencies are discussed and resolutions are mapped out. Items that must be addressed before accreditation can be granted are emphasized. Items that have been corrected during the on-site and any recommendations are specially noted.
- 8. The assessor completes the Assessment Report, to be signed by the laboratory representative. A copy of this report is left at the laboratory, mailed to NVLAP, and kept by the assessor.

APPENDIX A

PART 7 - NATIONAL VOLUNTARY LABORATORY ACCREDITATION PROGRAM PROCEDURES

Subpart D - Conditions and Criteria for Accreditation

- 7.31 Application of accreditation conditions and criteria.
- Conditions for accreditation. 7.32
- 7.33 Criteria for accreditation.

SUBPART D - CONDITIONS AND CRITERIA FOR ACCREDITATION

- Sec. 7.31 Application of accreditation conditions and criteria.
- To become accredited and maintain accreditation, a laboratory must meet the conditions for (a) accreditation set out in Section 7.32 and the criteria set out in Section 7.33 as tailored for specific LAPs.
- The conditions leading to accreditation include acceptance of the responsibilities of an accredited ക laboratory and requirements for information disclosure.
- (c) The criteria are tailored and interpreted for the test methods, types of test methods, products, services or standards of the relevant LAP. These tailored criteria are the technical requirements for accreditation developed through the procedures of Section 7.15.
 (d) In applying the conditions, criteria, and technical requirements for accreditation, the Director of OPSP shall not:
 - - (1) Prohibit accreditation solely on the basis of a laboratory's affiliation or nonaffiliation with manufacturing, distributing, or vending organizations, or because the laboratory is a foreign fim: or
 - (2) Develop, modify, or promulgate test methods, standards, or comparable administrative rules.
- Sec. 7.32 Conditions for accreditation.
- To become accredited and maintain accreditation, a laboratory shall agree in writing to: (a)
 - (1) Be assessed and evaluated initially and on a periodic basis;
 - (2) Demonstrate, on request, that it is able to perform the tests representative of those for which it is seeking accreditation;
 - (3) Pay all relevant fees:

 - (4) Participate in proficiency testing as required.
 (5) Be capable of performing the tests for which it is accredited according to the latest version of the test method within one year after its publication or within another time limit specified by the Director of OPSP:
 - (6) Limit the representation of the scope of its accreditation to only those tests or services for which accreditation is granted; (7) Limit all its test work or services for clients to those areas where competence and capacity are
 - available;
 - (8) Limit advertising of its accredited status to letterheads, brochures, test reports, and professional, technical, trade, or other laboratory services publications, and use the NVLAP logo under guidance provided by the Director of OPSP;
 - (9) Inform its clients that the laboratory's accreditation or any of its test reports in no way constitutes or implies product certification, approval, or endorsement by NBS;

 - (10) Maintain records of all actions taken in response to testing complaints for a minimum of one year;
 (11) Maintain an independent decisional relationship between itself and its clients, affiliates, or other organizations so that the laboratory's capacity to render test reports objectively and without bias is not adversely affected;
 - (12) Report to the Director of OPSP within 30 days any major changes involving the location, ownership, management structure, authorized representative, approved signatories, or facilities of the
 - laboratory; and (13) Return to the Director of OPSP the certificate of accreditation for possible revision or other action should it:
 - Be requested to do so by the Director of OPSP; Voluntarily terminate its accredited status; or (i)
 - 'ii)
 - (iii) Become unable to conform to any of these conditions or the applicable criteria of Section 7.33 and related technical requirements.
- To become accredited and maintain accreditation, a laboratory shall supply, upon request, the (b) following information: (1) Legal name and full address; (2) Ownership of the laboratory:

 - (2) Ownership of the laboratory; (3) Organization chart defining relationships that are relevant to performing testing covered in the accreditation request
 - (4) General description of the laboratory, including its facilities and scope of operation;

Name and telephone number of the authorized representative of the laboratory;

- (6) Names or titles and qualifications of laboratory staff nominated to serve as approved signatories of test reports that reference NMAP accreditation; and (7) Other information as may be needed for the specific LAP(s) in which accreditation is sought.

Sec. 7.33 Criteria for accreditation.

- (a)
- Quality System.
 (1) The laboratory shall operate under an internal quality assurance program appropriate to the type, range, and volume of work performed. The quality assurance program must be designed to ensure the required degree of accuracy and precision of the laboratory's work and should include key elements of document control, sample control, data validation, and corrective action. The quality assurance program must be documented in a quality manual or equivalent (e.g., operations notebook) which is available for use by laboratory staff. A person(s) must be identified as having responsibility for maintaining the quality manual.
 (2) The quality manual must include as appropriate:

 (i) The laboratory's quality assurance policies including procedures for corrective action for detected test discrepancies;
 (ii) Quality assurance responsibilities for each function of the laboratory;
 - - - Quality assurance responsibilities for each function of the laboratory;
 - (ii) (iii) Specific quality assurance practices and procedures for each test, type of test, or other specifically delineated function performed;
 - Specific procedures for retesting, control charts, reference materials, and (iv)
 - interlaboratory tests; and Procedures for dealing with testing complaints. (v)
 - (3) The laboratory shall periodically review its quality assurance system by or on behalf of management to ensure it's continued effectiveness. These reviews must be recorded with details of any corrective action taken.
- (b) <u>Staff</u>. (1) The laboratory shall:
 - Be staffed by individuals having the necessary education, training, technical knowledge, (i)
 - and experience for their assigned functions; and Have a job description for each professional, scientific, supervisory and technical position, including the necessary education, training, technical knowledge, and (ii) experience.

 - (2) The laboratory shall document the test methods each staff member has been assigned to perform.
 (3) The laboratory shall have a description of its training program for ensuring that new or untrained staff are able to perform tests properly and uniformly to the requisite degree of precision and accuracy.
 - (4) The laboratory shall be organized:
 - (i) So that staff members are not subjected to undue pressure or inducement that might influence their judgment or results of their work; and In such a way that staff members are aware of both the extent and the limitation of their
 - (ii) area of responsibility.
 - (5) The laboratory shall have a technical manager (or similar title) who has overall responsibility for the technical operations of the laboratory.
 - (6) The laboratory shall have one or more signatories approved by the Director of OPSP to sign test reports that reference NVLAP accreditation. Approved signatories shall:

 (i) Be competent to make a critical evaluation of test results; and

 - (1) (11) Occupy positions within the laboratory's organization which makes them responsible for the adequacy of test results.
- (c) Facilities and Equipment
 - activities and Equipment.
 (1) The laboratory shall be furnished with all items of equipment and facilities for the correct performance of the tests and measurements for which accreditation is granted and shall have adequate space, lighting, and environmental control, and monitoring to ensure compliance with prescribed testing conditions.
 (2) All equipment must be properly maintained to ensure protection from corrosion and other causes of determents for a procedure procedure for those items of equipment which
 - deterioration. Instructions for a proper maintenance procedure for those items of equipment which require periodic maintenance must be available. Any item of equipment or component thereof which has been subjected to overloading or mishandling, gives suspect results, or has been shown by calibration or otherwise to be defective, must be taken out of service and clearly labelled until it has been repaired. When placed back in service, this equipment must be shown by test or calibration to be performing its function satisfactorily. (3) Records of each major item of equipment must be maintained. Each record must include:
 - - The name of the item of equipment; (ii) (iii) The manufacturer's name and type, identification and serial number; Date received and date placed in service;

 - (iv) Current location, where appropriate;
 - Details of maintenance; and
 - (ví) Date of last calibration, next calibration due date, and calibration report references.

