A1. TITLE AND APPROVAL PAGE

QUALITY ASSURANCE PROJECT PLAN
for
Pullman Yard Building Hazard Abatement
FY2018 City of Atlanta Brownfields Community-Wide Assessment Program

Conducted Under
EPA Brownfields Cooperative Agreement Recipient (CAR) No. BF 00D59517-0

Prepared for:

68 Mitchell Street, Suite 5100
Atlanta, Georgia 30303

Prepared by:

Cardno

2000 First Drive, Suite 220
Marietta, Georgia 30062
678.443.1197

Submittal Date:
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Signature Approval:

Keith Ziobron

Cardno Project Manager:

Signature

Keith Ziobron, P.E. April 18, 2019

Printed Name / Date

Roger B. Register

Cardno QA/QC Reviewer:

Signature

Roger Register April 18, 2019

Printed Name / Date

Assessment EPA Project Officer/ EPA Designated Approving Official (DAO):

Signature

Derek Street

Printed Name / Date

City of Atlanta Brownfields Program Director:

Signature

Jessica Lavandier

Printed Name / Date

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A3. DISTRIBUTION LIST

The following individuals will receive copies of the approved Quality Assurance Project Plan (QAPP) and any subsequent revisions:

- Derek Street, City of Atlanta Brownfields Community-wide Assessment Project Officer & EPA Designated Approving Official (DAO), EPA Region 4, Sam Nunn Federal Center, 61 Forsyth Street Southwest, RCR Division, Brownfields Section, 10th Floor, Atlanta, Georgia 30303-8960, Phone: 4404.562.8519, Email: street.derek@epa.gov

- Camilla Warren, City of Atlanta Brownfields Revolving Loan Fund (RLF) Project Officer & EPA Designated Approving Official (DAO), EPA Region 4, Sam Nunn Federal Center, 61 Forsyth Street Southwest, RCR Division, Brownfields Section, 10th Floor, Atlanta, Georgia 30303-8960, Phone: 404.562.8574, Email: warren.camilla@epa.gov

- Jessica Lavandier, Authorized Representative and Brownfields Program Manager, City of Atlanta, 68 Mitchell Street SW, Atlanta, GA 30303, Phone: 404.330.6000, Email: jlavandier@atlantaga.gov

- Roger Register, Quality Assurance/Quality Control Officer, Cardno, 2000 First Drive, Suite 220, Marietta, Georgia 30062, Phone: 850.556.1369, Email: r2br2rr@gmail.com

- Keith Ziobron, P.E., Cardno Project Manager, 2000 First Drive, Suite 220, Marietta, Georgia 30062, Phone: 678.443.1197, Email: keith.ziobron@cardno.com

- Douglas Strait, P.E., Cardno Assistant Project Manager, 2000 First Drive, Suite 220, Marietta, Georgia 30062, Phone: 678.433.1194, Email: douglas.strait@cardno.com

- Maureen Meulen, Atomic Entertainment, LLC (Atomic) Project Manager, 120 Rogers Street NE, Atlanta, GA 30317, Phone: 323.739.3999, Email: maureen@atomicent.com

- Field Project Manager / Field Team Leader to be determined

- Field QA/QC Officer to be determined

- Ioana Pacurar, Project Manager, Analytical Environmental Services, Inc. (AES), 3080 Presidential Drive, Atlanta, GA 30340, Phone: 770.457.8177, Email: ipacurar@aesatlanta.com

A4. PROJECT/TASK ORGANIZATION

Cardno was selected by the City of Atlanta (City) as their Qualified Environmental Professional (QEP) and is responsible for preparing the QAPP for the asbestos and lead-based paint abatement at Pullman Yard and assisting the City in programmatic support services and grant management activities under their Environmental Protection Agency (EPA) Cooperative Agreement Recipient Number BF 00D59517-0. A project organization chart is included in Appendix A. The following are the individuals participating in the project and their specific roles and responsibilities:

Derek Street, EPA Region 4 City of Atlanta Brownfields Community-wide Assessment Grant Project Officer/DAO - The EPA Project Officer is responsible for overseeing and monitoring the City's Community-wide Assessment grant. As part of that responsibility, he ensures the processes described in the work plan are followed and the terms and conditions of the grant are met. The Brownfields Region 4 Quality Assurance Manager's DAO provides technical assistance to the Region 4 Project Officer working on Brownfields sites. The DAO's role is to provide technical reviews of QAPPs and QAPP Addenda that are generated. This includes the approval of this QAPP and any revisions.

Camilla Warren, EPA Region 4 City of Atlanta Brownfields RLF Grant Project Officer/DAO - The EPA Project Officer is responsible for overseeing and monitoring the City's RLF grant. As part of that responsibility, she ensures the processes described in the work plan are followed and the terms and conditions of the grant are met. The DAO's role is to provide technical reviews of the QAPPs and QAPP Addenda that are generated. This includes the approval of this QAPP and any revisions.

Jessica Lavandier, City of Atlanta Brownfields Program Manager – She is responsible for the overall strategic direction of the project and ensures project activities are executed in accordance with the approved Work Plan and the Terms and Conditions of the Cooperative Agreement.

Keith Ziobron, Cardno Project Manager – He is the primary decision maker for the project and the primary user of the data to determine whether or not further action is required at the site. He will also coordinate the project activities and his specific responsibilities are:

1. Approving the QAPP and subsequent revisions in terms of Brownfields specific requirements;
2. Overall responsibility of the investigation;
3. Coordinating field and laboratory activities;
4. Conducting project activities in accordance with the QAPP and work order;
5. Validating field data;
6. Reporting to the GA EPD Project Manager and the City's Brownfields Program Director regarding the project status per the work order and preparing interim and final reports to GA EPD and the City;
7. Making final project decisions with the authority to commit the necessary resources to conduct the project;
8. Instituting corrective actions for problems encountered in the field sampling activities;
9. Communicating corrective actions to the Field Team Leader to remedy problems encountered in the field and coordinating with the lab director to correct any corresponding problems encountered in the chemical analyses;

10. Compiling documentation detailing any corrective actions and providing them to the GA EPD Project Manager.

Doug Strait, P.E., Cardno Assistant Project Manager – He will perform the following duties:

1. Assist the project manager with the QAPP and subsequent revisions in terms of Brownfields specific requirements;
2. Instituting corrective actions for problems encountered in the field sampling activities;
3. Communicating corrective actions to the Field Team Leader to remedy problems encountered in the field and coordinating with the lab director to correct any corresponding problems encountered in the chemical analyses;
4. Compiling documentation detailing any corrective actions and providing them to the GA EPD Project Manager.

Roger Register, Cardno QA/QC Reviewer – He provides documentation audits and technical review to assist in promoting, implementing, and documenting QA compliance. The Cardno QA/QC Reviewer is isolated from the implementation Cardno Project Manager. This allows lateral support as a peer to the Cardno Project Manager without introducing unintentional biases from conducting the work.

Maureen Meulen, Atomic Project Manager – She is the owner of the Pullman Yard facility and manages the redevelopment of the property. She will be the main point of contact between the City of Atlanta and the contractors performing the cleanup work. As the recipient of the RLF loan, she is responsible to ensure the processes described in the work plan are followed and the terms and conditions of the loan and grant are met.

Field Project Manager/Field Team Leader (To Be Determined) – They will perform the following duties:

1. Provide periodic oversight of field activities.
2. Review and enforce field sampling procedures and protocols for work performed by others.
3. Audit contractors relative to Davis-Bacon Act compliance.
4. Upon receipt from the Project Manager, make available the approved QAPP documents and subsequent revisions to the members of the field sampling team.
5. Report problems in the field to the Project Manager.
6. Implement corrective actions in the field as directed by the Project Manager. Corrective actions will be documented in the field logs and provided to the Project Manager.

Field QA/QC Reviewer (To be Determined) – They will provide documentation audits and technical review to assist in promoting, implementing, and documenting QA compliance during field activities, laboratory reports, and of deliverables generated from field activities. The Field QA/QC Reviewer is isolated from the implementation from the Field Project Manager. This allows lateral support as a peer to the Field Project Manager without introducing unintentional biases from conducting the work.

Laboratory Director –The Laboratory Director is responsible for the following:

1. Coordinating the analysis of the samples and the laboratory validation of the data;
2. Coordinating the receipt of the samples at the laboratory, selecting the analytical team, ensuring internal laboratory audits are conducted per the Laboratory's Quality Assurance Manual (QAM), and

distributing the applicable sections of the QAPP and subsequent revisions to members of the analytical team;

3. Instituting corrective actions for problems encountered in the chemical analyses and reporting laboratory problems affecting the project data to the Cardno Project Manager and Cardno QA/QC Reviewer. Corrective actions for chemical analyses will be detailed in a QA report that will be provided via electronic and conventional mail.

A5. PROBLEM DEFINITION/BACKGROUND

The City of Atlanta (City) received a United States Environmental Protection Agency (EPA) Brownfields Assessment Grant in 2017 (BF 00D59517). This funding is being used in part to prepare the appropriate documents for the cleanup of the Pullman yard property which is to be partially funded with the City's EPA Brownfields RLF Grant. The cleanup is on one parcel of property totaling approximately 26.84 acres developed with 11 vacant buildings. Majority of the buildings are warehouse or former industrial buildings, with large open spaces. The western and northern portions are paved and/or gravel/parking areas, with the buildings on the north portion of the site. A former railroad spur traverses the western portion of the subject site. The remaining portion of the site is undeveloped wooded land. The subject site is illustrated by the Site Location Map included as Appendix B

Historically, the subject site (Subject Site or Subject Property) has a long industrial history, primarily associated with its connection with the railroad. Beginning in the early 1900s, the site was a chemical process equipment manufacturer. In the 1920s, the subject site was operated by the Pullman Company, a manufacturer of passenger railcars. In the 1960s, the subject site operated as an iron and metal manufacturer as well as a manufacture of railcars until the 1980s. In 1990, the subject site was acquired by the Georgia Building Authority (GBA) where it was decommissioned and has remained vacant since.

Numerous environmental assessments have been conducted at the property since 2006, including multiple hazardous material surveys to identify asbestos and lead-based paint throughout the buildings. The most recent Phase I ESA was conducted in June 2017 by United Consulting, Inc. (United) and identified the following recognized environmental conditions (RECs):

- The past industrial operations on the Subject Site, dating back to 1904.
- The adjacent Warehouse Partnership property with a historic release.

Based on prior environmental findings, and United's soil and groundwater sampling data, the Subject Site was placed within the Georgia EPD's Brownfield Program in May 2017 with the purchase of the property by Atomic Entertainment Development, LLC (Atomic). As part of the application into the Georgia EPD Brownfield Program, a Prospective Purchaser's Corrective Action Plan (PPCAP) was completed on behalf of Atomic by United in May 2017. The PPCAP outlined potential cleanup options, specifically the excavation and removal of metal and SVOC impacted soils. A PPCAP Addendum No. 1 was completed and approved by the Georgia EPD in August 2017. The PPCAP and PPCAP Addendum references the need for asbestos and lead-based paint abatement.

United completed three Hazardous Material Surveys between June 2017 and May 2018 to assess the on-site structures for asbestos and lead-based paint (LBP). Numerous building materials were identified as asbestos containing, including roof flashing, caulking, sealant, floor tiles, pipe wrap, etc. Numerous painted surfaces were identified as lead containing, including steel beams and columns, various walling, and windows. The latest sampling report, dated May 1, 2018, summarized all prior sampling data and developed a data package with diagrams showing locations of impacted materials. This report is included in Appendix C.

The purpose of this cleanup is only to abate and remove the asbestos and/lead-based paint located throughout the buildings. Grant funding for the actual cleanup will be provided via the City's Brownfields RLF grant using hazardous substance funding. Air monitoring and clearance sampling will be conducted on the Subject Site to evaluate the effectiveness of the engineering controls and work practices during removal of ACM and work involving lead, as well as to document the standard of cleaning.

A6. PROJECT/TASK DESCRIPTION AND SCHEDULE

Based on the findings of the previous investigations and the existence asbestos and lead-based paint throughout the building materials of the on-site structures, the following section provides recommended actions to be conducted as part of the asbestos and lead-based paint abatement prior to the redevelopment of the property. Air monitoring and clearance sampling as well as general observations will be made to aid in the decision making process.

Task 1: Asbestos and lead-based paint abatement

Prior to beginning the field investigation, separate Site Health and Safety Plans (HASP) for Cardno personnel and contractors retained by the owner will be prepared to meet the requirements of the Occupational Safety and Health Administration (OSHA) Standard 1910.120. This document will outline potential hazards, the level of personal protection to be used, and the procedures to be followed for monitoring and emergency situations at the subject site.

It is assumed that the fieldwork will be performed in Level C personal protection equipment (PPE, i.e. air purifying respirator, Tyvek suite, cut resistant gloves, steel-toed boots, hard-hats, safety glasses, and hearing protection). Personal and area air monitoring will be conducted for asbestos and lead to verify the need for PPE.

The proposed scope of work will be completed in conjunction with the approved plans from the property owner (Atomic Entertainment, Inc.) and in keeping with negotiated requirement of the Georgia State Historic Preservation Office (SHPO).

For asbestos, it is anticipated that the scope of work will include the removal of identified ACM within asbestos regulated areas consisting of a combination of non-contained regulated areas, contained regulated areas with filtration, and contained negative pressure enclosures. Air sampling will be performed to document representative airborne fiber concentrations. Upon evaluation of completion of ACM removal, air clearance sampling will be performed to assist with documenting completeness of cleaning.

For lead-based paint, it is anticipated that the scope of work will include the removal of the identified LBP within regulated areas consisting of a combination of non-contained regulated areas and contained regulated areas. Air sampling will be performed to document representative airborne lead concentrations. Upon evaluation of completion of LBP removal, lead dust sampling will be performed to assist with documenting completeness of cleaning.

This QAPP establishes minimum requirements for asbestos and lead-based paint abatement, air monitoring, and air sampling, and the adherence to an asbestos and/or lead-based paint abatement work plan/design should be followed with specific details for evaluated asbestos and lead work areas, abatement activities, and documentation of completeness of cleaning.