- (d) <u>Calibration</u>. The laboratory shall:

 - (1) Calibrate new testing equipment before putting it into service;
 (2) Recalibrate, at regular intervals, in-service testing equipment with the calibration status readily available to the operator;
 (3) Perform checks of in-service testing equipment between the regular calibration intervals, where
 - relevant;

 - (4) Maintain adequate records of all calibrations and recalibrations; and(5) Provide traceability of all calibrations and reference standards of measurement where these standards exist. Where traceability of measurements to primary (national or international) standards is not applicable, the laboratory shall provide satisfactory evidence of the accuracy or reliability of test results (e.g., by participation in a suitable program of interlaboratory comparison).
- (e) <u>Test Methods and Procedures</u>. The laboratory shall: (1) Conform in all respects with the test methods and procedures required by the specifications against which the test item is to be tested, except that whenever a departure becomes necessary for technical reasons the departure must be acceptable to the client and recorded in the test report;
 - (2) Have data to prove that any departures from standard methods and/or procedures due to apparatus design or for other reasons do not detract from the expected or required precision of the measurement
 - (3) Maintain a test plan for implementing testing standards and procedures including adequate instructions on the use and operation of all relevant equipment, on the handling and preparation of test items (where applicable), and on standard testing techniques where the absence of such instructions could compromise the test. All instructions, testing standards, specifications, manuals, and reference data relevant to the work of the laboratory must be kept up-to-date and made readily available to the staff;
 - (4) Maintain measures for the detection and resolution of in-process testing discrepancies for manual
 - and automatic test equipment and electronic data processing equipment, where applicable; (5) Maintain a system for identifying samples or items to be tested, which remains in force from the date of receipt of the item to the date of its disposal, either through documents or through marking to ensure that there is no confusion regarding the identity of the samples or test items
 - and the results of the measurements made; and (6) Maintain rules for the receipt, retention, and disposal of test items, including procedures for storage and handling precautions to prevent damage to test items which could invalidate the test results. Any relevant instructions provided with the tested item must be observed.
- (f) <u>Records</u>. The laboratory shall:
 - (1) Maintain a record system which contains sufficient information to permit verification of any issued report;
 - (2) Retain all original observations, calculations and derived data, and calibration records for one year unless a longer period is specified; and
 - (3) Hold records secure and in confidence, as required.

(g) <u>Test Reports</u>. (1) The laboratory shall issue test reports of its work which accurately, clearly, and unambiguously Each test report must include present the specified test results and all required information. Each test report must include the following information as applicable:

- (i) (ii) (iii)
- Name and address of the laboratory; Identification of the test report by serial number, date, or other appropriate means; Name and address of client;
- (iv) Description and identification of the test specimen, sample, or lot of material represented;
- (v) Identification of the test specification, method, or procedure used;
- (vi) Description of sampling procedure, if appropriate;
- (vii) Any deviations, additions to, or exclusions from the test specifications;
- (viii) Measurements, examinations, and derived results supported by tables, graphs, sketches, and photographs, as appropriate, and any failures identified; A statement of measurement uncertainty, where relevant; Identification of the organization and the person accepting technical responsibility for
- (ix)
- (x) the test report and date of issue;
- A statement that the report must not be reproduced except in full with the approval of the (xi) laboratory; and A statement to the effect that the test report relates only to the items tested.
- (xii)
- (2) The laboratory shall issue corrections or additions to a test report only by a further document suitably marked, e.g. "Supplement to test report serial number," which meets the relevant requirements of Section 7.33(g)(1).
 (2) The laboratory shall issue corrections or additions to a test report only by a further document requirements of Section 7.33(g)(1).
- (3) The laboratory shall retain a copy of each test report issued for one year unless a longer period is specified by the Director of OPSP.
 (4) The laboratory shall ensure that all test reports endorsed with the NVIAP logo are signed by an
- approved signatory.

APPENDIX B

National Voluntary Laboratory Accreditation Prooran

LAB BULLETIN

Lab Bulletin No. 3A

U.S. Department of Commerce

of Standards and Technology

National Institute

January 1, 1985

INFORMING THE PUBLIC OF YOUR ACCREDITATION STATUS

Summary

This Bulletin supersedes NVLAP Lab Bulletin No. 3 dated October 1, 1981. It reflects significant changes made to the NVLAP procedures (Title 15, Part 7, of the Code of Federal Regulations) which became effective on December 10, 1984.

The Bulletin is addressed primarily to personnel at accredited laboratories who are responsible for communicating the laboratory's accreditation status to clients and the public, through advertising, issuance of test reports, use of the NVLAP logo, etc.

The Bulletin's purpose is to "provide guidance on referencing the laboratory's accredited status, and use of the NVLAP logo by the laboratory and its clients," in accordance with provisions of the NVLAP Procedures.

Background

NVLAP was established to assist industry and government in identifying competent testing laboratories. NVLAP accreditation means that a laboratory is competent to perform specific test methods in selected fields of testing. The NVLAP Procedures are the bases upon which the entire program operates and accomplishes accreditation of laboratories. Parts A and B of the Procedures provide general information and the method by which a new Laboratory Accreditation Program (LAP), in a new field of testing, may be requested and established. Parts C and D of the Procedures , of more concern to accredited laboratories, describe how a laboratory becomes accredited and the conditions and criteria for initial and continued accreditation. This Bulletin is concerned principally with issues in Part D of the Procedures.

Requirements and Guidance

To become accredited and maintain accreditation a laboratory shall: limit the representation of the scope of its accreditation to only those tests or services for which accreditation is granted.

A laboratory accredited by NVLAP may use the following statement on its letterheads and in trade or other publications: "Accredited by the National Bureau of Standards, National Voluntary Laboratory Accreditation Program for selected test methods for --(identify product or service area(s))." This statement could, for example, be placed at the bottom of the laboratory letterhead.

A laboratory's letterhead containing a reference to its NVLAP accreditation may be used in any direct solicitation for business from potential customers. It is recommended that a copy of the NVLAP Certificate and Scope of Accreditation be appended to such a solicitation. To become accredited and maintain accreditation a laboratory shall: limit advertising of its accredited status to letterheads, brochures, test reports, and professional, technical, trade, or other laboratory services publications, and use the NVLAP logo under guidance provided by NBS.

A statement about NVLAP accreditation and the NVLAP logo may be used on reports and data sheets containing test data obtained by a laboratory provided the tests or services are performed in accordance with the terms of its accreditation. The NVLAP logo may not be used on test reports or data sheets during any period of suspended or expired accreditation or after voluntary or involuntary termination of accreditation.

The nature or type of product advertising prohibited by NVLAP procedures includes any advertising that is intended to encourage a consumer to purchase a product because it was tested by an accredited laboratory, whether that advertising appears in consumer media, the business media, or at a point of sale to consumers.

News stories and advertising by laboratories of their accredited status in the trade press is not only permissible but encouraged. The use of advertisements in the trade press is consistent with NVLAP procedures.

The "consumer media" to be avoided include popular periodicals such as Time, Good Housekeeping, etc., and newspapers such as the Washington Post or the New York Times. The term "consumer media" does not include business publications such as Barron's, or the Wall Street Journal which are oriented to the business community and in which products per se normally are not advertised.

To become accredited and maintain accreditation a laboratory shall: inform its clients that the laboratory's accreditation or any of its test reports in no way constitutes or implies product certification, approval, or endorsement by NBS

Laboratory accreditation by NBS confers recognition that a laboratory has been found competent to perform specific test methods or services in a selected field(s) of testing. Laboratories must avoid all inference that accreditation under NVLAP carries with it an endorsement, approval, or recommendation of the products tested by the laboratories.

To become accredited and maintain accreditation a laboratory shall: assure that all test reports endorsed with the NVLAP logo are signed by an approved signatory.

An approved signatory is an officer or employee of the laboratory, identified by name or position, who has been accepted by NVLAP as being responsible for the issuance of test reports under this condition of NVLAP accreditation. A laboratory seeking initial accreditation or reaccreditation must specify (a) one or more individuals, or (b) position(s) within the organization for which it requests acceptance as an approved signatory.

Computer or machine generated test reports that contain the NVLAP logo need not be signed but must have the printed name of the approved signatory.

Questions About Accreditation

If you have questions about what is an acceptable method of advertising in areas not specifically covered in this Lab Bulletin or about the propriety or acceptability of a particular statement, advertising media, or use of information about your NVLAP accreditation status, please contact NVLAP before your publicity program is implemented.



POLICY BULLETIN

Policy Bulletin No. 19

August 1986

SATISFACTORY PROFICIENCY TESTING IS A REQUIREMENT FOR ACCREDITATION

Accreditation by the National Bureau of Standards, under the National Voluntary Laboratory Accreditation Program (NVLAP), requires that a laboratory meet all performance requirements and criteria as determined by on-site assessments and proficiency testing.

If, as the result of on-site assessments, deficiencies are found, the laboratory must satisfactorily resolve those deficiencies, in order to obtain initial accreditation or maintain accreditation.

Unsatisfactory participation in any NVLAP proficiency testing program is a technical deficiency which must be resolved in order to obtain initial accreditation or maintain accreditation.

Unsatisfactory participation in NVLAP proficiency testing programs is defined as, but not limited to, one or more of the following:

- 1. Failure to meet specified proficiency testing performance requirements prescribed by a standard or test method for which the laboratory is seeking accreditation. (Example: ANSI Standard N13.11 for the Dosimetry LAP.)
- Failure to participate in a regularly scheduled "round" of proficiency testing for which the laboratory has received instructions and/or materials.
- 3. Failure to submit laboratory control data as required. (Example: Within laboratory control data to be submitted twice annually for the Concrete LAP.)
- 4. Performance as a statistically outlying laboratory in two successive rounds of proficiency testing or showing a general pattern of outlying test results over three or more rounds.
- 5. Failure to produce test data within acceptable limits of error when testing NBS Standard Reference Materials or special artifacts whose properties are well characterized and known to NBS/NVLAP.

NVLAP will notify the laboratory of proficiency testing deficiency(s) and actions to be taken to resolve the deficiency(s). Denial or suspension of accreditation will result from failure to resolve deficiencies.

APPENDIX D

NVLAP POLICY GUIDE 10

This Policy Bulletin presents NVLAP definitions of the types of laboratory facilities that may be granted NVLAP accreditation, the requirements and conditions that must be satisfied in order to achieve accreditation, and procedures that NVLAP will follow in evaluating various types of facilities for their conformance to accreditation criteria.

Definitions:

a. <u>Main (laboratory) facility</u>:

- permanently (at all times) maintains staff, equipment, procedures, documentation, and facilities necessary to perform the tests, for which it seeks accreditation;
- (2) implements all quality assurance procedures;
- (3) maintains and retains all records, and issues test reports; and
- (4) may be a permanently fixed site or a permanent mobile facility.

b. <u>Subfacility</u> is physically separate from, but considered an extension of, its main facility. Although it may have all staff, equipment, procedures, and documentation necessary to perform the requisite tests, it receives technical direction and quality assurance management from the <u>main facility</u>.

- A permanent subfacility maintains staff, equipment, procedures, documentation, and facilities necessary to perform the tests, for which it seeks accreditation, at all times. It may be a permanently fixed site or a permanent mobile facility and is expected to remain in operation for at least one year.
- 2. A <u>temporary subfacility</u> is provided with staff, equipment, procedures, documentation, and facilities necessary to perform the tests, for which it seeks accreditation, on an interim basis, to meet the needs of the <u>main facility</u>. A <u>temporary subfacility</u> may be established at a fixed site or in a mobile facility and is expected to remain in operation less than one year.

Conditions for Accreditation:

NVLAP accreditation of a laboratory <u>main facility</u> does not extend to accreditation of <u>subfacilities</u> unless the <u>subfacilities</u> have been evaluated separately. These facilities are uniquely identified in the NVLAP accreditation documents. A NVLAP accredited laboratory must not present or report test data, produced at any nonaccredited, <u>subfacility</u> as having been produced under the status of NVLAP accreditation.

NVLAP offers accreditation to laboratories that are found competent to perform specific test methods or types of tests in specified fields of testing. Competence is defined as the ability to meet specific technical criteria relating to quality assurance, staff, equipment, facilities, procedures, records, and reports. Technical criteria may or may not be equally applicable to <u>main facilities</u> and <u>subfacilities</u>. Accreditation of <u>subfacilities</u> may require NVLAP criteria that address the use and maintenance of equipment and facilities, and the implementation of procedures, that are particularly applicable to the performance of specific test methods in <u>subfacilities</u>. NVLAP must develop specific technical criteria upon which to base an objective evaluation of staff, facilities, equipment, and procedures employed in applicable <u>subfacilities</u>.

NVLAP will accredit a <u>main facility</u> if the facility complies with all applicable NVLAP criteria.

NVLAP will accredit a subfacility (in addition to the main facility) if:

- a. the laboratory <u>main facility</u> meets all NVLAP accreditation criteria;
- the laboratory <u>main facility</u> satisfactorily documents and maintains quality assurance procedures addressing the applicable <u>subfacility</u>; and,
- c. the subfacility complies with all applicable NVLAP criteria.

Procedures:

In principle, NVLAP will require that <u>subfacilities</u>, to be included in a laboratory's accreditation, undergo on-site assessments and participate in proficiency testing. NVLAP staff, with the guidance of NVLAP technical experts, will determine the need for and extent of such evaluations based on the number and location of similar <u>subfacilities</u> managed by the laboratory, the nature of the quality assurance system, and any special technical considerations. Decisions on the need for and extent of the evaluations may not be made until after the accreditation of the <u>main facility</u>. The conditions and requirements for evaluation of subfacilities providing specific testing services are described in NVLAP documents pertaining to the relevant accreditation program.

Laboratories seeking NVLAP accreditation should clearly state, on the NVLAP Application Form, what type(s) of <u>subfacilities</u> are to be included in the accreditation. NVLAP fees for on-site assessments and proficiency testing will be based on the number of facilities seeking accreditation that are required to undergo on-sites and participate in proficiency testing. A single administrative/technical support fee is charged to the laboratory (<u>main</u> <u>facility</u>).

<u>APPENDIX E</u>

GENERAL OPERATIONS CHECKLIST

Instructions to the Assessor: This checklist addresses general accreditation criteria prescribed in Section 7.33, Subpart D, of the NVLAP Procedures (Title 15 of the Code of Federal Regulations).

<u>Place an "X" beside any of the following items which represent a deficiency</u>. Place a "C" beside each item which you are commenting on for other reasons. <u>Record the item number and your written deficiency explanations and/or comments</u> <u>on the appropriate comment form(s)</u>. Place a check beside all other items you observed or verified at the laboratory.

Quality System

- ____1. The laboratory operates under an internal quality assurance program appropriate to the type, range, and volume of work performed.
- 2. The quality assurance program is documented in a Quality Assurance Manual or equivalent (e.g., operations notebook) available for use by laboratory staff.
- 3. A person(s) is identified as having responsibility for maintaining the Quality Assurance Manual.

The Quality Assurance Manual includes, as appropriate:

- ____4. Quality assurance responsibilities for each staff position in the laboratory;
- 5. Specific quality assurance practices and procedures for each test or type of test, such as control charts, use of reference materials, and interlaboratory tests;
- 6. Procedures for document control, sample control, data validation, corrective action for detected test discrepancies;
- 7. Procedures for dealing with testing complaints from clients.
- 8. The laboratory periodically reviews its quality assurance system to ensure the system's continued effectiveness and records the review and any specific corrective actions taken to resolve testing deficiencies.