Task 2: Additional Considerations

The previous lead-based paint sampling investigations conducted by United identified painted surfaces above the EPA and US Housing and Urban Development (HUD) Guidelines, Chapter 7, 1997 Revision which identified lead-based paint as containing equal to or exceeding one milligram per square centimeter (1.0 mg/cm^2) or 0.5% by weight. Numerous samples were identified by United that contained lead below this reporting limit but above the detection concentration. There are OSHA regulations and requirements which should be taken into consideration during any renovation and demolition activities that may disturb any concentration of lead containing building materials or paint.

Although additional testing of painted components is not necessarily needed, the extent of painted components to be impacted and the future use of the areas should be considered so as to minimize the quantity of lead contaminated paint to be removed and to identify alternative methods to address lead paint hazards for certain portions of the Subject Site.

There is potential for data gaps to be identified with respect to the identified asbestos and/or lead-based paint which would need to be addressed prior to disturbance during removal activities. However, for the purpose of this report, it is anticipated that no additional design phase investigation is necessary. Additional testing may be required during cleanup if conditions warrant. However, the level and degree of characterization completed to date appear sufficient to provide basis for the completion of this report and quality assurance of sampling methodologies and requirements needed.

Schedule

The City's Brownfields RLF grant will have a general schedule that will guide the process for this assessment. The field activities are anticipated to commence within 90 days of the final QAPP approval. Sample collection and fieldwork associated with the asbestos and lead-based paint should take anywhere from 3 - 12 months depending on redevelopment plans. Due to the nature of air monitoring, results will be received as early as possible. If the results indicate that asbestos and lead contamination are in excess of permissible limits, the Field Team leader will notify the Cardno Project Manager.

A7. QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT

The following seven steps are used to determine the criteria for project specific data quality objectives (DQO) when performing cleanup projects funded under the City's Brownfields RLF grant.

1) State the Problem:

   Asbestos and lead-based paint identified throughout the on-site structures. This contamination impacts the Subject Site's building materials, and has the potential to harm human health and the environment.

2) Identify the Decision

   Abate asbestos and lead-based paint prior to renovation and/or demolition.

3) Identify Inputs to the Decision

   - Previous asbestos and lead-based paint inspections conducted at the Subject Site
   - Historical records and documents

4) Define the Study Area Boundaries

   Asbestos and lead-based paint material locations are included in United's Hazardous Material Survey Addendum 2 as Appendix C.

5) Develop a Decision Rule

   Proceed with asbestos and lead-based paint abatement.

6) Specify Limits on Data Gaps/Errors

   Limits on data gaps and errors associated with analytical sampling are specified throughout this document. There is potential for data gaps to be identified with respect to the previous reports and the identified asbestos and/or lead-based paint which would need to be addressed prior to its disturbance during removal activities. In addition, data gaps were identified in the cost estimates for the abatement in that different subcontractors providing different scopes of work.

7) Optimize Design

   The optimized design and sampling requirements will be discussed in an abatement design plan(s) as outlined by others.

A8. SPECIAL TRAINING REQUIREMENTS/CERTIFICATIONS

This section outlines the minimum training requirements for personnel conducing project activities. Current training records and certificates are kept in personnel files located at the respective headquarters of the project personnel. Deficiencies and the need for new training are identified during annual personnel evaluations. Personnel deficient in any of the following requirements will not conduct project activities.

Hazardous Waste Operations and Emergency Response (HAZWOPER)

The Field Team Leader will ensure that all on-site project personnel have current certificates of training for the 40-hour Occupational Safety and Health Administration (OSHA) HAZWOPER Training Class with annual 8-hour refresher courses. All personnel mobilizing to the site shall carry a Certificate of Training identification card.

Field Team Training

Field Team Technicians are provided hands-on training in graduated phases of explaining, observing, demonstrating, and performing field sampling techniques and standard operating procedures by experienced field personnel. Additional training in field equipment technologies, quality assurance, ethics, and other skills are provided through in-house instruction, online, and external workshops and courses. Field competency is checked through personnel evaluations with direct input from the Field Team Leaders and project managers.

Certifications

- Asbestos and lead-based paint abatement activities will be performed by a Georgia-licensed asbestos contractor that utilizes workers and supervisors who are trained in accordance with OSHA Asbestos Standard for Construction (29 CFR 1926.1101) for Class I, II, and III activities and a Georgia-Certified Lead Services Firm that employs Georgia-Certified Lead-Based Paint workers and supervisors.
- The asbestos and lead-based paint air monitoring will be conducted by a qualified sub-contractor to Cardno.
- Lead-based paint clearance sampling will be conducted by a Georgia-Certified Lead Services Firm that employees Georgia certified Lead Inspectors and/or Risk Assessors.
- The selected laboratory, Analytical Environmental Services, Inc. (AES), will perform the analysis of the environmental samples in compliance with any and all applicable regulations and standards.

If other personnel (City, EPA, RLF contractors, etc.) are to visit the Subject Site during cleanup activities, then they will ensure their Brownfields program personnel have at a minimum an OSHA 40-Hr HAZWOPER training certification. If they are to enter any regulated contained areas, then additional training certifications may be required. All training certifications will need to be verified as a pre-requisite for site visit(s).

A9. DOCUMENTATION AND RECORDS

All technical documents and records will be maintained in accordance with the requirements set forth in the US EPA Region 4, Science and Ecosystem Support Division (SESD), "*Field Branches Quality System and Technical Procedures*"(http://www.epa.gov/region04/sesd/fbqstp). Some of the required documentation includes the following:

- Facility study plan (scope of work)
- Original chain of custody records and field log books/notes
- Records obtained during the cleanup
- Field notes with field crew signatures or initials on all records/notes
- Record of use of field sampling and decontamination supplies, and equipment tracking
- Progress/status reports
- Correspondence directly-related to the project
- Data validation/quality assessment reports
- Completion reports
- Project audit and QA/QC reports

Chain-of-custody records accompany all samples from origin through disposal. All sample containers are labeled with sample location identification (ID), preservative, sampler name, analyses required, and date/time of collection. The sample location ID is linked to the labels, chain-of-custody, and field notes. The chain-of-custody record typically includes the following information:

- Project name and address
- Date and times of sample collection
- Name of sampler
- Sample location ID
- Number of samples
- Analyses required with preservation method
- Timeframe (days) sample results are needed

Field notes are recorded during all site visits and typically include:

- Names of personnel, subcontractors, and others on-site
- Date and chronological summary of field activities
- Ambient conditions
- Sample location descriptions, sample ID
- Sampling equipment
- Field decontamination procedures

- Field calibration records
- Types of quality control samples collected
- Sampler signature
- Results of QC checks
- Documentation of all problems encountered in the field with corrective action resolution

The laboratory typically provides electronic copies of the analytical results generally within 14 days of sample receipt. Paper copies will be supplied by the laboratory upon request or will be printed from the electronic copy by the Field Project Manager. Upon receipt, Laboratory Data are reviewed by the Field Project Manager and Field QA/QC Officer. The electronic copy is placed in the project file maintained on the server, which is routinely “backed-up” to ensure data integrity.

The laboratory analytical report will be submitted to the Field Project Manager. The narrative report will include a minimum of the following required information:

- The dates of sample receipt, preparation, and analysis
- The condition of the samples upon receipt
- Sample preparation and analysis
- Any problems encountered during sampling, handling, storage, preparation, or analysis, and their solution
- Any variance from the standard operating procedures
- And a discussion of the quality of the reported analytical data

Project records will include all correspondence, field logs and data sheets, laboratory analytical reports, audit findings, progress reports, and a final report. The final report will be submitted to the City and EPA Region 4 Brownfield Project Officer/Manager by the Field Project Manager.

The Field Project Manager will submit the final report to the City of Atlanta within fifteen (15) days of receipt of laboratory data or as described in the final Site Specific QAPP schedule. The report will include the analytical data report, documentation of field activities, a summary of all collected field data, a written report of the audit of field activities (see Section C1 below), and copies of the original field data worksheets for this project. The narrative report will include at least discussions of all field activities, any problems encountered and their solutions, any divergences from the QAPP procedures, and a discussion of field data quality. The report typically include the following components:

- Executive Summary
- Introduction/Background
- Site Description and Physical Setting
- Site Investigation (Scope, Methodology)

- Analytical Results
- Summary and Conclusions

The laboratory will manage the original raw data and data validation report for projects in both hard copy and electronic format. This information will be made available to the Field Project Manager or Field QA/QC Officer upon request. The Laboratory Director will maintain information on where the records are stored, and will identify who will be responsible for records management and how long specific types of records or documents will be maintained.

B1. SAMPLING DESIGN PROCESS

Asbestos and Lead-Based Paint Abatement

The precise sampling design for the asbestos and lead-based paint abatement is independent of final redevelopment plans. This QAPP establishes minimum requirements for sampling; however, the site-specific asbestos and/or lead-based paint work plan/design should be followed which will provide specific details for sampling.

In order evaluate the effectiveness of engineering controls and work practices around the Subject Site, daily air monitoring samples will be collected using 0.8-µm, 25-mm diameter MCEF cassettes that have been specifically designated for asbestos analysis by the manufacturer, at a minimum in the following locations: inside containment, outside containment, negative air exhaust, and clean room of the decontamination system. The numbers and locations may vary depending upon actual conditions observed in the field.

Baseline, daily, and post-response action final clearance sampling will be collected in accordance with the prepared asbestos cleanup design in accordance with EPA National Emission Standard for Hazardous Air Pollutants (NESHAP) and EPA 40 Code of Federal Regulations (CFR) Part 763 Appendix A to Subpart E. The baseline, daily, and final clearance air samples collected are considered critical.

The number and volume of air samples will be in accordance with the following and defined in the abatement design plan issued by Atomic’s consultants and include at a minimum the following:

1. Air samples to establish a baseline before start of work.
2. Representative daily samples will be collected inside and outside each contained regulated work area.
3. During abatement activities, periodic personal air monitoring will performed to evaluate personal exposure.
4. After asbestos abatement, a visual clearance will be conducted by an AHERA-accredited Project Supervisor.
5. Final clearance sampling will be conducted when applicable.

The following air samples will be collected to establish a baseline before start of work:

- Location sampled – Within each contained regulated work area and adjacent to the work area
- Minimum number samples – Five (5)

- Analytical method – NIOSH 7400
- Minimum Detection Limit (Fibers/cc) – 0.005
- Minimum Volume (L) – 400
- Flow Rate (LPM) – 10.0

From start of work through the project decontamination, representative daily samples will be collected inside and outside each contained regulated work area:

- Location sampled – Inside and outside of each contained regulated work area
- Number samples – 4-6
- Analytical Method – NIOSH 7400
- Detection Limit (Fibers/cc) – 0.01
- Flow Rate (LPM) – 10.0

If interior airborne fiber counts exceed 0.1 f/cc, additional samples will be taken as necessary to monitor fiber levels.

If any air samples taken outside of the contained regulated work area exceeds the 0.01 f/cc then abatement contractor will be required to immediately and automatically stop all work and take remedial action.

During abatement activities, periodic air monitoring will be performed to evaluate the personal exposure to asbestos and lead-based paint on abatement workers and supervisors. For each abatement activity, personal air sampling will be performed to verify the level of PPE is adequate. The number and volume of personal air monitoring will be further detailed in an abatement work plan/design.

After asbestos abatement, a visual clearance will be conducted by an AHERA-accredited Project Supervisor. The visual clearance will consist of verifying the adequate removal of ACM and the absence of visible emissions in accordance with ASTM E1368.

After visual clearance of asbestos abatement, final clearance sampling will be conducted inside each contained regulated work area. Final clearance samples will be collected using 0.45-μm, 25-mm diameter MCEF cassettes that have been specifically designated for asbestos analysis by the manufacturer.

- Location sampled – within each work area
- Minimum number of samples – 13
    - Five samples per abatement contained regulated area
    - Five samples per ambient area position at locations representative of the air entering the abatement site
    - Two field blanks to be taken by removing the cap
    - A sealed blank with each sample set
- Analytical Method –TEM AHERA
- Criteria Limit –70 Str./mm^2

- Minimum Volume (L) 1,200
- Flow Rate (LPM) – 10.0

Final Clearance sampling following lead-based paint abatement will be completed by a licensed Lead Inspector in accordance with HUD Guidelines for Evaluation and Control of Lead-Based Paint Hazards Chapter 15 Clearance. Visual clearance will be completed to verify the removal of LBP and absence of visible dust and potential lead emissions, followed by wipe sampling for the presence of dust per EPA & HUD clearance criteria via ASTM 1728. The number of samples are dependent on Table 15.1 of the Chapter 15, and typically require the following:

- Floors –minimum of four per building with four or more rooms, plus within 10 feet of each contained regulated area
- Window sills or window trough – minimum of four per building with four or more rooms

Criteria are listed as follows:

- Floors – 40 µg/ft^2
- Interior window sills – 250 µg/ft^2
- Window troughs – 400 µg/ft^2
- Exterior concrete – 800 µg/ft^2

The number and volume of clearance sampling will be further detailed in an abatement design plan.

B2. SAMPLING & ANALYTICAL METHOD REQUIREMENTS

The following asbestos and lead-based paint sampling requirements may apply.

Parameter	**Method**	**Air**				
		Min Volume	Flow Rate	Container	Preservation	Hold Time
Asbestos	NIOSH 7400	> 400 L	0.5 – 16 LPM	0.8 um MCE	N/A	30 days
Asbestos	NIOSH 7402	> 400 L	0.5 – 16 LPM	0.45 um MCE	N/A	30 days
Lead	NIOSH 7300/7303	> 35 L	1 – 4 LPM	0.8 um MCE	N/A	30 days

N/A = Not Applicable MCE = Mixed Cellulose Filter LPM = Liters Per Minute

Matrix	Parameter	Method	Container	Preservative	Hold Time	Min. Volume
Building Materials	Asbestos	PLM – EPA/600/R-93/116*	Plastic	None	30 days	20 – 40 ml
Building Materials	Asbestos	NIOSH 7402	Plastic	None	30 days	20 – 40 ml

Matrix	Parameter	Method	Container	Preservative	Hold Time	Min. Volume
Paint Chips	Lead	NIOSH 7300 ICP	Plastic	None	28 days	20 – 40 ml
Wipe Samples	Lead	NIOSH 7082M ICP	Plastic	None	28 days	20 – 40 ml
Soil	Lead	SW6010D	Glass jar	None	28 days	4 oz

*Appendix E to Subpart E of 40 CFR Part 763 and EPA/600/900/R-93/116

Any investigation derived waste (IDW) generated during this assessment that may be characterized as hazardous waste (disposable personal protection equipment, etc.) will be containerized and properly labeled until appropriate analytical tests are conducted to determine its waste characterization. IDW generated (building materials, etc.) on site that is characterized as non-hazardous will be disposed of as non-hazardous municipal waste or returned to the source if doing so does not endanger human health or the environment or violate federal or state regulations. Any identified containerized hazardous waste that is stored on site will be manifested and shipped to a permitted treatment and/or disposal facility. All management of IDW will be conducted in accordance with EPA Region 4 SESDPROC-202-R3 SOP, included in Appendix D.