<u>Staff</u>

- 9. The laboratory has a job description for each position, including any required education, training, technical knowledge, and experience.
- ____10. The laboratory documents the test methods each staff member has been assigned to perform.
- 11. The laboratory has a written description of its training program.

- __12. Staff members are not subjected to undue pressure or inducement that might influence their judgement or the results of their work.
- ____13. Staff members are aware of both the extent and limitation of their area of responsibility.
- ____14. The laboratory has a technical manager (or similar title) with overall responsibility for the technical operations of the laboratory.

Facilities and Equipment

- ____15. The laboratory has all necessary items of equipment and facilities for the correct performance of the tests and measurements for which accreditation is being sought.
- 16. All equipment is properly maintained.
- 17. Instructions for proper equipment maintenance are available.
- ____18. Equipment which has been overloaded or mishandled, gives suspect results, or is defective, is taken out of service until repaired.
- ____19. When placed back in service, repaired equipment is shown to be performing satisfactorily.
- ____20. Records of each major item of equipment are maintained including:
 - a. name of the item of equipment;
 - b. manufacturer's name;
 - c. type, identification, and serial number;
 - d. date received and date placed in service;
 - e. current location, where appropriate;
 - f. details of maintenance;
 - g. date of last calibration, next calibration due date, and calibration report references.

<u>Calibration</u> The laboratory:

- ____21. Calibrates new or repaired testing equipment before putting it into service;
- ____22. Calibrates, at regular intervals, in-service testing equipment with the calibration status readily available to the operator;
- ____23. Performs checks of in-service testing equipment between the regular calibration intervals, where relevant;
- 24. Maintains adequate records of all calibrations;
- 25. Provides traceability of all calibrations and reference standards of measurement where these standards exist or satisfactory evidence of the accuracy or reliability of test results.

Test Methods and Procedures The laboratory:

- 26. Performs the test methods and procedures for which accreditation is being sought.
- ____27. Explains departures from standard test methods to clients and records departures in the test report;
- ____28. Has data to prove that departures from standard methods do not detract from the expected or required precision of the measurement;
- ____29. Keeps instructions, testing standards, specifications, manuals, and reference data up-to-date and readily available to the staff;
- 30. Maintains a system for identifying samples or items to be tested;
- 31. Maintains procedures for receipt, retention, and disposal of test items.

<u>Records</u> The laboratory:

- ____32. Maintains a record system with sufficient information to permit report verification;
- ____33. Retains original observations, calculations and derived data, and calibration records for one year unless a longer period is specified;
- 34. Holds records secure and confidential.

Test Reports

35. The laboratory issues accurate, clear test reports.

Test reports include, as applicable, the:

- ____36. Name and address of the laboratory or other appropriate, unique identification;
- ____37. Identification of the test report by serial number, date, or other appropriate means;
- 38. Name and address of client or other appropriate unique identification;
- ____39. Description and identification of the test specimen, sample, or lot of material represented;
- 40. Identification of the test specification, method, or procedure used;
- 41. Description of sampling procedure, if appropriate;
- 42. Deviations, additions to, or exclusions from the test specifications;

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- 43. Measurements, examinations, and derived results supported by tables, graphs, sketches, and photographs, as appropriate, and any failures identified;
- 44. A statement of measurement uncertainty, where relevant;
- ____45. A statement that the report must not be used by the client to claim product endorsement by NVLAP or any agency of the U. S. Government;
- ____46. A statement to the effect that the test report relates only to the items tested.
- ____47. All test reports endorsed with the NVLAP logo contain the signature of an approved signatory.

APPENDIX F

SPECIFIC OPERATIONS CHECKLIST

AIRBORNE ASBESTOS LABORATORY ACCREDITATION PROGRAM

Instructions for the Assessor: This checklist addresses specific accreditation criteria prescribed in Section IV. <u>TECHNICAL REQUIREMENTS</u> of the Airborne Asbestos Analysis Handbook. These criteria <u>do not</u> supercede the <u>Criteria for Accreditation</u>, based on Section 7.33 of the NVLAP Procedures, which are addressed in the CENERAL OPERATIONS CHECKLIST.

<u>Place an "X" beside any of the following items that represent a deficiency</u>. Place a "C" beside each item that you are commenting on for other reasons. <u>Record the item number and your</u> <u>explanation for the deficiency and/or comments on the appropriate comment form(s)</u>. Place a check beside all other items you observed or verified at the laboratory.

QUALITY SYSTEM

- The laboratory's quality assurance analyses represent at least 10% of the total number of analyses performed.
- 2. Quality assurance checks are performed routinely, covering all time periods, sample types, instruments, tasks, and personnel. The selection of samples is semi-random and, when possible, the checks on personnel performance are executed without their prior knowledge.
 - 3. A quality assurance supervisor is designated to define and maintain the Quality System and associated Quality Manual.
- 4. The Quality Manual contains a listing or reference to a listing of:
 - a. staff qualifications and assignments
 - b. equipment
 - 5. The Quality Manual contains the laboratory's procedures and applicable records (or reference to procedures and records) that describe:
 - a. staff training
 - b. routine maintenance checks of the TEM
 - c. alignment of the TEM
 - d. calibration of the following:
 - TEM diffraction camera constant
 - TEM magnification
 - energy dispersive x-ray unit
 - TEM beam dose/chrysotile damage
 - magnification of instrument used for grid opening measurement
 - e. sample handling
 - log in
 - -- unique sample identification system
 - -- criteria for acceptance or rejection
 - sampling documentation must include:
 - -- unique air filter identification
 - -- air volume pulled through filter

-- filter pore size

-- sample chain of custody

Note: Examples of rejection criteria include: insufficient sampling documentation (as above), bulk samples included with air filter samples, filter cassettes open, filters overloaded with particulate, particle loading on filter uneven, sampling parameters do not meet AMERA sampling criteria, filters not uniquely identified, tampering with cassettes evident, sample that laboratory is not capable of preparing properly, etc.

- storage, disposal, and archiving of prepared grids and sample cassettes f. preparation of filters
 - mixed cellulose ester filters, including
 - -- collapsing technique
 - -- etching technique
 - -- carbon coating technique
 - -- filter dissolution technique
 - -- other techniques that may cause fibers to be obscured or lost
 - polycarbonate filters, including
 - -- carbon coating technique
 - -- filter dissolution technique
 - -- other techniques that may cause fibers to be obscured or lost
- g. Evaluation of quality of prepared grids and criteria for acceptance including:
 - fraction of grid openings covered by the replica section (coherent or noncoherent) is greater than approximately 50%
 - the following criteria are relative to the grid squares covered by the replica section:
 - -- fraction of intact grid openings is greater than approximately 50%
 - -- fraction of area of undissolved filter is less than approximately 10%
 - -- fraction of grid squares with overlapping or folded replica film is less than approximately 50%
 - the following general criteria are met for acceptance of grids:
 - -- at least 20 grid squares have no overlapping or folded replica, < 5% holes and < 5% opaque area due to incomplete filter dissolution. 'Opaque area' means that the sample preparation artifact is sufficiently opaque to the electron beam that recognition and analysis of fibers will be difficult or impossible.
 - h. checking for contamination of the following:
 - the filter lot (sealed blank)
 - the field filters
 - the general laboratory
 - all other areas and materials used in the preparation and analysis of air filter samples as needed following evidence of contaminated laboratory blank filters
- ____i. analysis of the type and concentration of fibrous particulate on the specimen grids
 - j. determination of area of grid squares
 - k. methodology for examining a grid square and for counting and analyzing particles
 - (a detailed description is necessary--a copy of EPA method is not sufficient)
 - methodology for recording grid orientation in the microscope
 - particle loading acceptance criteria (>25% by area particulate loading, uneven particle loading are rejected)
 - unique grid and grid square labelling system (indexed grids)
 - grid square traversing method, including the use of orthogonal scans.

Note: The intent is to completely cover the grid square without having structures missed or counted twice. To do this, parallel, overlapping traverses are made across a grid square. Care is taken to move only one translator during a traverse. If an asbestos structure is encountered and stage movement is required for analysis, then the stage is returned to the original traverse position before continuation of the traverse.

- recording rules
- structure counting rules
- criteria for identification of asbestos electron diffraction patterns and rejection of non-asbestos patterns, including those that closely resemble asbestos.

Note: These criteria shall include measurement of indicative d-spacing and symmetry data as necessary.

- criteria for identification of asbestos EDXA spectra and rejection of nonasbestos patterns, including those that closely resemble asbestos
- criteria for differentiating asbestos minerals from at least the following phases shall be documented: the pyroxenes, halloysite, palygorskite, sepiolite, antigorite, lizardite, talc, and vermiculite
- the combination of the laboratory's analytical criteria and instrumental analysis conditions for asbestos identification correctly classify at least 90% of both bundles and single fibrils of asbestos structures $\geq 1 \ \mu m$ in length in known standard materials traceable to NIST (such as the bulk asbestos SRM 1866).
- determination of pass-fail for AHERA clearance
- 1. testing of precision and accuracy of laboratory (and of each microscopist) using at least the following:
 - analyses of reference materials
 - analyses of NIST proficiency testing materials
 - interlaboratory analyses
 - repeat preparation and analysis of same sample by same analyst and by different analyst
 - intermicroscope analyses, if the lab has more than one TEM
 - laboratory blank analyses (not field/sealed blank analyses) and other contamination checks
 - verified analyses (note: minimum of two analysts or two laboratories are required)
 - repeat analysis of the same grid square by the same analyst
 - repeat analysis of the same grid square by a different analyst

Note: The analysis of reference materials tests the laboratories' overall performance, accuracy, and/or precision. Interlaboratory analysis is used to help prevent internal laboratory bias from developing or continuing. Repeat preparation and analysis are used to attain data on the precision of the test method and laboratory results. Intermicroscope tests help prevent instrument- based bias. Data on blank analyses help determine the lab's analytical sensitivity. The rest of the tests check operator precision and accuracy. The balance of the number of each of these analyses is dependent on the laboratory and analyst. Laboratories must first ensure that their analysts are characterizing filters accurately; thus, verified and reference analyses are the highest priority. Labs that have attained an acceptable level of statistical control (see section on verified analysis) for their analysts can move verified and reference analyses to a maintenance level and concentrate on errors associated with the sample preparation (blanks), the natural sample variation (repeat preparation and analysis), and interlaboratory bias. If verified analyses are performed in a random fashion without the knowledge of the initial analyst, then the last two items -- repeat analysis of the same grid square by the same and different analysts -- are redundant and not necessary.

m. methods for verifying report calculations including:

- calculation of area of filter necessary to be analyzed to reach a required analytical sensitivity
- structure per unit volume and filter area calculations
- statistical calculations
- clearance pass/fail criteria calculation
- _6. The Quality Manual contains a schedule (for routine timing and frequency) planned for the following:
 - a. procedures relating to the ATEM including routine checks of system, alignment of the microscope and calibration of ATEM related parameters
 - b. contamination checks using laboratory blanks
 - c. determining the precision and accuracy of each microscopist
 - d. determining overall precision, accuracy, and limit of detection of laboratory results, both by analyst and for the overall laboratory.
 - must be performed for at least 10% of analyses
 - must be summarized at least monthly using control charts
- _7. The Quality Manual contains standardized methods for recording the following:
 - a. log in, criteria for acceptance or rejection
 - b. evaluation of quality of prepared grids
 - c. ATEM sample analysis data
 - d. any other forms used in the laboratory associated with ATEM asbestos analysis
- 8. The maximum allowed contamination levels of filter blanks are:
 - a. for filter lot, laboratory and low temperature asher blanks
 - a cumulative average level of 18 structures per mm-
 - a single preparation level of 53 structures per mm²
 - b. for field blanks
 - 70 structures per m²

9. Frequency for blank level checks

- a. blank preparation
 - a minimum of one laboratory filter blank per sample set or 10% of samples (whichever is greater)
 - prepare field and sealed blanks with each series of samples (if these blanks are identified and known to lab, otherwise prepare all filter samples with the series)
- properly record and archive prepared grids (even if not analyzed)
- b. blank analysis
 - minimium of one laboratory filter blank per 25 filter analyses
 - when air samples are found to be above 70 structures/mm², lab analyzes laboratory blank
 - when full indoor/outdoor analysis is performed, lab analyzes field and sealed

blank (if known to laboratory, otherwise all filters in sample set must be analyzed)

Note: Definitions of blanks are as follows:

- 1. <u>Filter lot blanks</u>: sealed filter from filter manufacturer
- 2. <u>Field filter blanks</u>: field blanks samples are processed by removing the cassette cap for not more than 30 seconds before sampling
- 3. <u>Sealed filter blank:</u> carried with sample series filters through whole process, but not opened during sampling operations
- 4. <u>Laboratory blank:</u> obtained by leaving an unused filter exposed in the clean area while a sample set of filters are prepared
- 5. <u>Other blanks</u>: as needed to determine and correct source of contamination, including TEM specimen holders, evaporators, Jaffe wick, low temperature asher, lab air and fallout samples, etc.

Note: The responsibility of the laboratory for the blanks related to the AHERA sampling depend on their interaction with the sampling organization and whether the laboratory performs the "Z" or compliance test. If the Z-test is performed by the laboratory, then the field and sealed blanks must be known to the laboratory. The laboratory is responsible for the analysis of the filter lot blanks only when contracted to analyze them by the sampling organization.

10. The following documents are available in the laboratory as reference:

- a. NVLAP Airborne Asbestos Handbook
- b. general references on analytical electron microscopy, transmission electron microscopy, asbestos analysis, and crystallography
- c. ATEM manufacturer's operation manual
- d. multichannel analyzer manufacturer's operation manual
- e. Environmental Protection Agency, "Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Normandatory--and Mandatory Section to Determine Completion of Response Actions", Appendix A to Subpart E, 40 CFR part 763, October 30, 1987 or the current U.S. Environmental Protection Agency TEM method for the determination of completion of response actions.

The lab must have references available and be knowledgeable on the following topics, however, the exact reference is not required:

- f. for Verified Asbestos Analysis see E.B. Steel and J.A. Small, Anal. Chem., 57, pp. 209-213, 1985.
- g. for spot size measurement see D.B. Williams, <u>Practical Analytical Electron</u> <u>Microscopy in Materials Science</u>, Philips Electronics Instruments, Inc., Mahwah, New Jersey, 1984, p. 34-35 (for TEM or STEM mode), D.B. Williams, "Standardized Definitions of X-ray Analysis Performance Criteria in the AEM", in A.D. Romig Jr. and W.F. Chambers, Eds., <u>Microbeam Analysis 1986</u>, pg 443-448, San Francisco Press, San Francisco, 1986 (for TEM mode), and J.I. Goldstein, et al., <u>Scarning Electron Microscopy and X-ray Microanalysis</u>, page 48, Plenum Press, New York, 1981 (for STEM mode).
- h. for k-factor measurement see D.C. Joy, A.D. Romig, J.I. Goldstein, <u>Introduction</u> to <u>Analytical Electron Microscopy</u>, Plenum Press, NY, 1986 or <u>Practical Analytical</u> <u>Electron Microscopy in Materials Science</u>, Philips Electronics Instruments, Inc.,

Mahwah, New Jersey, 1984. An example of k-factors applied to asbestos minerals is given in Beaman, D.R. and D.M. File, Anal. Chem., <u>48</u>, 101-110, 1976.

i. reference data on the crystallography and chemical composition of minerals that analytically interfere with asbestos

<u>STAFF</u>

- 11. The laboratory maintains personnel records for each staff member including:
 - a. position description/job responsibilities
 - b. resume of qualifications
 - c. training
 - d. assigned laboratory procedures
 - e. results of periodic quality assurance testing reviews including intraoperator tests, interoperator tests and interlaboratory tests
 - f. accuracy and precision summary data (including verified analysis)
 - g. correction of deficiencies
- 12. Staff position(s) cover the following tasks:
 - a. laboratory sample coordinator
 - b. technical supervisor
 - c. ATEM analyst
 - d. quality assurance supervisor
 - e. other tasks as necessary

Note: One person may perform more than one task.