B3. SAMPLE HANDLING & CUSTODY REQUIREMENTS

Field and laboratory personnel will be aware, at all times, of the need to properly maintain all samples, whether in the field or in the laboratory, under strict chain of custody protocols and in a manner to retain physical sample properties and chemical composition. The handling and transportation of the samples will be accomplished in a manner that not only protects the integrity of the sample, but also documents sample custody. In general, packing, marking, labeling, and shipping of samples will be conducted in accordance with the SOP shown in Appendix E (*EPA, Region 4, Field Sampling Procedures: Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples, SESDPROC-209-R2, February 15, 2015*). Samples will be packed and shipped in accordance with applicable and current US Department of Transportation (DOT) regulations and/or International Air Transport Association (IATA) standards. The following sections detail sample handling and custody requirements from sample collection to final delivery to the certified laboratory.

Upon collection, samples will be transferred immediately from the sampling device into appropriate laboratory-supplied containers. All samples collected will have discrete sample identification numbers. The unique sample identifications are necessary to identify and track each of the many samples collected for analysis during the duration of the project. Whenever possible, sample labeling procedures from previous investigations will be followed or continued. Sample collection containers used during field activities will be labeled with unique sample numbers.

Samples will be packaged in a manner to prevent breakage or cross contamination during shipping. A chain of custody form will be completed for each set of collected samples. The purpose of the COC procedure is to prevent misidentification of samples, prevent tampering of the samples during shipment and storage, allow easy identification of tampering, and allow for easy tracking of possession. If the chain of custody is broken at any time from sample collection through analysis, the Field Project Manager and Field QA/QC Officer will be notified.

When collection samples leave the sampler's immediate control (e.g. shipment to laboratory), the sampler will sign and date the chain of custody form(s) to relinquish the samples. The chain of custody form will be placed into a sealable bag. A custody seal will be placed on shipping containers when applicable. The custody seal will bear the collector's name and the date signed. The custody seal is used to ensure that the samples in the shipping container have not been tampered with, therefore ensuring sample integrity. If samples are delivered by the sampler directly to the laboratory, the custody seal may not be used.

B4. ANALYTICAL METHODS AND REQUIREMENTS

Samples collected under the scope of this project will be submitted for laboratory analysis of constituents as specified in Section B2.

Due to the nature of the abatement work, asbestos and lead-based paint air monitoring and clearance sampling results will be received as early as possible from the laboratory. Remaining samples collected will likely be subject to a standard laboratory turnaround time of five (5) business days.

B5. FIELD QUALITY CONTROL REQUIREMENTS

Quality control in the field will be conducted in accordance with the following SOP, shown in Appendix F: *EPA, Region 4, Quality System Procedures: Field Sampling Quality Control, SESDPROC-011-R4, April 16, 2017.*

Due to the nature of abatement work, quality control requirements includethe following:

Field Duplicate Samples: A field duplicate is a second sample collected at the same location as the original sample and will be used to assess sampling and laboratory precision. Duplicate air samples will be collected simultaneously or in immediate succession, following identical collection procedures, and treated in the same manner during sample shipment, storage, and analysis. The sample containers will be assigned an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel. Field duplicate samples will be collected at a one-to-twenty ratio.

Field Blank Samples: A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected. For asbestos and lead air sampling, field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated.

For asbestos clearance sampling, field blanks are required by 40 CFR Part 763 to be collected at the following locations for each regulated contained work area:

- Near the entrance to each abatement area
- In one ambient area

Sealed Blank: A sealed blank is a sample that is prepared in the laboratory to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected. For asbestos and lead-based paint clearance sampling, the sealed blanks are to be carried with each sampling set and not to be opened in the field.

Relevant asbestos and lead laboratory quality assurance manual (QAM) documentation are included as Appendix G.

B6. LABORATORY QUALITY CONTROL REQUIREMENTS

The selected laboratory, Analytical Environmental Services, Inc. (AES), will follow quality control procedures at all times for samples to be analyzed. Relevant asbestos and lead laboratory quality assurance manual (QAM) documentation are included as Appendix G.

Additional considerations that identify each parameter and matrix based on AES laboratory quality control requirements are included below.

Lead and Lead-Based Paint

Matrix	**Parameter**	**Method**	**Laboratory Control Spike (LCS) Range**	**Relative Percent Different**	**Matrix Spike (MS) Range**	**Relative Percent Difference**
Paint	Lead in Paint	NIOSH 7082	80-120%	25%	75-125%	25%
Air	Lead in Air	NIOSH 7300/7303	80-120%	20%	N/A	N/A
Solid - Wipes	Lead in Wipes	NIOSH 7082M	80-120%	25%	N/A	N/A
Soil	Lead in Soil	SW6010D	80-120%	20%	75-125%	20%

Asbestos

Data Quality Indicator	Measurement Performance Criteria	QC Sample and/or Activity Used to Measure Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Bulk Samples			
Precision-Laboratory (TEM)	≥80% true positive ≤20% false negative ≤10% false positive	Verified Analysis	A
Precision-Laboratory (PLM)	AES participates in Proficiency Testing through NVLAP and in Inter-laboratory Round Robin analyses.	Inter-analyst QC	A
Accuracy/Bias (PLM/TEM)	Vendor-specific Limits	Standard Reference Manuals	A
Accuracy/Bias - Contamination (PLM/TEM)	Asbestos < QL	Method Blanks	A
Data Completeness (PLM/TEM)	Laboratory 95%	Data Completeness Check	A
Precision-Laboratory (PLM)	Monthly precision summary reports are generated for each analyst.	Intra-analyst QC whereby 20% of samples are rechecked by secondary analyst. Additionally, 5% of samples are analyzed as blind samples to determine conformity of results.and tabulated on a Blind Recount Form.	A
Air Samples			
Precision-Laboratory (TEM)	TEM: ≥80% true positive ≤20% false negative ≤10% false positive	Verified Analysis	A
Accuracy/Bias (PCM)	Vendor-Specific Limits	Daily Reference Sample	A
Accuracy/Bias-Contamination	Asbestos<QL	Media Certification Check	A
Data Completeness (PLM/TEM)	Laboratory 95%	Data Completeness Check	A

Laboratory QC	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible	Data Quality Indicator
Bulk Samples					
Method Blank (PLM/TEM)	One per day	Asbestos < Quantification Limit	Re-clean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/ Bias Contamination
Inter-analyst QC (PLM)	Not less than semi-annual participation in Inter-laboratory Round Robin testing.	Vendor-specific limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ Bias
Standard Reference Materials (PLM/TEM)	TEM: Annually PLM: 3 samples per week/analyst	Vendor-specific limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ Bias
Intra-analyst QC (PLM)	20% PLM	PLM: RPD≤100	Reanalyze and qualify data	Analyst and Data Validator	Precision
Laboratory Duplicate (PLM)	One per 20 samples	PLM: RPD≤100	Reanalyze and qualify data	Analyst and Data Validator	Precision
Air Samples					
Method Blank (TEM)	One per 10 samples	Asbestos<QL	Re-clean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/ bias-Contamination
Replicated Analysis (TEM)	One per 10 samples	TEM: ≥80% true positive ≤20% false negative ≤10% false positive	Reanalyze and qualify data	Analyst and Data Validator	Precision
Daily Reference Sample	One per day	Vendor-specific limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ bias

B7. FIELD EQUIPMENT AND CORRECTIVE ACTION

An inspection checklist and initial calibration check will be completed by a field team member upon arrival at the site, prior to the commencement of any site investigation activities. A maintenance kit, which will include extra batteries, calibration standards, and commonly needed spare parts, will be made available at the site.

Any preventive or corrective maintenance completed will be documented in the field notes. If any equipment fails the initial testing and inspection, a second attempt to calibrate the meter will be performed. If any equipment fails the second calibration attempt, spare equipment can be obtained from inventory or rented from an environmental sampling supply vendor.

Due to the nature of abatement work, additional field equipment checks are listed below.

For air sampling, a leak check will be completed on air sampling systems by activating the air pump with the closed sampling cassette in line. Any flow indicating a leak must be eliminated before initiating the sampling operation.

All air sampling equipment pumps must be calibrated to accurately assess the pump flow rate in liters per minute (L/min) to ensure adequate flow rate for the desired sampling parameter. Pump calibration will be calibrated before and after each use, and should be calibrated with a field rotometer. Field rotometers will be calibrated 1-2 times per year with a primary standard calibration device. Pump calibration will follow protocols as established in EPA Method 7400. A sample cannot be analytically analyzed if calibration is not adequate.

B8. LAB EQUIPMENT AND CORRECTIVE ACTION

The selected laboratory's (AES) QAM/LQM addresses the testing, inspection, and maintenance for the analytical instruments. Relevant asbestos and lead laboratory quality assurance manual (QAM) documentation are included as Appendix G.

B9. ANALYTICAL SENSITIVITY AND PROJECT CRITERIA

Analytical method sensitivity and project criteria for the analytical methods within the scope of this project is determined by the selected laboratory, AES. Minimum detection limits for asbestos and lead will comply with the applicable regulations as required. Additional considerations that identify each parameter, analytical reporting and detection ranges, and relevant project criteria are included in the following table:

Matrix	Parameter	Analytical Reporting Limit Range	Analytical Detection Limit Range	Project Criteria
Paint	Lead Paint NIOSH 7082	0.01 wt%	0.0016 wt%	EPA and HUD definition of LBP
Air	Lead Air NIOSH 7300/7303	0.2 ug total	0.0591 ug total	OSHA PEL
Solid - Wipes	Lead Wipes NIOSH 7082M	5 ug total	0.9061 ug total	EPA, HUD, and GA EPD Lead Clearance

Matrix	Parameter	Analytical Reporting Limit Range	Analytical Detection Limit Range	Project Criteria
Soil	Lead – SW6010D	5 mg/kg	0.187 mg/kg	Georgia EPD Notification Concentration; EPA and HUD soil hazard levels
Solid	Asbestos PLM - EPA600/R-93/116	<1%	N/A	EPA AHERA
Solid	Asbestos Bulk PLM- 400 Point Count	<0.25%	N/A	EPA AHERA
Solid	Asbestos TEM Semi-quant	<1%	N/A	EPA NESHAP
Air	Asbestos TEM AHERA	.005 s/cc	N/A	EPA NESHAP
Air	Asbestos PCM - NIOSH 7400	~ 7 fibers/mm^2 volume dependent	N/A	OSHA PEL

Relevant asbestos and lead laboratory quality assurance manual (QAM) documentation are included as Appendix G.

B10. DATA MANAGEMENT AND DOCUMENTS

Data for this project will be produced in two locations: onsite and at the laboratory. Data collected onsite will be recorded on field data worksheets and into field logbooks, which will become a part of the project file. These documents and records are also maintained in accordance with the requirements set forth in the US EPA Region 4, Science and Ecosystem Support Division (SESD), "*Field Branches Quality System and Technical Procedures*". A sample of some of the required documentation includes the following:

- Field personnel signatures or initials on all records/notes with a waterproof pen.
- Use of field sampling and decontamination supplies and equipment are tracked with an in-house system.
- Sampling containers are prepared by the laboratory and shipped with a packing list documenting contents.
- Preservatives used by the laboratory are traceable by preparation date, vendor, and lot number.
- Sampling containers are pre-cleaned at the laboratory.
- All equipment is maintained and calibrated in accordance with manufacturers' specifications.

All relevant observations or digressions from the procedures in this QAPP, deemed notable by any field team member, will also be recorded in the field logbook. The Field Project Manager will submit copies of the field data worksheets and logbooks with the field activity report when field activities are complete as a deliverable, when applicable; or as part of the final report.

The laboratory provides electronic copies of the analytical results generally within 14 days of sample receipt. Paper copies will be supplied by the laboratory upon request or will be printed from the electronic copy by the Field Project Manager. The Field Project Manager and Field QA/QC Officer will be responsible for reviewing the data to verify its usability, ensuring the analytical report meets requirements, and for forwarding it to the EPA Project Officer, when applicable.

After the laboratory report is reviewed by the Field Project Manager and Field QA/QC Officer, data is then formatted into tables and compared to regulatory limits to determine if contamination is present at the subject property. Upon completion of formatting of the Analytical Data Table, the data will be reviewed for accuracy by the Field QA/QC Officer. Site figures and maps including analytical results and sample locations may be prepared for submittal with final reports. These figures and maps are also reviewed for accuracy by the Field QA/QC Officer.

AES will manage the original raw data and data validation report for projects in both hard copy and electronic format. This information will be made available to the Field Project Manager or Field QA/QC Officer upon request. The Laboratory Director/QA/QC Manager will maintain information on where the records are stored and will identify who will be responsible for records management and how long specific types of records or documents will be maintained.

Atomic's selected contractor's project records will include all correspondence, field logs and data sheets, laboratory analytical reports, audit findings, progress reports, and a final report. The final report will include copies of field notes and logs, analytical laboratory results, a summary of activities completed with any deviations from the approved QAPP, conclusions, and recommendations and will be submitted to the MCDA and EPA Region 4 Brownfields Project Officer by the Field Project Manager.

All records and reports and checklist from the USEPA Region 4 Designated Approving Official will be stored in the physical project file located at Atomic's selected contractor's main office. The project file will be eventually archived for a minimum period of ten (10) years.

C1. ASSESSMENT AND RESPONSE ACTIONS

This type of assessment includes asbestos and lead-based paint abatement. These assessments may be performed to determine if hazardous materials remain on-site and have been properly abated. This is determined through visual inspections, personal air monitoring, air sampling, clearance sampling, risk-assessment, and corrective action.

The Field Project Manager will meet at least weekly with the Field QA/QC Officer and field team personnel to discuss any problems and ensure that all planned samples are being collected. The Project Manager will review data on a weekly basis, observe sampling, and determine corrective actions, if warranted.

Contract laboratories will participate in Performance Evaluation studies at least twice yearly and satisfy NELAP requirements. Personnel responsible for data assessment will check the results of every sampling event for precision and completeness. Technical and/or quality system audits of environmental or laboratory contractors may be initiated on a prescribed schedule or on an as-needed basis in response to identified or suspected problems. Assessment and response actions will be documented and submitted to the EPA Region 4 Brownfields Project Officer. Identified deficiencies will be followed up with written corrective action plans prepared by the Field Project Manager.

C2. PROJECT REPORTS

Execution of proposed field activities will not commence until this Site Specific QAPP is approved by the EPA.

The Field Project Manager will prepare a closeout report, which will be reviewed for technical accuracy and data quality by the Field QA/QC Officer or similar senior technical staff (as appropriate). The closeout report will include a summary description of project activities, a summary of all data, the field activity report, a discussion on any problems encountered during the project and the corrective actions taken, a discussion of the conclusions drawn from the results and the rationale for those conclusions, and the results of the data quality assessment. The closeout report will be distributed to the project team. The report will then be reviewed for conformance with internal document standards. Final reports will be forwarded to the EPA Project Officer and Ms. Jessica Lavandier of the City of Atlanta Department of City Planning.

D1. FIELD DATA EVALUATION

At a minimum, field data will be evaluated in accordance with the following SOP, shown in Appendix F: *EPA, Region 4, Quality System Procedures: Field Sampling Quality Control, SESDPROC-011-R4, April 16, 2017.* The Field Project Manager will validate the field data and discuss any problems identified during the project with the Field Team Leader. Data will be reviewed for integrity by checking all field entries for errors and consistency. Data validation will be accomplished through a series of checks and reviews intended to assure that the reported results are of a verifiable, reproducible, and acceptable quality.

A data usability review that includes an assessment of field procedures (including field notes, boring logs, field screening results, and field analytical data) completeness, comparability, representativeness, precision, and bias (accuracy) of the data will be performed by the Field Project Manager. The findings of this review will be documented and presented in the final report.

If verification or validation indicates that samples have been collected and/or analyzed out of compliance with the QAPP (for instance deviations from the acceptance criteria for quality control defined in this QAPP and its addendums), resampling may be required. The Field Project Manager must contact the EPA Project Officer in the event that there are any deviations from the QAPP and the Brownfields EPA Project Officer will determine if the data is acceptable or if resampling is required. If data is accepted that deviates from the QAPP, the data will be used for screening purposes only and annotated as such.

D2. LABORATORY DATA EVALUATION

The Laboratory Director/QA/QC Manager will review and verify the laboratory data generated under their corrective action system for accuracy according to the laboratory's QAM/LQM, as detailed in Section B8 of this document. Quality control checks are performed on field data by reviewing the chain of custody forms and results from the lab for each sampling event. All sample results will be reviewed and correlated to field measurements and observations. The validation process will include:

- Narrative review
- Quality control blanks meet criteria
- Appropriate preservatives were used and hold times were met
- Quality control data (spikes, duplicates) are acceptable
- Surrogate spike recoveries are acceptable
- Unacceptable data are identified and corrective actions are initiated
- Data qualifiers are assigned (by lab) if necessary:

In addition to evaluating data qualifiers associated with laboratory analyses, a comparison of the sample duplicate(s) and the corresponding sample result(s) will be made to evaluate the reproducibility of the sample results based on the laboratory analysis and sample collection and transportation procedures. For this comparison, if the duplicate or sample result is less than five (5) times the reporting limit then the comparison is made by the absolute difference between the results (S-D). If these differences are within two times (2X) the "acceptable" limits, they are considered "slightly high"; anything beyond that would be considered "high". If both sample and duplicate results are greater than five times (5X) the reporting limit (the higher of the two RLs, if they're not the same), then precision is assessed by the %RPD (difference in results divided by the average of the two results X 100); <35% RPD = “good/acceptable”, >35% but < 50% = variability is “slightly high”, >50% = “high”.

Based on the data qualifiers provided by the laboratory, and on the sample/sample duplicate comparison described above; data will be categorized as fully quantified, qualified, or unusable. Unusable data will not be utilized in the project decision process. Raw data will be included in all submitted project reports.

The Field QA/QC Officer will perform verification and validation of laboratory data for conformance with the data objectives stated in this QAPP. Data verification will include completeness, correctness, and conformance evaluations. Data validation will be performed to assess the quality and usability of the data generated. Data verification and validation will be performed in accordance with EPA’s “*Guidance on Environmental Data Verification and Validation”* (EPA QA/G8), dated November 2002. Results of the data verification and validation, including potential influence on the data quality will be summarized in the final report.

Typical validation activities include the following:

Item	Activity
Data Deliverables and QAPP	Ensure that all required information on sampling and analysis was provided (including planning documents).
Analytes	Ensure that required lists of analytes were reported as specified.
Chain-of-Custody	Examine the traceability of the data from time of sample collection until reporting of data. Examine chain-of-custody records against contract, method, or procedural requirement.
Holding Time	Identify holding time criteria, and either confirm that they were met or document any deviations. Ensure that samples were analyzed within holding times specified in method, procedure, or contract requirements. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made (consistent with procedural requirements), and that approval to proceed was received prior to analysis.
Sample Handling	Ensure that required sample handling, receipt, and storage procedures were followed, and that any deviations were documented.
Sampling Methods and Procedures	Establish that required sampling methods were used and that any deviations were noted. Ensure that the sampling procedures and field measurements met performance criteria and that any deviations were documented.

Item	Activity
Analytical Methods and Procedures	Establish that required analytical methods were used and that any deviations were noted. Ensure that the QC samples met performance criteria and that any deviations were documented.
Data Qualifiers	Determine that the laboratory data qualifiers were defined and applied as specified in methods, procedures, or contracts.
Deviations	Determine the impacts of any deviations from sampling or analytical methods and SOPs. Consider the effectiveness and appropriateness of any corrective action.
Sampling Plan	Determine whether the sampling plan was executed as specified (i.e., the number, location, and type of field samples were collected and analyzed as specified in the QAPP).
Sampling Procedures	Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (e.g., techniques, equipment, decontamination, volume, temperature, preservatives, etc.).
Co-located Field Duplicates	Compare results of collocated field duplicates with criteria Established in the QAPP.
Project Quantitation Limits	Determine that quantitation limits were achieved, as outlined in the QAPP and that the laboratory successfully analyzed a standard at the QL.
Confirmatory Analyses	Evaluate agreement of laboratory results.
Performance Criteria	Evaluate QC data against project-specific performance criteria in the QAPP (i.e., evaluate quality parameters beyond those outlined in the methods.).
Data Qualifiers	Determine that the data qualifiers applied were those specified in the QAPP and that any deviations from specifications were justified.
Validation Report	Summarize deviations from methods, procedures, or contracts. Include qualified data and explanation of all data qualifiers.

D3. *DATA USABILITY AND PROJECT VERIFICTION*

Analytical data generated in accordance with approved methodologies will be considered definitive and quantitative based on the results and findings of the validation process.

The Field Project Manager will validate the field data and discuss any problems identified during the project with the Field Team Manager. Any problems and associated corrective actions will be documented in the field logs and the final report. The Field Project Manager will discuss any problems along with proposed corrective actions with the Field QA/QC Officer.

Because data generated with significant deviations from the requirements of the QAPP will be rejected and because of the nature of the work (biased sampling), all data will have the same expected uncertainties and there will be no limitations on data use. The following is a list of considerations for data usability assessment:

Item	Assessment Activity
Data Deliverables and QAPP	Ensure that all necessary information was provided, including but not limited to validation results
Deviations	Determine the impact of deviations on the usability of data.
Sampling Locations, Deviations	Determine if alterations to sample locations continue to satisfy the project objectives.
Chain-of-Custody, Deviation	Establish that any problems with documentation of custody procedures do not prevent the data from being used for the intended purpose.
Holding Times, Deviation	Determine the acceptability of data where holding times were exceeded.
Damaged Samples, Deviation	Determine whether the data from damaged samples are useable. If the data cannot be used, determine whether resampling is necessary.
PT Sample Results, Deviation	Determine if the implications of any unacceptable analytes (as identified by the PT sample results) on the usability of the analytical results. Describe any limitations on the data.
SOPs and Methods, Deviation	Evaluate the impact of deviations from SOPs and specified methods on data quality.
QC Samples	Evaluate the implications of unacceptable QC sample results on the data usability for the associated samples. For example, consider the effects of blank contamination.
Matrix	Evaluate matrix effects (interference or bias).
Meteorological Data and Site Conditions	Evaluate the possible effects of meteorological (e.g., wind, rain, temperature) and site conditions on sample results. Review field reports to identify whether any unusual conditions were presented and how the sampling plan was executed.
Comparability	Ensure that results from different data collection activities achieve an acceptable level of agreement.
Completeness	Evaluate the impact of missing information. Ensure that enough information was obtained for the data to be useable.
Background	Determine if background levels have been adequately established (if appropriate).
Critical Samples	Establish that critical samples and critical target analytes/COCs were collected and analyzed. Determine if the results meet criteria specified in this QAPP.
Data Restrictions	Describe the exact process for handling data that do not meet PQOs (i.e., when measurement performance criteria are not met). Depending on how those data will be used, specify the restrictions on the use of those data

Item	Assessment Activity
	for environmental decision-making.
Usability Decision	Determine if the data can be used to make a specific decision considering the implications of all deviations and corrective action.
Usability Report	Discuss and compare overall precision, accuracy, representativeness, comparability, completeness, and sensitivity for each matrix, analytical group, and concentration level. Describe limitations on the use of the project if criteria for data quality indicators are not met.

Field modifications regarding sampling analysis may be necessary for circumstances such as auger refusal, limited access areas, or when enough sample volume could not be collected for various reasons. Re-sampling may be necessary if results are deemed unacceptable for various reasons such as exceeding laboratory holding times or to confirm previous sampling and/or excavation activities, etc. These variables will be further defined throughout this QAPP based on the specific contaminants of concern. Upon receipt of the laboratory data, the data will be reviewed to verify its usability. Upon determination, data is then formatted into tables and compared to regulatory limits to determine if concentrations of COCs exceed CTLs at the subject property. Upon completion of formatting the Analytical Data Table; data will be reviewed for accuracy by the Field QA/QC Officer.

The Field QA/QC Officer will evaluate the usability of individual sample results at the parameter level. Analytical results will be evaluated based on sensitivity criteria described through this QAPP. Data limitations will be documented along with how the data should be used. Conclusions and recommendations drawn from all assessment information will be documented in the final report. Site figures and maps including analytical results and sample locations are frequently prepared for submittal with final reports. These figures and maps are also reviewed for accuracy by the Field QA/QC Officer.

Usable data will be tabulated and compared to applicable GEPD and EPA target concentrations. Concentrations which exceed these targets will be highlighted for easy identification. The Field QA/QC Officer will compare and review the laboratory data to the table for completeness, correctness, and accuracy. Usable data will be provided on site figures and other graphical representation and will also be reviewed for completeness, correctness, and accuracy.

The Field Project manager will conduct an overall project evaluation using the field and laboratory evaluations, tabular and graphical data presentations, and analytical sensitivity criteria to determine its value in developing the site conceptual model and assist with the decision making process.

LIST OF ABBREVIATIONS

ABCA	Analysis of Brownfields Cleanup Alternatives
AOC	Area of Concern
ASTM	American Society for Testing and Materials
bgs	Below Ground Surface
BS	Blank Spike
BSD	Blank Spike Duplicate
BSA	Brownfields Site Assessment
BSRA	Brownfields Site Rehabilitation Agreement
BTEX	Benzene, Toluene, Ethylbenzene, and Total Xylenes
C	Celsius
CD	Compact Disc
COC	Contaminants of Concern
CTL	Cleanup Target Levels
DAO	(EPA) Designated Approving Official
DEFT	Decision Error Feasibility Trials
DO	Dissolved Oxygen
DPT	Direct Push Technology
DQO	Data Quality Objective
DRO	Diesel Range Organics
e.g.	exempli gratia - for example
ESA	Environmental Site Assessment
ECD	Electron Capture Device
FID	Flame Ionization Detector
GC	Gas Chromatography
GC-MS	Gas Chromatography – Mass Spectrometry
GIS	Geographic Information Systems
GPS	Global Positioning Satellite
GRO	Gasoline Range Organics
HAZWOPER	Hazardous Waste Operations and Emergency Response
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Plasma
ID	Identification
i.e.	*id est* - that is
ISHB	Inactive Hazardous Sites Branch
IUPAC	International Union of Pure and Applied Chemistry
kg	kilogram
L	Liter
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MCL	Maximum Contaminant Level
MDLs	Method Detection Limits
MIP	Membrane Interface Probe

LIST OF ABBREVIATIONS

mL	Milliliter
MNA	Monitored Natural Attenuation
MTBE	Methyl tert-butyl ether
MW	Monitor Well
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NC	North Carolina
NCBP	North Carolina Brownfields Program
NELAC	National Environmental Laboratory Accreditation Conference
NCDEQ	North Carolina Department of Environmental Quality
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OVA	Organic Vapor Analyzer
PAHs	Polynuclear Aromatic Hydrocarbons
PCB	Polychlorinated biphenyl
PE	Performance Evaluation
P.E.	Professional Engineer
P.G.	Professional Geologist
PID	Photo-ionization Detector
PQLs	Practical Quantification Limits
QA	Quality Assurance
QAM	Quality Assurance Manual
QAP	Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAP	Remedial Action Plan
RCRA	Resource Conservation and Recovery Act
REC	Recognized Environmental Condition
RL	Reporting Limit
RPD	Relative Percent Difference
RQAO	Regional Quality Assurance Designated Approving Official
RSC	Regional Screening Levels
SESD	Science and Ecosystem Support Division
SPLP	Synthetic Precipitate Leaching Procedures
SRG	Soil Remediation Goals
SS	Soil Sample
SW	Solid Waste
SVOC	Semi-Volatile Organic Compounds
SOP	Standard Operating Procedure
TAL	Target Analyte List
TCL	Target Compound List