- ____13. Technical Supervisor shall be qualified to conduct ATEM studies and its application to crystalline materials, be knowledgeable in the field of asbestos analysis including:
 - a. the application of electron diffraction to minerals
 - b. application of energy dispersive x-ray analysis to minerals
 - c. the preparation of filter materials
 - d. the handling, sample preparation, analysis, storage, disposal, Quality System, contamination monitoring and control for asbestos analysis
- ____14. All staff are capable of performing the duties assigned and capable of obtaining help as needed.
- ____15. ATEM analysts must have an average accuracy equal to or greater than 80% of true positives, less than or equal to 20% false negatives, and an average of less than or equal to 10% false positives determined by Quality System tests including verified asbestos analysis on both standard and field samples.
- ____16. Lab shows (with data/records) that the analyst can:
 - a. obtain and measure accurately all required morphology, diffraction, and chemical composition properties on the ATEM
 - b. draw proper conclusions from these data for the identification of asbestos \underline{vs} nonasbestos particles
 - c. accurately determine the concentration of fibers in a filter sample

d. follow the written lab procedures (log in, sample preparation, analysis, etc.) as assigned

<u>IRAINING</u>

- 17. All personnel are trained in performing their specified tasks.
- 18. ATEM analysts are trained in:
 - a. ATEM use, calibration, alignment, electron micrography
 - b. EDXA system, x-ray collection and interpretation
 - c. electron diffraction measurement and interpretation
 - d. asbestos counting rules for single and complex fibers
 - e. asbestos counting methods including:
 - -- grid and grid square selection (nonadjacent, semi-random)
 - -- x-y stage translation and parallel traverses
 - -- stage positioning and repositioning
 - f. asbestos identification including:
 - -- morphology criteria
 - -- electron diffraction criteria
 - -- energy dispersive x-ray criteria
 - g. asbestos mineralogy
 - -- including the chemical composition, crystallography, and associated minerals for the six regulated asbestos minerals and those minerals which closely resemble asbestos
 - h. recognition of acceptable/unacceptable sample preparations
 - i. recognition of sample and instrumental artifacts
- ____19. Training is validated and documented through quality assurance methods such as Verified Asbestos Analysis.
- ____20. Verified Asbestos Analysis is performed routinely by each laboratory with sufficient frequency and on sufficient types of samples to determine each operators initial and continuing analysis performance. Samples that have approximately 1000-5000 structures/mm² shall be used to achieve statistically significant information on new analysts. After initial training, a variety of asbestos loadings including routine AHERA samples are used to validate the analysts' results. The samples include loadings seen in typical AHERA samples up to the 1000-5000 structures/mm² so that the lab will have sufficient data to characterize analyst performance over this entire range of asbestos loadings. At least 1/5th of the verified analyses shall be performed on samples with 1000-5000 structures/mm² loadings. Filter blanks, unless known to be contaminated, shall not be used for verified counting.

NOTE: Verified Asbestos Analysis consists of multiple operators independently analyzing a grid square and comparing results. This requires that beam currents be low enough that at least two consecutive analysts can observe electron diffraction from the same fiber. Verified Asbestos Analysis generally includes at least one qualified analyst with a proven average accuracy of greater than or equal to 85% true positives and less than or equal to 5% false positives. Labs may find it advantageous to use up to 4 operators on one analysis to characterize initial operator performance, if a characterized analyst is not available. Grid squares are counted in the same manner (same grid orientation, same starting point, same initial traverse direction and pattern) and the size, number of structures, identification data, and location or drawing of the structures are recorded. These data are compared for the multiple independent analyses and any questionable structures are reanalyzed and confirmed. A description of Verified Asbestos Analysis is included in E.B. Steel and J.A. Small, Anal. Chem., 57, pp. 209-213, 1985. [The largest problem in verified analysis and cross comparing two or more analyst's fiber data is making sure that the same fibers are being counted and compared. This reference description uses the fiber's absolute location and morphology (size and shape) to define the counted fiber to be compared--an alternative way is to use fiber morphology and orientation, by drawing the fiber].

FACILITIES AND EQUIPMENT

- 21. The laboratory handles, stores, and prepares air filter samples in rooms that are separate from potentially contaminating materials such as samples that may contain bulk asbestos.
- 22. Safe working conditions are maintained including:
 - a. safe handling of asbestos
 - b. use of a fune hood when working with filter-dissolving reagents such as chloroform, dimethyl formamide, acetone, acetic acid, etc.
- 23. The following facilities are available:
 - a. clean room or areas for sample preparation
 - b. electron microscopy facility
 - c. facility for storage of filters, prepared grids
- ____24. The following are available in the clean room or area:
 - a. class 100 (or cleaner) HEPA filtered air under positive pressure
 - b. exhaust hood for safe use of filter dissolution reagents
 - c. laboratory blank (see section on Quality System)
- ____25. The following sample preparation equipment or equivalent is available in the clean room or area:
 - a. condensation washer and/or Jaffe wick with the appropriate reagents and supplies
 - b. tweezers
 - c. scalpel holder, surgical blades
 - d. microscope slides
 - e. double sided adhesive tape
 - f. stainless steel mesh (if condensation washer used)
 - e. lens tissue
 - g. indexed copper 200-mesh TEM grids (also referred to as finder grids). Only grids with uniquely identifiable areas may be used.
 - h. wick material (if Jaffe wick used)
 - i. supply of particle-clean, fiber-free water
 - j. other materials as needed

Note: AHERA method requires that labs have the capability of preparing both mixed cellulose ester (mce) and polycarbonate filters. This is also a necessary requirement to have a working proficiency testing system. NIST cannot supply some labs with mce proficiency testing materials while supplying others with polycarbonate filters.

26. If sample preparation areas outside the clean room or area are to be used for any

purpose, the following conditions are satisfied:

- a. the areas are separate from bulk preparation areas
- b. the areas are monitored for contamination
- c. personnel are instructed in contamination prevention
- _____27. The low temperature plasma asher satisfies the following conditions:
 - a. supplied with oxygen
 - b. allows for control of speed of evacuation and venting air flow in order to minimize disturbance of particles on filter surface
 - c. is not used for bulk samples (asbestos or other)
 - _28. The carbon evaporator attains high vacuum and the following are available:
 - a. spectrochemically pure carbon rods
 - b. a carbon rod sharpener
 - c. gold wire for evaporation (or have sputter coater with gold target)
 - d. controlled venting to atmospheric pressure
- _____29. Electron microscopes have the following under routine asbestos analysis conditions:
 - a. capability of operation at a voltage between 80-120 kV
 - b. capability of producing an electron diffraction pattern of single fibrils of chrysotile
 - c. capability of displaying and resolving hollow tube of chrysotile
 - d. capability of precise fiber length (at 0.5 and 5.0 μ m) and diffraction pattern measurement, regardless of image (fiber or pattern) orientation (often fulfilled through use of a fluorescent screen with calibrated gradations in the form of circles or at least two linear dimensions)
 - e. mechanical stage with linear, reproducible movements along two perpendicular directions
 - f. capability of producing a spot at crossover that is ≤ 250 nm during EDXA analysis
 - g. capability of recording brightfield images and electron diffraction patterns on electron micrographs or on other suitable media

Note: It is recommended, but not required, that the electron microscope is equipped with a holder capable of obtaining zone axis diffraction patterns (either a double-tilt or rotation-tilt holder).