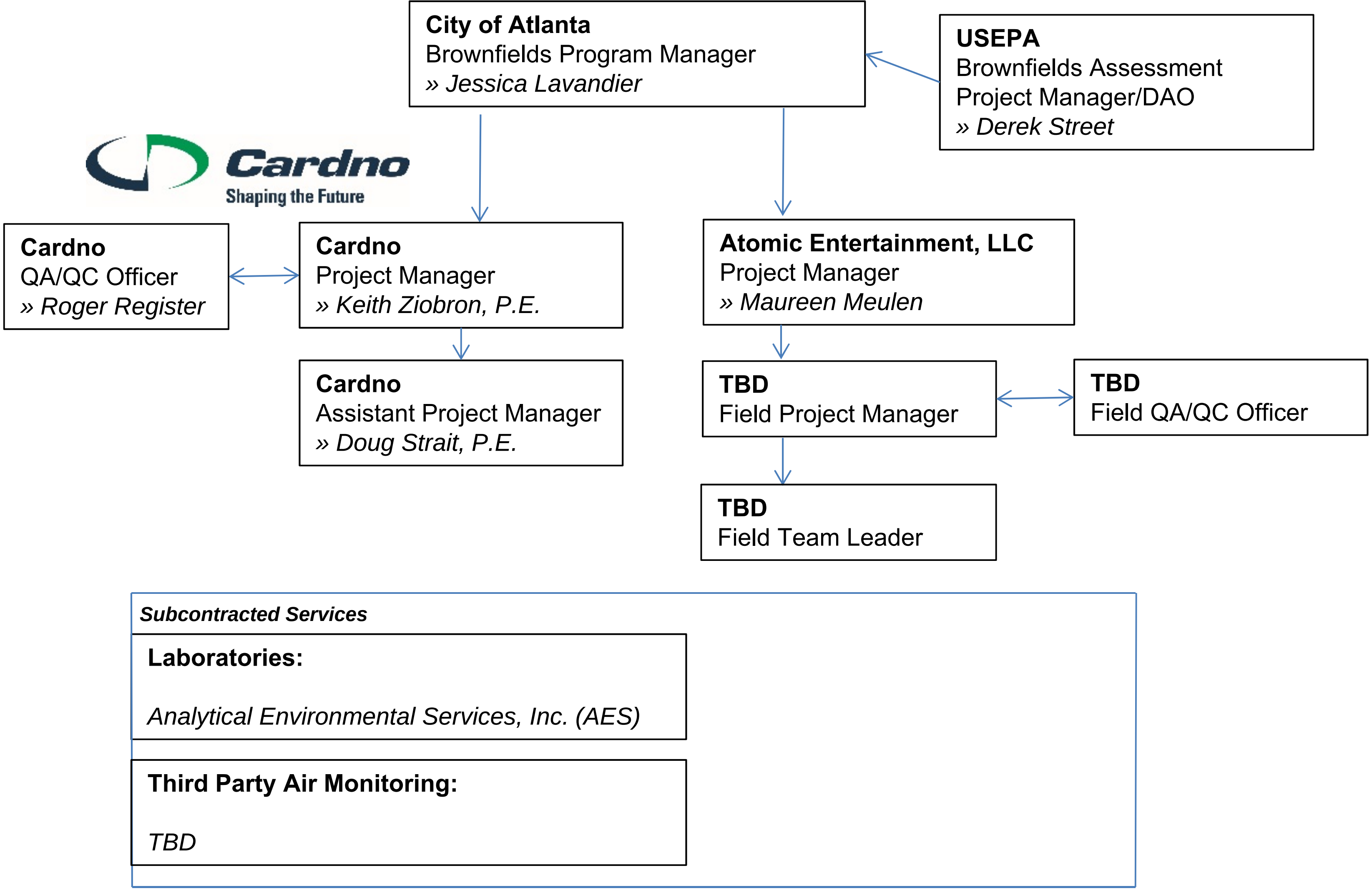
LIST OF ABBREVIATIONS

TCLP	Toxicity Characteristic Leaching Procedure
TPH	Total Petroleum Hydrocarbons
TQM	Total Quality Management
USC	United Soil Classification
U.S. EPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UST	Underground Storage Tank
µg	microgram
ug	microgram
VOC	Volatile Organic Compounds

Appendix A

Project Organization Chart

Quality Assurance Project Organizational Chart



City of Atlanta
Brownfields Program Manager
» Jessica Lavandier

USEPA
Brownfields Assessment
Project Manager/DAO
» Derek Street

Cardno
QA/QC Officer
» Roger Register

Cardno
Project Manager
» Keith Ziobron, P.E.

Cardno
Assistant Project Manager
» Doug Strait, P.E.

Atomic Entertainment, LLC
Project Manager
» Maureen Meulen

TBD
Field Project Manager

TBD
Field QA/QC Officer

TBD
Field Team Leader

Subcontracted Services

Laboratories:

Analytical Environmental Services, Inc. (AES)

Third Party Air Monitoring:

TBD

Appendix B

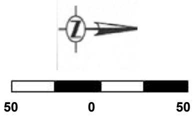
Site Location Map

LEGEND

🟥 Approximate Site Boundary (For reference purposes only, not a surveyed boundary)

Cardno Shaping the Future

"This is not a map of survey."



Not to Scale

QAPP
Pullman Yard
Fulton County, Atlanta, GA
Cardno Project: 0002420000

Appendix B
Site Location Map

Source: GoogleEarth

Appendix C

United's Soil Contamination Map
October 2018

UNITED
CONSULTING

May 1, 2018

Maureen Meulen
Atomic Entertainment Development, LLC
120 Rogers Street NE
Atlanta, GA 30317

RE: Amendment 2 – Hazardous Material Survey – Location and Estimated Quantities Data Package for Asbestos Containing Materials and Lead Base Paint
Pullman Yard Property
225 Rogers Street, Atlanta, DeKalb County, Georgia
Project No. ATENT-17-GA-01288-01

United Consulting has completed the limited Asbestos Containing Materials (ACM) and Lead Base Paint (LBP) survey at the Pullman Yard property (Project Site). The purpose of this survey was to locate and/or identify common building materials for the presence of asbestos fibers or lead base paint, that may be present at the Project Site.

Bulk sampling was performed in substantial conformance with the United States Environmental Protection Agency's (EPA's), "Guidance for Controlling Asbestos-Containing Materials in Buildings" (EPA 560/5-85-024, 1985). Lead Base Paint testing was performed using an XRF, a real-time instrument, and sampling was performed in accordance to EPA/HUD sampling guidelines. No LBP samples were sent to the laboratory. Sample locations were chosen in the field, based on the identification of visible suspect materials.

The enclosed data package includes an overview diagram (provided by Lord Aeck, Sargent, project architect), and individual building data and photographs showing the locations of the impacted materials. Laboratory identified ACM are shown in yellow, and LBP identified materials are shown in blue.

A site plan overview is provided, a table listing the individual building numbers, the roofing square footage, approximate building areas, and summary of ACM and LBP impact areas.

A separate page is provided identifying the identified locations of materials which need to be abated or managed under an O&M plan. For example, there is a page showing the identified locations of ACM inside a building (if applicable), there is a separate page showing the identified locations of ACM outside of a building (if applicable), there is a page showing identified locations of a LBP inside (if applicable), and there is a page showing the identified locations of LBP outside the building (if applicable).

The National Emissions Standard for Hazardous Air Pollutants (NESHAP) requires the removal of ACM prior to activities which would disturb them. United Consulting recommends that the ACM be removed by a qualified asbestos abatement contractor, prior to renovations or demolition, using State of Georgia accredited personnel, in accordance with applicable federal, state and local regulations governing the removal of ACM.

A Ten-Day Notification should be forwarded to the Georgia Department of Natural Resources (DNR), Georgia Environmental Protection Division (EPD), by the building owner or demolition contractor prior to the start of any building demolition activities.

Representative areas of the Project Site were sampled on a limited basis where a visual identification of suspect materials could be made. United Consulting shall not be held responsible for errors, miscalculations, assumptions, misinterpretations or other problems or liabilities arising from, or associated with, firms or individuals bidding on asbestos abatement work that rely solely, or in part, on this document.

This report has been prepared on behalf of the client, Atomic Entertainment Development. Should any other person, partnership, or corporation desire to rely upon this report, it will be necessary for United Consulting to update the report for the new user.

Sincerely,

UNITED CONSULTING

Leonard J. Diprima, Jr., P.G,
Associate Environmental Specialist

Luke von Oldenburg, CHMM, CIH, CSP
Industrial Hygienist

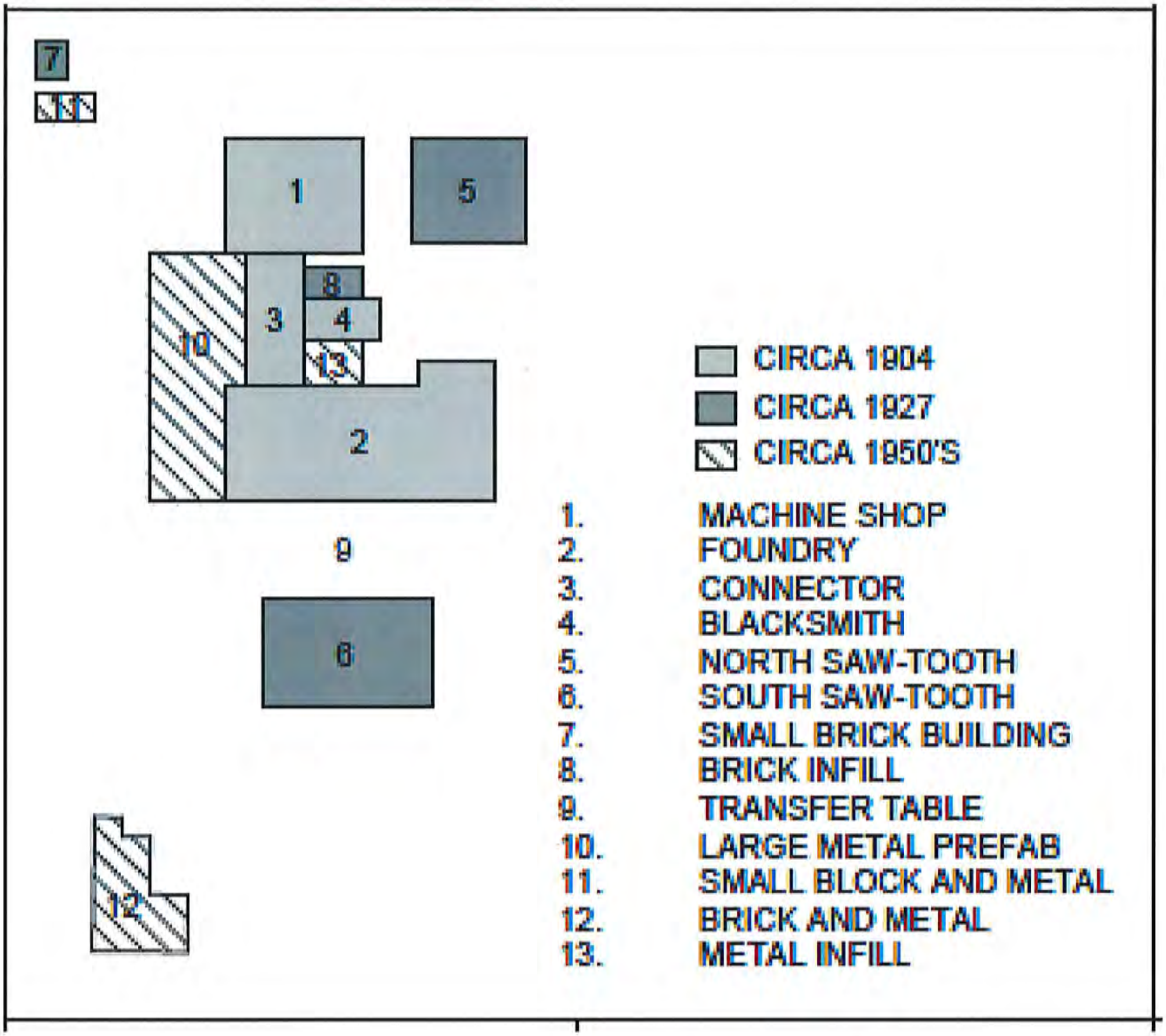
LVO/LJD/slv

cc: Andrea L. Rimer, Troutman Sanders LLP
Ben Norton, Brasfield & Gorrie

SharePoint: ATENT-17-GA-01288-01

APPENDICES

Attachment A Laboratory Results



▭ CIRCA 1904
■ CIRCA 1927
▨ CIRCA 1950'S

1.	MACHINE SHOP
2.	FOUNDRY
3.	CONNECTOR
4.	BLACKSMITH
5.	NORTH SAW-TOOTH
6.	SOUTH SAW-TOOTH
7.	SMALL BRICK BUILDING
8.	BRICK INFILL
9.	TRANSFER TABLE
10.	LARGE METAL PREFAB
11.	SMALL BLOCK AND METAL
12.	BRICK AND METAL
13.	METAL INFILL

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

ACM – Lead Base Paint Summary

Building Number	Roof* Area sq. ft.	Building foot print sq. ft.	Lead Inside	Lead Outside	Asbestos Inside	Asbestos Outside	Comment
1	16,915	~18,000	Yes	No	No	Yes	Lead – steel and hand rail Asbestos Roof Flashing
2	37,387	~34,250	Yes	No	No	Yes	Lead steel Asbestos Roof
3	9,210	~8400	Yes	No	Yes	Yes	Lead – yellow hand rails Asbestos roof and flashing Asbestos mastic, tile, under paper
4 - west	3,787	~1600	Yes	No	No	Yes	Lead – yellow walls Asbestos roof
4 - east		~1600	No	No	No	Yes	Asbestos Roof
5	12,045	~13,800	Yes	No	Yes	Yes	Lead – steel Asbestos caulking Asbestos Roof
6	17,412	~20,350	Yes	No	Yes	Yes	Lead – steel Asbestos caulking Asbestos Roof
7	1,162	~1200	Yes	No	-	No	Lead Steel rafters
8	2,128	~2400	Yes	No	No	Yes	Lead – white interior wall paint Asbestos roof
10	20,315	~23,500	Yes	Yes	Yes	No	Lead – Yellow painted pipe and steel Asbestos – pipe wrap
11	-	~	No	No	No	Yes	Asbestos – Silver Mastic
12	10,264	~10,200	Yes	No	No	Yes	Lead – Orange steel Asbestos Sealant along building
13	~3,040	~3,040	No	No	Yes	Yes	Asbestos Pipe Wrap Asbestos Roof

Notes:

*Roof Areas are calculated by Lord Aeck Sargent – The information is attached to this package

Aside from "Roof Areas," building areas, perimeters, lengths and volumes are approximations based on site visits, measuring wheel, and provided PDF drawings.

The above measurements are related to the buildings and not the amount, size, or volume of asbestos or lead base paint.

Final responsibility of the estimations of the amount of Asbestos and Lead Base Paint to be abated will rely solely on the contractor who will be performing the task of abatement and removal.

As with all environmental investigations, there may be hidden pipe chases, vaults, etc... and other areas which may not have been visible during the surficial site walk.

The exterior pipe chase, located on the western edge of Building 10, is approximately 390 feet long. We do not know the start or the end of the pipe chase. Currently there appears to be two sets of pipes partially wrapped in asbestos insulation.

Roof Schedule

Type	Area	Comments
Generic - 6"	4,337 SF	BUILDING 1
Generic - 6"	8,420 SF	BUILDING 1
Generic - 6"	4,158 SF	BUILDING 1
Clearstory - Windows	1,854 SF	BUILDING 1
Clearstory - Windows	1,854 SF	BUILDING 1
Generic - 6"	26,006 SF	BUILDING 2
Generic - 6"	2,139 SF	BUILDING 2
Generic - 6"	2,093 SF	BUILDING 2
Generic - 6"	4,033 SF	BUILDING 2
Generic - 6"	3,116 SF	BUILDING 2
Clearstory - Windows	1,553 SF	BUILDING 2
Clearstory - Windows	1,115 SF	BUILDING 2
Generic - 6"	9,210 SF	BUILDING 3
Generic - 6"	1,961 SF	BUILDING 4
Generic - 6"	1,826 SF	BUILDING 4
SawTooth Solid Roofs	2,409 SF	BUILDING 5
SawTooth Solid Roofs	2,409 SF	BUILDING 5
SawTooth Solid Roofs	2,409 SF	BUILDING 5
SawTooth Solid Roofs	2,409 SF	BUILDING 5
SawTooth Solid Roofs	2,409 SF	BUILDING 5
Clearstory - Windows	1,157 SF	BUILDING 5
Clearstory - Windows	1,111 SF	BUILDING 5
Clearstory - Windows	1,111 SF	BUILDING 5
Clearstory - Windows	1,111 SF	BUILDING 5
Clearstory - Windows	1,111 SF	BUILDING 5
SawTooth Solid Roofs	4,353 SF	BUILDING 6
SawTooth Solid Roofs	4,353 SF	BUILDING 6
SawTooth Solid Roofs	4,353 SF	BUILDING 6
SawTooth Solid Roofs	4,353 SF	BUILDING 6
Clearstory - Windows	2,065 SF	BUILDING 6
Clearstory - Windows	2,065 SF	BUILDING 6
Clearstory - Windows	2,065 SF	BUILDING 6
Clearstory - Windows	2,065 SF	BUILDING 6
Flat Roof	1,162 SF	BUILDING 7
Generic - 6"	2,128 SF	BUILDING 8
Metal-Low Slope	343 SF	BUILDING 10
Metal-Low Slope	19,972 SF	BUILDING 10
Roof in Question	4,971 SF	BUILDING 10
Metal-Low Slope	6,192 SF	BUILDING 12
Flat Roof	4,072 SF	BUILDING 12
Concrete - BLDG 12	41 SF	BUILDING 12
Grand total: 41	155,878 SF	

Window Schedule

Steel
- 67 Openings
- 9,338 S.F.

Wood
- 192 Openings
- 22,875 S.F.
- 6 Louvers

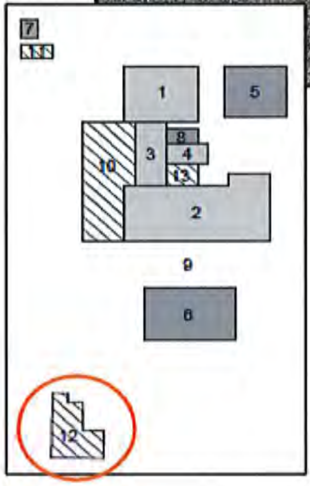
Aluminum
- 3 Openings
- 360 S.F.

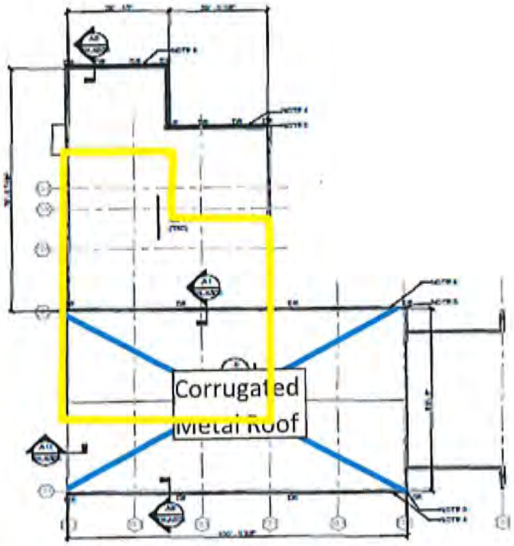
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 12

Asbestos

Roof Sealantg





Building 12 – Outside Roof

Asbestos roof sealant - asbestos

Lower asphalt roof – The Sealant between the two buildings and sealant along the edge is positive for Asbestos.

Estimated: Lower asphalt roof perimeter ~ 280 linear feet

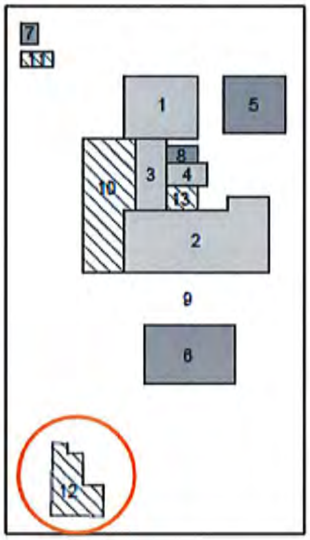
The overall tar/gravel roof is Non-detect for asbestos.

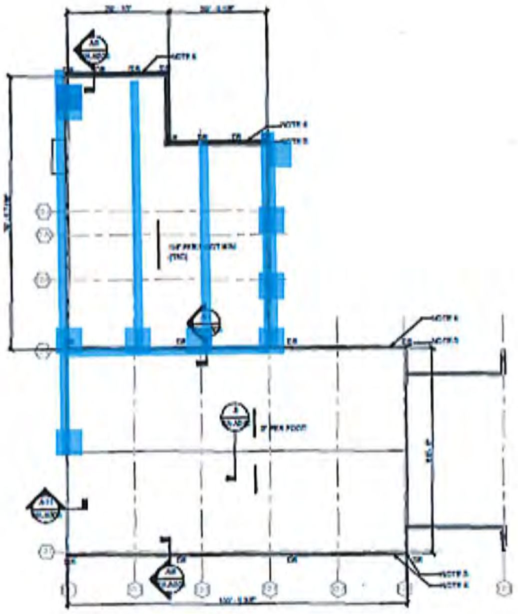
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 12

Lead Base Paint

Steel beams (lower sections)





Building 12 Inside – Lower section

LBP – vertical and horizontal, steel supports, orange I-Beams (mostly in the lower roof section).

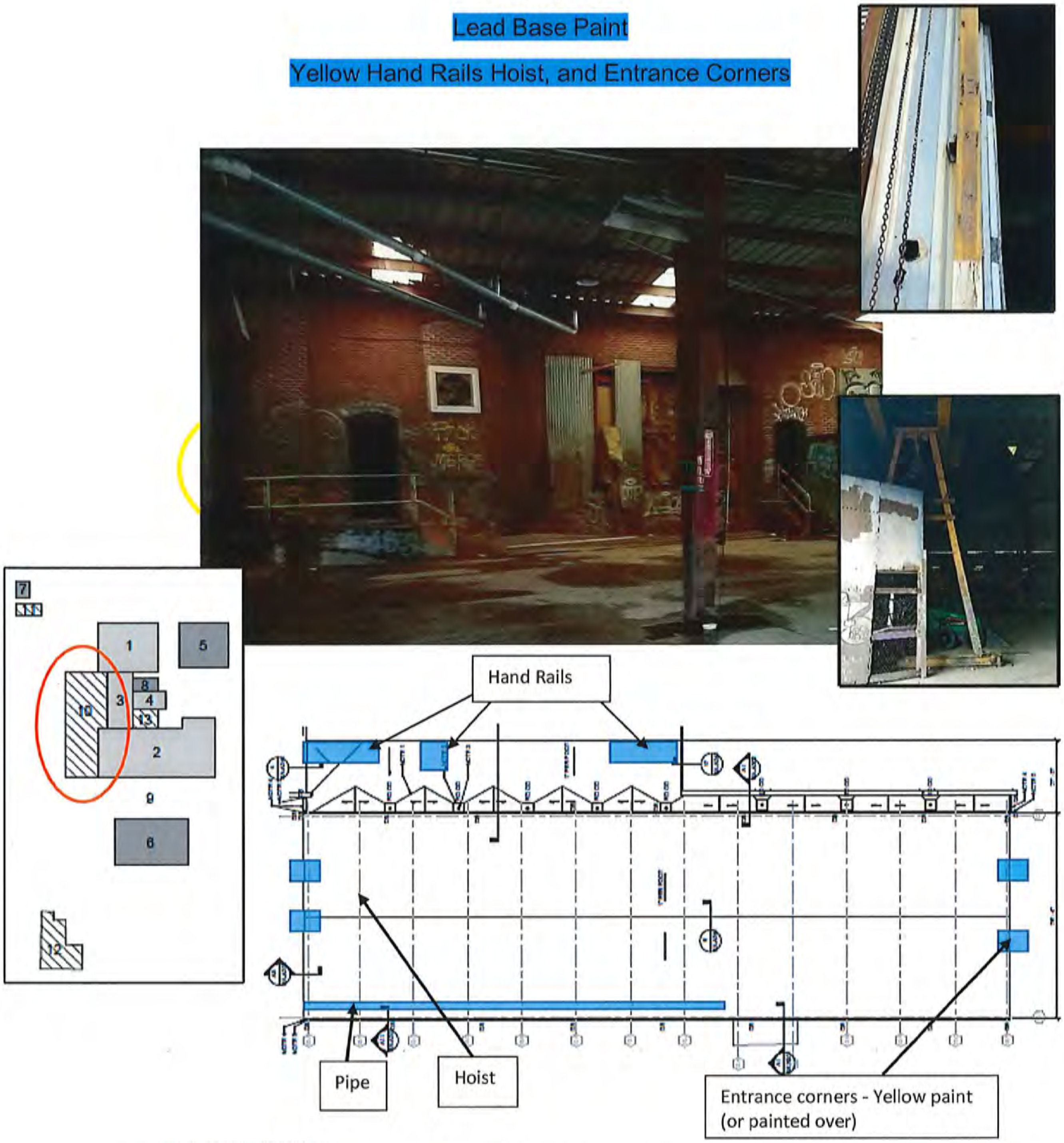
Estimated: ~600 linear feet of steel.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 10

Lead Base Paint

Yellow Hand Rails Hoist, and Entrance Corners



Building 10: Inside -

LBP – Hand Rails - Painted Yellow – (Three sets of rails, Estimated: ~125 linear feet). Hoist, Water pipe (yellow paint) along west wall (Estimated: ~ 120 linear feet) and the Posts/corners at entrances on north and south ends (note yellow paint).

Note, the metal roof and walls are Non-LBP.