- ____30. An energy dispersive x-ray analyzer system is interfaced to the electron microscope. At a minimum the primary asbestos analysis microscope shall have an EDXA.
- 31. The EDXA unit, under routine analysis conditions, meets the following specifications:
 - a. 175 eV or better resolution at Mn Ka peak
 - b. proven detection of Na peak in standard crocidolite or equivalent

Note: A low background holder may be necessary to meet this requirement

c. capable of obtaining statistically significant Mg and Si peaks from a single fibril of chrysotile

- 32. The multichannel analyzer has the following:
 - a. software capable of obtaining background corrected peak intensity or integral for Na, Mg, Al, Si, Ca, Fe and other elements as needed
 - b. capability of accumulation and display of x-ray spectrum (minimum 0.9-10 kV)
 - c. capability of making a hard copy of x-ray spectrum
- 33. The following standards and certificates are available:
 - a. standard optical grating replica for magnification calibration
 - b. NIST SRM 1876 or traceable standard as available
 - (Note: the reissue of SRM 1876 may not be available until the winter of 1989)
 - c. optional -- NIST RM 8410, 8411
 - d. other laboratory standards for chrysotile, grunerite (Amosite), crocidolite, training and quality assurance standard air samples, etc. -- as in any other method, the laboratory has the primary responsibility for developing or obtaining a set of standards useful for checking the identification of asbestos and the fiber concentration of asbestos on filter materials. These internal standards can be drawn from samples received by the lab or developed by the lab through water filtration of asbestos mixtures or by other methods. The samples then must be well characterized within the lab for use as standards.
 - -- anthophyllite, tremolite, actinolite standards are desirable and will be available from NIST in the future
 - e. calibration material(s) for x-ray system (including SRM 2063 or standard traceable to NIST)
 - f. gold film material for electron diffraction calibration
 - g. capability of recording brightfield images and electron diffraction patterns on electron micrographs or on other suitable media
- ____34. Laboratory is able to record and produce hard copies of images (on electron micrographs or other media) to document:
 - a. visibility of chrysotile hollow tubes and beam damage
 - b. visibility and measurement of electron diffraction patterns in particular chrysotile (002), (004), (110), (020), (130), and (200) reflections
 - c. complex arrangement of fibers
 - d. a range of magnifications from 1,000X to 100,000X in brightfield imaging mode
 - e. a range of diffraction camera lengths to enable accurate diffraction pattern measurement (approximately 20 to 80 cm)
- _____35. The facility for storage of filters and prepared grid allows for the following:
 - a. storage of the unused portions of filters in their cassettes for at least 30 days
 - b. storage of analyzed indexed grids for at least three years

CALIBRATION

Note: All calibrations should be performed with the instrument, stage, sample, x-ray detector and other parameters at routine asbestos analysis conditions (e.g. tilt, apertures, location, specimen height, accelerating voltage, etc.). Control charts of calibration data over time should be kept to record and show the variability of the results.

36. The electron microscope is properly aligned so that the electron beam travels down the

optic center of the column. This includes alignment of the electron gun (translation and tilt), apertures, lenses, etc., as described in the manufacturer's and laboratory's operating manual.

- ____37. The diffraction camera constant(s) of the electron microscope is calibrated using an evaporated gold film with a measured variation of ≤ 5%. The diffraction camera constant is calibrated under the same conditions used for asbestos analysis, and on both the phosphor screen and electron micrograph film and other viewing and recording media as applied in the laboratory for asbestos analysis.
- 38. The magnification of the electron microscope is calibrated using an optical diffraction grating replica to a measured variation of ≤ 5 %. The magnification is calibrated on the phosphor viewing screen, the electron micrograph, or on any other system used for fiber measurement.

Note: Tilting of the viewing screen and specimen grid during fiber measurement, or of the viewing screen during diffraction measurement is not recommended. Laboratories using tilts must demonstrate the required measurement accuracy/precision for all fiber and diffraction maxima orientations.

- _____39. The following parameters of the energy dispersive unit are calibrated:
 - a. x-ray energy vs channel number is calibrated to within ±10 eV.
 - b. the relative sensitivity (k-factors) factors to silicon for elements found in asbestos (Na, Mg, Al, Si, Ca, Fe) (see suggested references)
 - the precision of these k-factors is determined and documented
 - c. the Mg to Fe relative sensitivity factor on the SRM 2063 or other standard traceable to NIST shall be 1.5 or less. This number is stable over time and the variation should be within the statistical limits defined by the x-ray counting statistics
 - d. proven detection of Na peak in standard crocidclite or equivalent

Note: 1) The Na K-lines and Cu L-lines (potentially from the Cu TEM grid) have significant overlap and care must be taken to show that the Na is measured above the Cu L-line background. 2) Relative sensitivity factors and detector resolution are useful for monitoring detector performance. The parameters that are directly or indirectly monitored with these factors relate to quantitative analysis and general detector characteristics such as the detector efficiency, electronics, presence of absorbing layers on the detector window, etc. will be monitored by these measurements.

- 40. The beam dose is calibrated so that beam damage to chrysotile is minimized-specifically so that an electron diffraction pattern from NIST standard chrysotile sample single fibrils ≥ 1 μm in length is stable in the electron beam for at least 15 s.
- ____41. The fluorescent screen of the electron microscope is calibrated so that it is possible to a ± 5% precision:
 - a. determine if a fiber is ≥ 0.5 micrometers
 - b. determine if a fiber is $< \text{ or } \ge 5$ micrometers
 - _42. The spot size of the electron probe used for well resolved x-ray microanalysis is measured and found to be ≤ 250 nm (see reference section).

43. The calibration of the ATEM parameters is performed at a sufficient frequency and under asbestos analysis conditions to show that performance during routine analyses are within the following variations compared to calibrated performance:

a.	alignment	checked by each operator, each use
b.	diffraction camera constant	\leq 5% variation
c.	magnification	\leq 5% variation
d.	energy dispersive unit	± 10 eV/channel x-ray energy calibration; and average relative intensities measured on standard reference material SRM 2063 fall within error expected from counting statistics.
e.	beam dose/damage	90% of standard chrysotile fibrils longer than 1 μ m have diffraction patterns that are visible for \geq 15 s.
f.	spot size	\leq 25% relative variation

_____44. Analysts obtain values for verified analyses (these data are reported on a structures per grid square basis) as follows:

(for x-ray analysis)

- a. true positives ≥ 0.80
- b. false positives ≤ 0.10
- c. false negative ≤ 0.20
- ____45. Laboratory obtains mean analytical results on SRM 1876 that fall within 80% of the 95-95% confidence limits as published on the certificate.
- ____46. The frequency of the verified analyses are, at a minimum:
 - a. training all counts used in reports until verified status is attained
 - b. evaluation minimum of 1 per 200 grid square analyses
- ____47. To calibrate the quality of sample preparation, images of good preparations and of the types of problems that can occur in grid preparation are available for reference.
- ____48. To calibrate the low temperature asher, the time needed to ash a filter section completely has been determined, or a calibration curve for the weight <u>vs</u> ashing time of collapsed mce filter sections has been determined.
- 49. Calibration of grid opening measurement system must be performed. The measurement system attains an accuracy ≤ 5 %.