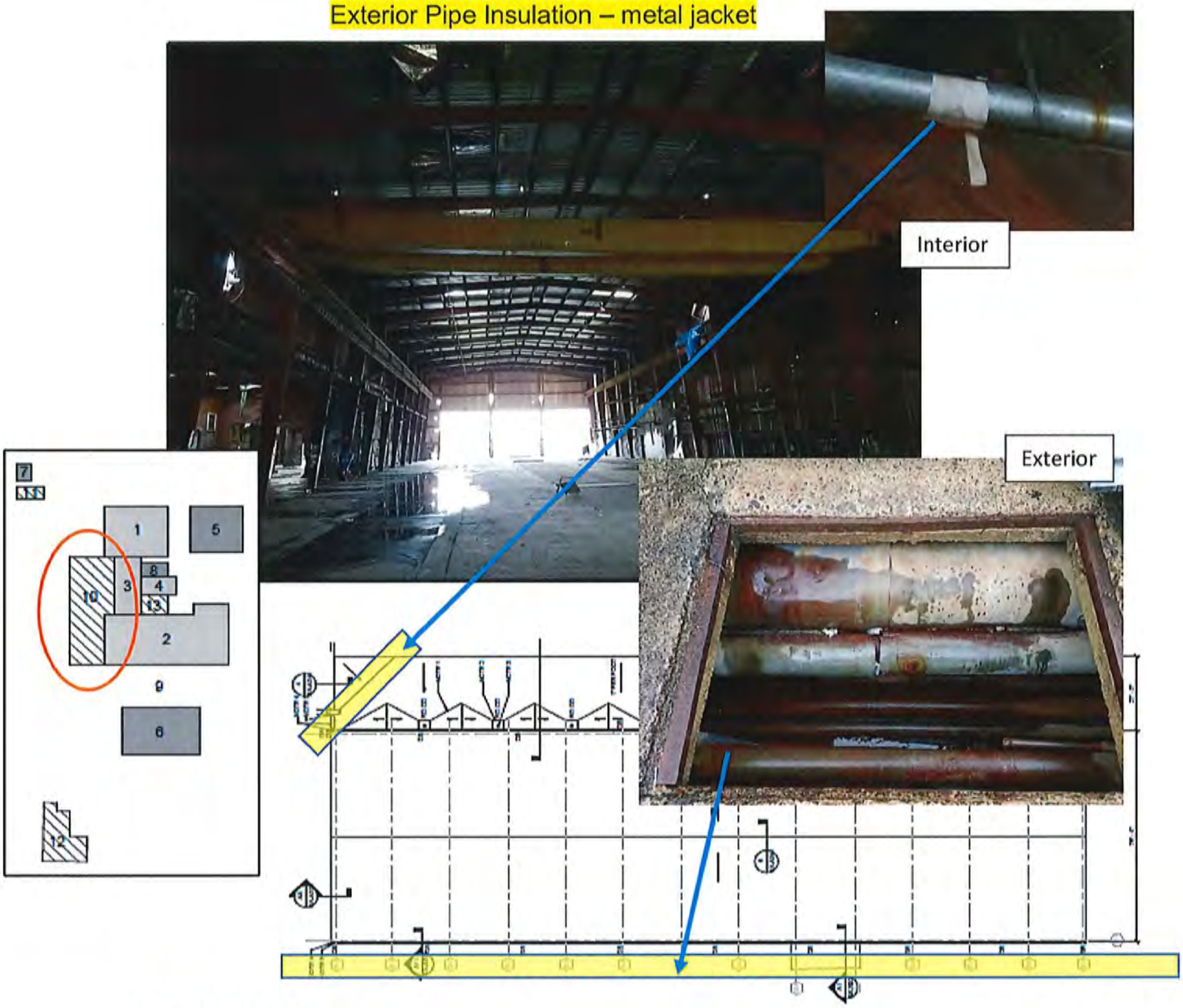
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 10

Asbestos

Interior Pipe Wrap

Exterior Pipe Insulation – metal jacket



Building 10 - Inside

Pipe Wrap - two joint spots.

Estimated: < 10 linear feet.

Building 10 - Outside

Pipe Insulation in chase.

Estimated: pipe chase length: ~ 390 linear feet (at least two lines with partial asbestos wrap).

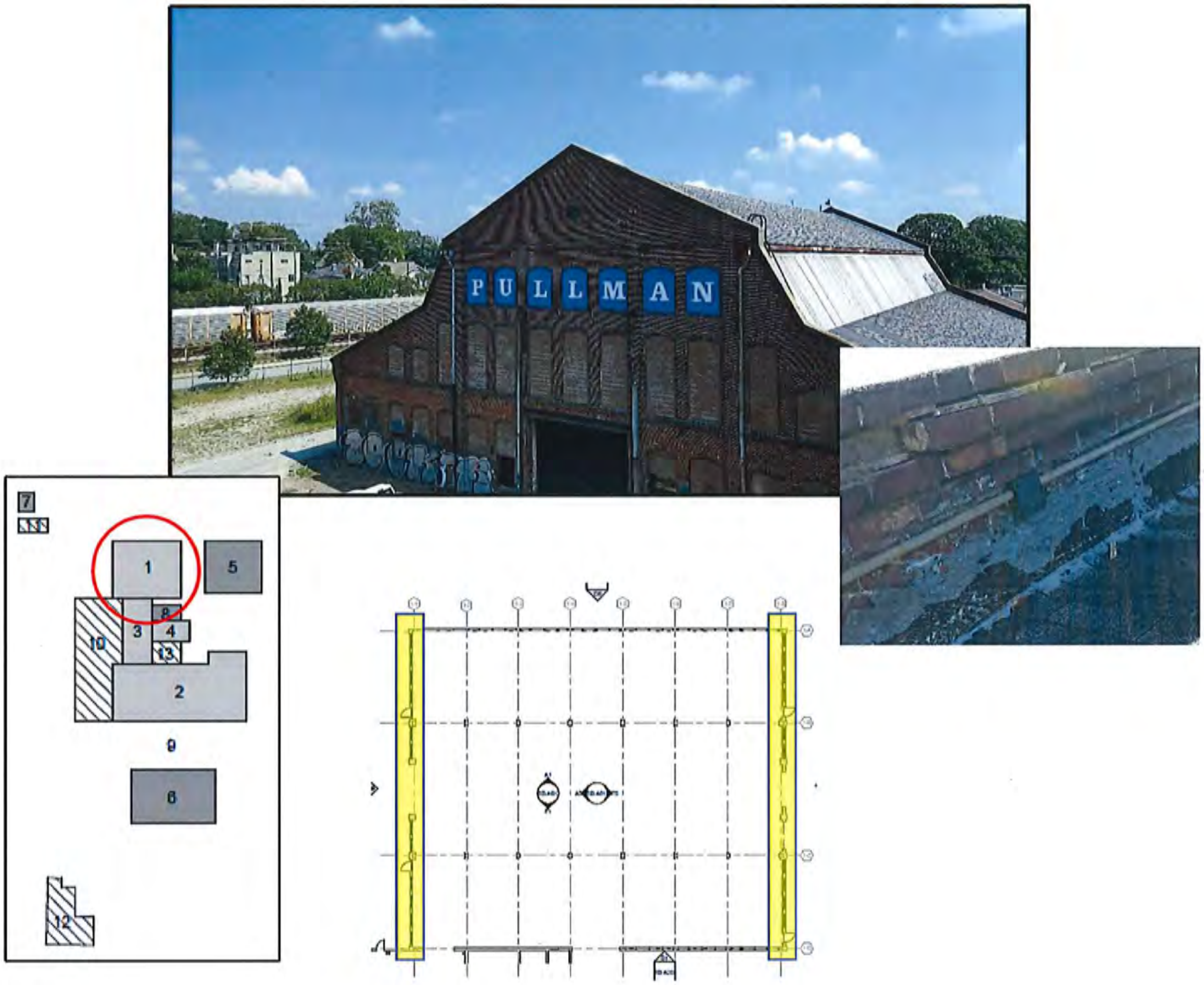
WE DO NOT KNOW WHERE THE PIPES IN THE PIPE CHASE START AND STOP.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 1

Asbestos

Roof Sealant (Flashing along edge and sealant)



Building 1 – Outside

Asbestos roof sealant along flashing paper.

Estimated: ~ 370 linear feet.

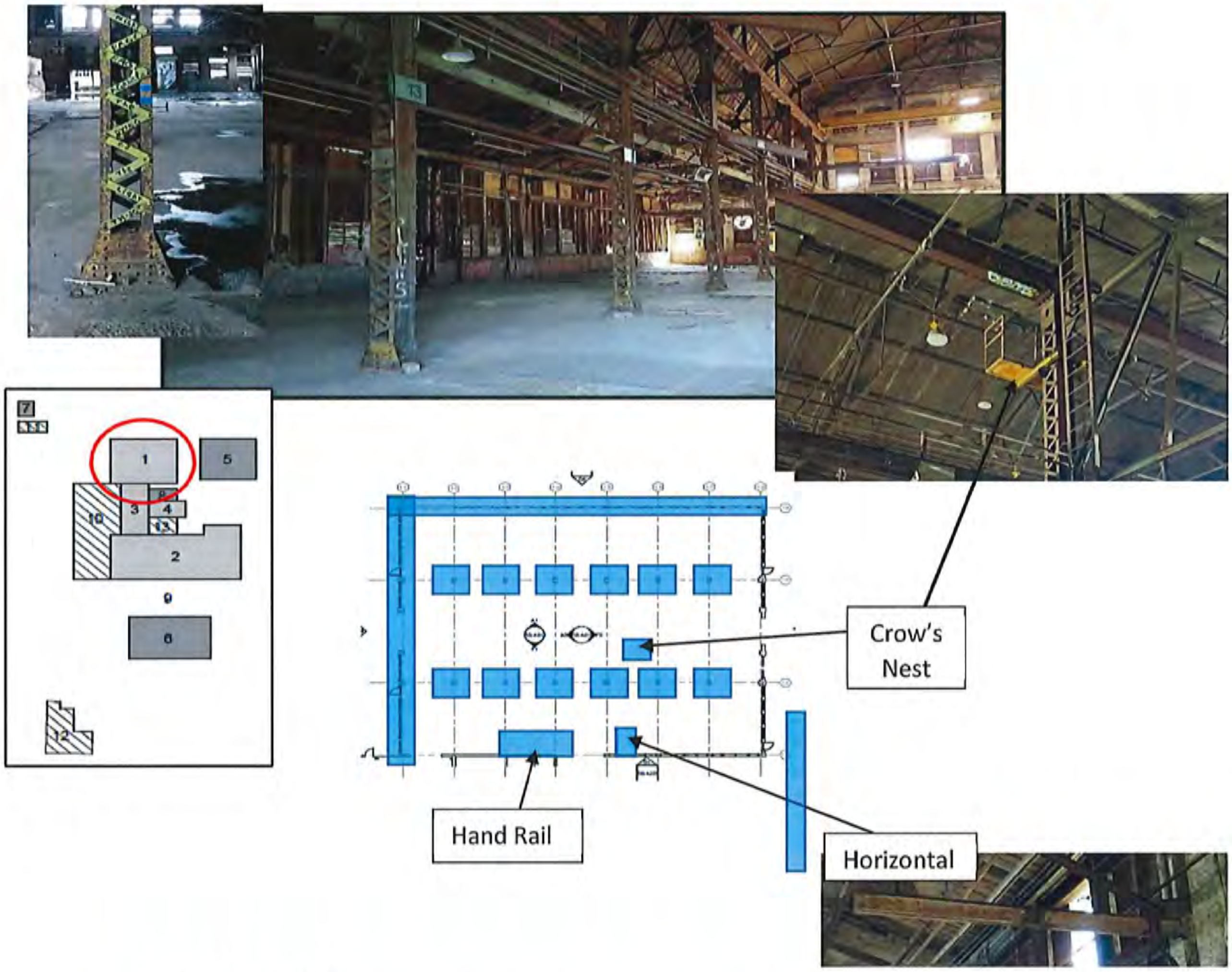
Asphalt Shingles on roof paper (non- asbestos) directly on wood.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 1

Lead Base Paint

Steel columns, crow's nest, hoist arms, lower wall, window jambs, yellow rail



Building 1 – Inside

(12 - vertical, painted, lattice post – the lower 10 feet only).

Estimated: ~120 linear feet

Upper Crow's nest (yellow) and horizontal I beam (yellow) - linear feet undetermined.

Hand painted rail (yellow).

Estimated: ~23 linear feet of

Wall surface – lower 4 feet of west, north and northeast walls.

Estimated: ~2200 sq. ft.

LBP - on the painted, wooden window jambs and frames.

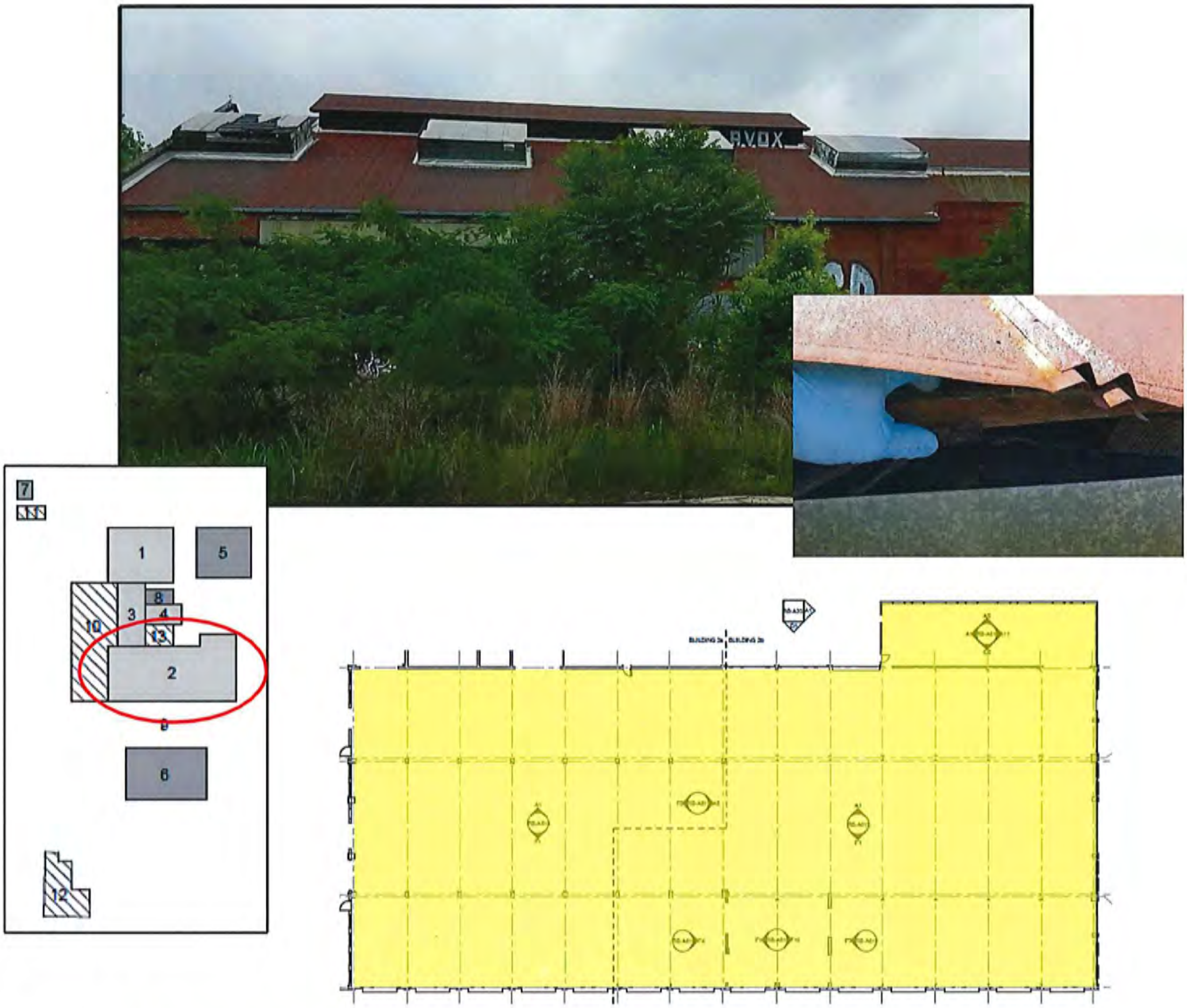
Estimated Number of Windows: 107

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 2

Asbestos

Roofing Felt/Paper



Building 2 - Outside

Asbestos roof flashing, sealant, and tar paper.