TEST METHODS AND PROCEDURES

- 50. The laboratory uses Environmental Protection Agency, "Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions", Appendix A to Subpart E, 40 CFR part 763, October 30, 1987 or the current U.S. Environmental Protection Agency update to the TEM method for the determination of completion of response actions.
- 51. Procedures (listed in section 5) are readily accessible to all applicable personnel.
- ____52. Personnel responsible for performing specific tasks are familiar with the laboratory's procedures pertaining to that assignment.

<u>RECORDS</u>

- 53. Records are readily accessible and security is maintained to assure survival of records for a minimum of three years.
- 54. Records are kept of all quality assurance activities for at least 3 years.
- 55. Records are kept of the results, deficiencies, and corrective actions of all procedures outlined in the Quality System, test methods and procedures sections of the handbook and checklists.
- 56. Records are kept of staff qualifications and assignments.
- 57. Records are kept related to staff training and evaluation such that:
 - a. records are kept of true positives, false positives, false negatives obtained by each analyst by verified analysis procedures
 - b. a list of problems leading to false positives and false negatives is compiled
 - c. record of procedures to correct personnel deficiencies are kept, including correction of analyst deficiencies found through Quality System checks.
- 58. Records of major pieces of equipment include information on:
 - a. manufacturer
 - b. model
 - c. serial number
 - d. major components
 - e. location of manuals
 - f. calibration
 - g. maintenance
- 59. Records related to samples include:
 - a. log in, rejection or acceptance of cassette
 - b. chain-of-custody
- ____60. Records related to contamination include results and timing of all checks of the following:
 - a. filter lot blanks
 - b. field blanks

- c. laboratory blanks
- d. all other areas and samples as needed to track contamination
- e. summary of contamination problems and resolution

61. Records related to calibration include:

- a. schedule and personnel responsible for calibration
- b. calibration measurements
- c. calibration results
- d. summary of calibration problems and resolution
- 62. Records related to the ATEM analysis of a filter include:
 - a. general information
 - operator (analyst must sign and date analysis sheet)
 - sample identification
 - client identification
 - date
 - ____b. instrument related information
 - instrument (if more than one available)
 - operating parameters including:
 - -- magnification
 - -- accelerating voltage
 - -- other, as needed to ensure alignment and calibration compliance with requirements
 - c. filter and grid related information
 - filter sampling data sheet as received with sample
 - filter type
 - determination of the average area of a grid square
 - grids prepared and their approximate location on filter
 - evaluation of prepared grids
 - grids and grid squares analyzed

(Note: grids must be archived for at least three years in a logical, retrievable fashion and indexed grids shall be used)

- orientation of grid in TEM
- d. original data records include (for AHERA analysis):
 - structure type (fiber, bundle, cluster, matrix)
 - the number of fibers that are ≥ 0.5 micrometers and < 5 micrometers
 - the number of fibers that are ≥ 5 micrometers
 - classification of structures as chrysotile, amphibole (as grunerite, riebeckite, anthophyllite, actinolite, or tremolite), or nonasbestos
 - measurement results of both EDXA and electron diffraction (at least perpendicular row spacing distance) for at minimum the number of structures identified as amphiboles that correspond to an asbestos concentration on the filter of over 70 structures/mm²
 - measurement results of electron diffraction for at minimum the number of structures identified as chrysotile that correspond to an asbestos concentration on the filter of over 70 structures/mm²
 - documentation of positive electron diffraction <u>or</u> EDXA for all chrysotile asbestos structures corresponding to a concentration on the filter of over 70 structures/mm² and documentation of positive EDXA or measured zone axis diffraction pattern for amphibole structures corresponding to a concentration

of asbestos on the filter of over 70 structures/mm²

- documentation or measurement of results of EDXA and/or measurement of a zone axis electron diffraction for at minimum the number of structures in the nonasbestos class that correspond to a concentration of over 70 structures/mm²

Note: For structures whose qualititative chemical composition is distinct from asbestos, e.g. gypsum, only documentation of the qualitative chemical composition is necessary. For structures that have similar qualitative composition, semiquantitative measurement of composition by EDXA and/or measurement of the diffraction pattern is required.

- micrograph numbers for the required one electron diffraction pattern for every five samples that contain asbestos and for any other patterns taken
- criteria used to classify particles as nonasbestos, that is the property or properties that differentiate it from asbestos

Note: Measurement results include sufficient quantitative data (e.g. x-ray intensities or diffraction maxima geometry) to identify positively asbestos as defined by laboratory identification criteria. Documentation of positive diffraction or EDXA means that the analyst records (e.g. checks off) that these properties visually and/or qualitatively match the lab's identification criteria.

____e. related to report to client:

- concentration of asbestos in structures per mm² on filter and structures per cc in sampled air
- number of asbestos structures counted
- type(s) of asbestos
- area analyzed
- volume of air sampled
- f. related to testing of precision and accuracy of structure counts
- 63. All records related to quality assurance testing are retained including results of:
 - a. analyses of reference materials
 - b. interlaboratory analyses
 - c. repeat preparation and analysis of same sample by same people and by different people
 - d. intermicroscope analyses, if the lab has more than one TEM
 - e. laboratory blank analyses (not field/sealed blank analyses) and other contamination checks
 - f. verified analyses (note: minimum of two analysts or two laboratories are required)
 - g. repeat analysis of the same grid square by the same analyst
 - h. repeat analysis of the same grid square by a different analyst
- _____64. A cumulative record of results from precision and accuracy testing are maintained and are summarized at least monthly.

TEST REPORTS

____65. The following is reported for each sample:

a. Laboratory Identification

- b. area of filter analyzed
- c. volume of air sampled (with reference to sampling data sheet)
- d. analytical sensitivity used for the analysis
- e. number of total asbestos structures and number of structures by asbestos type (chrysotile, grunerite, riebeckite, anthophyllite, tremolite, or actinolite)
- f. concentration in asbestos structures per square millimeter of filter and asbestos structures per cubic centimeter of air for total asbestos structures and with data broken down by size (≥ 5 and ≥ 0.5 to $< 5 \mu m$) and by asbestos type
- DELAYED g. statement of analytical error, including laboratory-analyst accuracy/precision and sample variability

Note: The implementation of section 65g is delayed pending data collection (from NVLAP participating labs) and evaluation by NIST. Guidance will be disseminated in the next year. The guidance will be based on laboratory evaluation of filter variability, analyst's-laboratory accuracy and precision.

- h. copy of ATEM analysis data record with analysts signature or initials
- i. signature of Approved Signatory

____66. The following additional information shall be supplied if asbestos abatement clearance is determined by the laboratory.

- a. calculation formula(e)
- b. all calculation variables and constants
- c. all calculation results
- 67. A description of any nonstandard preparation and analysis procedures is reported.

PROFICIENCY TESTING

- ____68. Analyses not contracted out to another laboratory.
- ____69. Laboratory has written procedure for handling, analysis, and use of NIST proficiency testing materials.
- ____70. Laboratory keeps NIST proficiency testing materials for use as in-house instructional materials, unless otherwise directed.
- 71. All analysts participate in proficiency testing.
- 72. Each analyst separately analyzes, records, and reports test results.
- ____73. One single result is reported back to NVLAP by the laboratory.
- 74. The test results are used for inter-analyst comparisons.
- ____75. Problems indicated by proficiency testing are discussed with appropriate laboratory personnel.
- 76. Plans are developed and implemented for resolving deficiencies.

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