Estimated: 39,200 sq. ft.

Metal Roof on top attached to wood strappings. The corrugated roof lays on top of Asphalt shingles and asbestos felt/roofing paper.

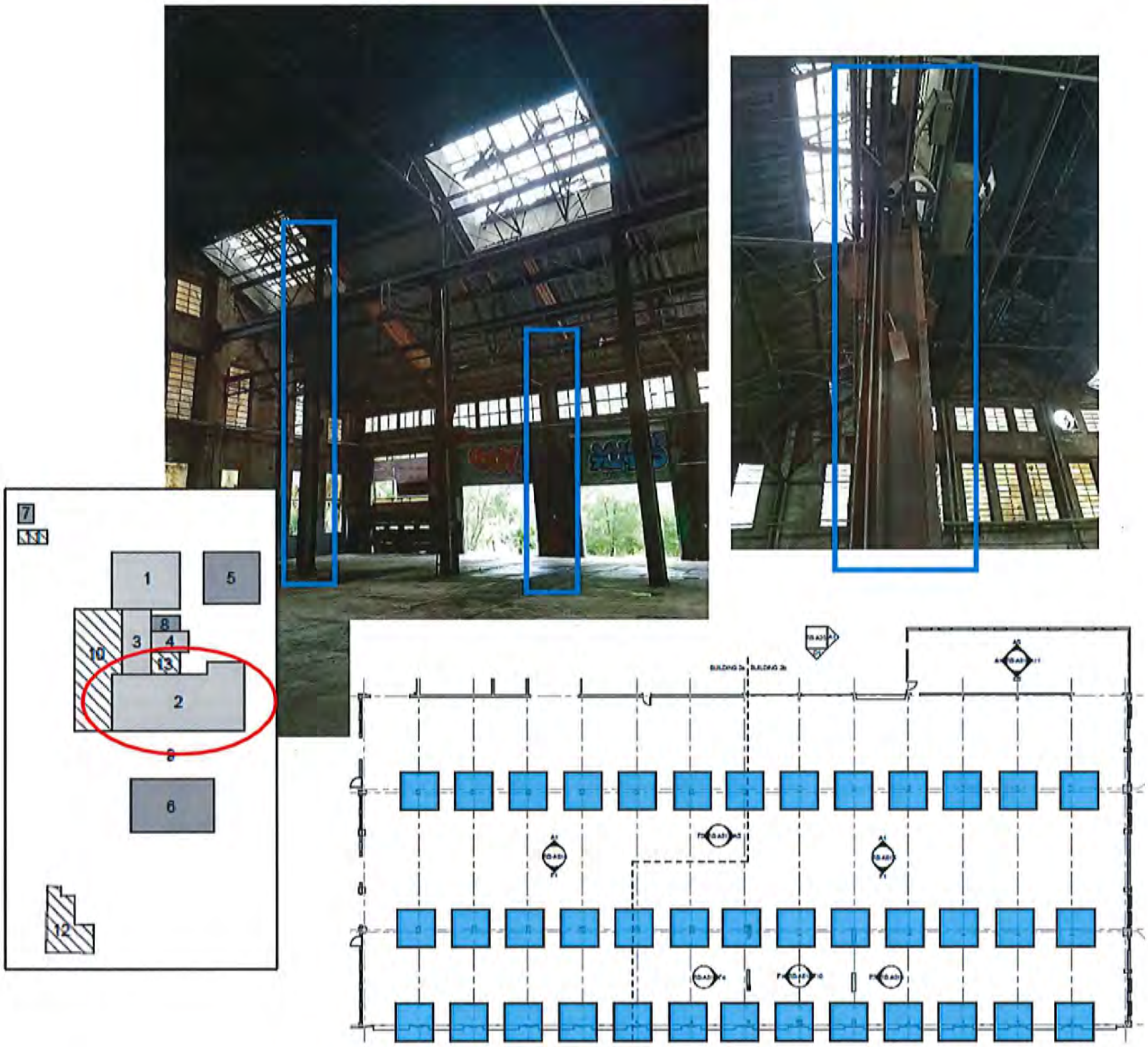
Gutter Sealant, (non- asbestos).

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 2

Lead Base Paint

Interior metal vertical beams



Building 2 - Inside

39 Vertical lattice beams – full length.

Estimated: each post ~25 feet each steel post; total ~975 linear feet

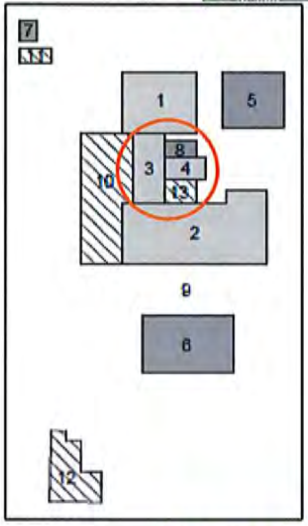
Painted walls were negative for LBP.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Buildings 3, 4, 8, 13

Asbestos

Roofing System



Building 13 is a flat roof – the Roofing system is Asbestos.

Estimated: Building 13 Area ~ 3040 sq. ft.

Building 4, and 8 are pitched roofs with asbestos felt paper and asbestos shingles.

Estimated: Building 4 Area ~ 3200 sq. ft.

Estimated: Building 8 Area ~2400 sq. ft.

Building 3 is pitched – metal roof with asphalt / tar paper roof system below a metal roof– asbestos.

Estimated: Building 3 Area ~8,400 sq. ft.

In addition, Building 3 has asbestos in the gray flashing sealant.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Buildings 3, 4, 8, 13

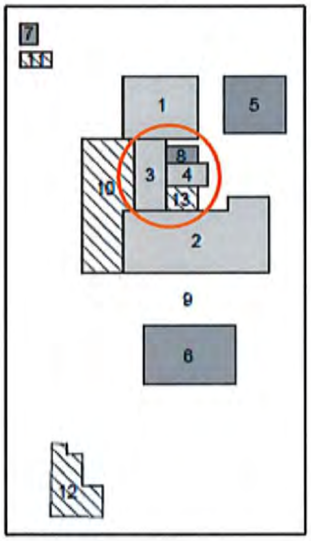
Asbestos

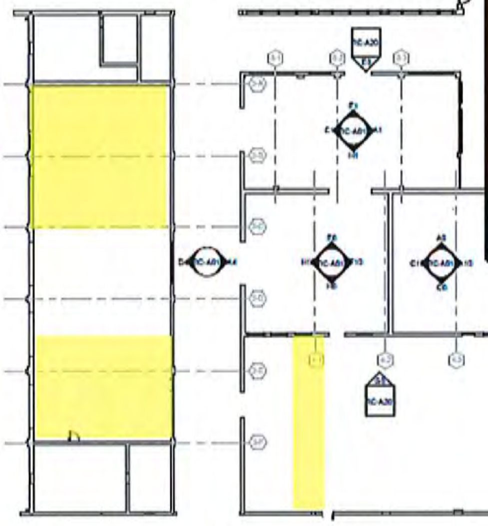
Floor tile, Pipe Insulation

13

3

3





Building 3 – Inside

Office area has asbestos floor tiles, mastic, and asbestos paper under the floor tile.

Estimated: ~ 2500 sq. ft.

Building 13 - Inside

Area: length of asbestos pipe wrap overhead.

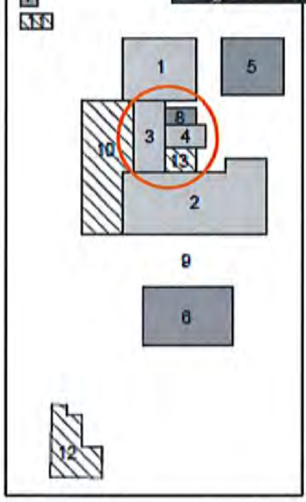
Estimated: ~ 27 linear feet.

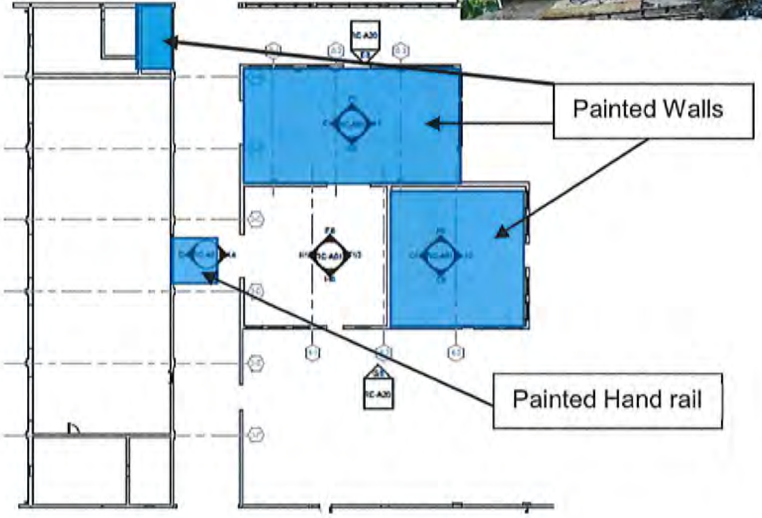
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Buildings 3, 4, 8, 13

Lead Painted

White Wall Paint Yellow Hand Rail, Green Wall Paint





Building 8 – white paint on all walls is LBP. Surface area undetermined.

Building 4 – Yellow Paint on walls is LBP. Surface area undetermined.

Building 3 - Painted hand rail.

Estimated: ~ 23 linear feet of rails plus base.

Building 3 - Green painted closet (access from Building 1).

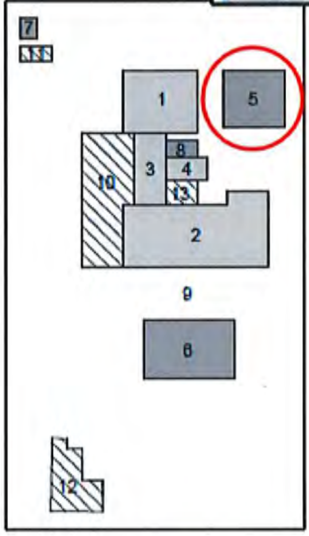
Estimated: lower 10 feet (room 8 x 20), ~1,600 sq. ft.

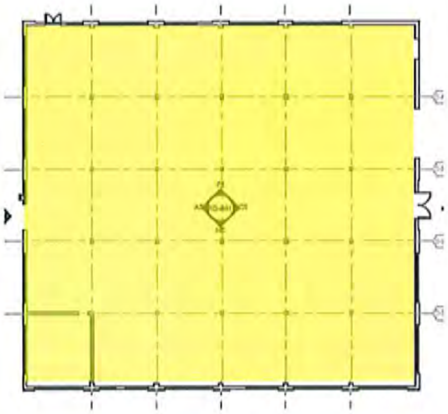
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 5

Asbestos

Roofing Felt/Paper and Mastic





Building 5 – Outside

Asbestos roof flashing sealant and Roofing paper

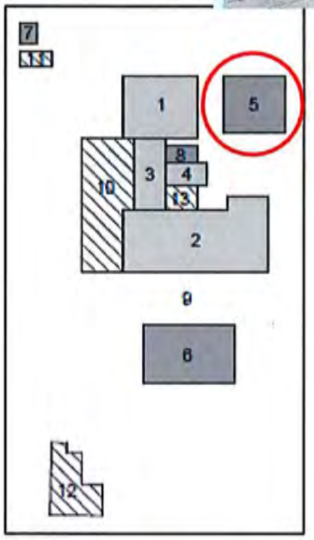
Estimated: ~12,000 sq. ft.

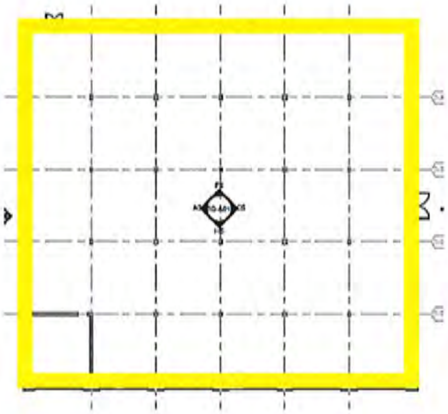
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 5

Asbestos

Window Caulk





Building 5 - inside

Asbestos window caulk – amount varies in each window.

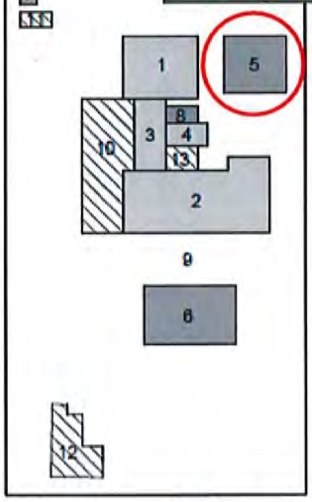
Estimated: ~900 windows panes

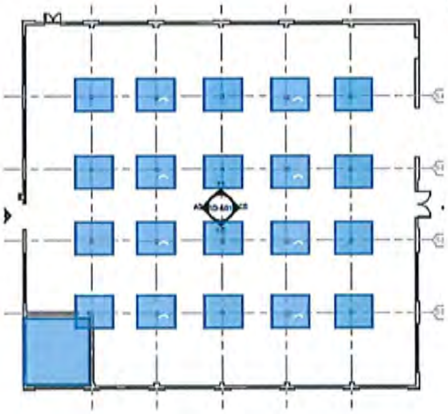
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Building 5

Lead Base Paint

Steel columns, cross beams and office





Building 5 - Inside

Vertical Steel support columns (Red and yellow paint – 14 feet tall) – LBP.

Estimated: ~280 linear feet

White, steel cross beams/trusses – LBP.

Estimated: undetermined

Corner office – white paint is LBP.

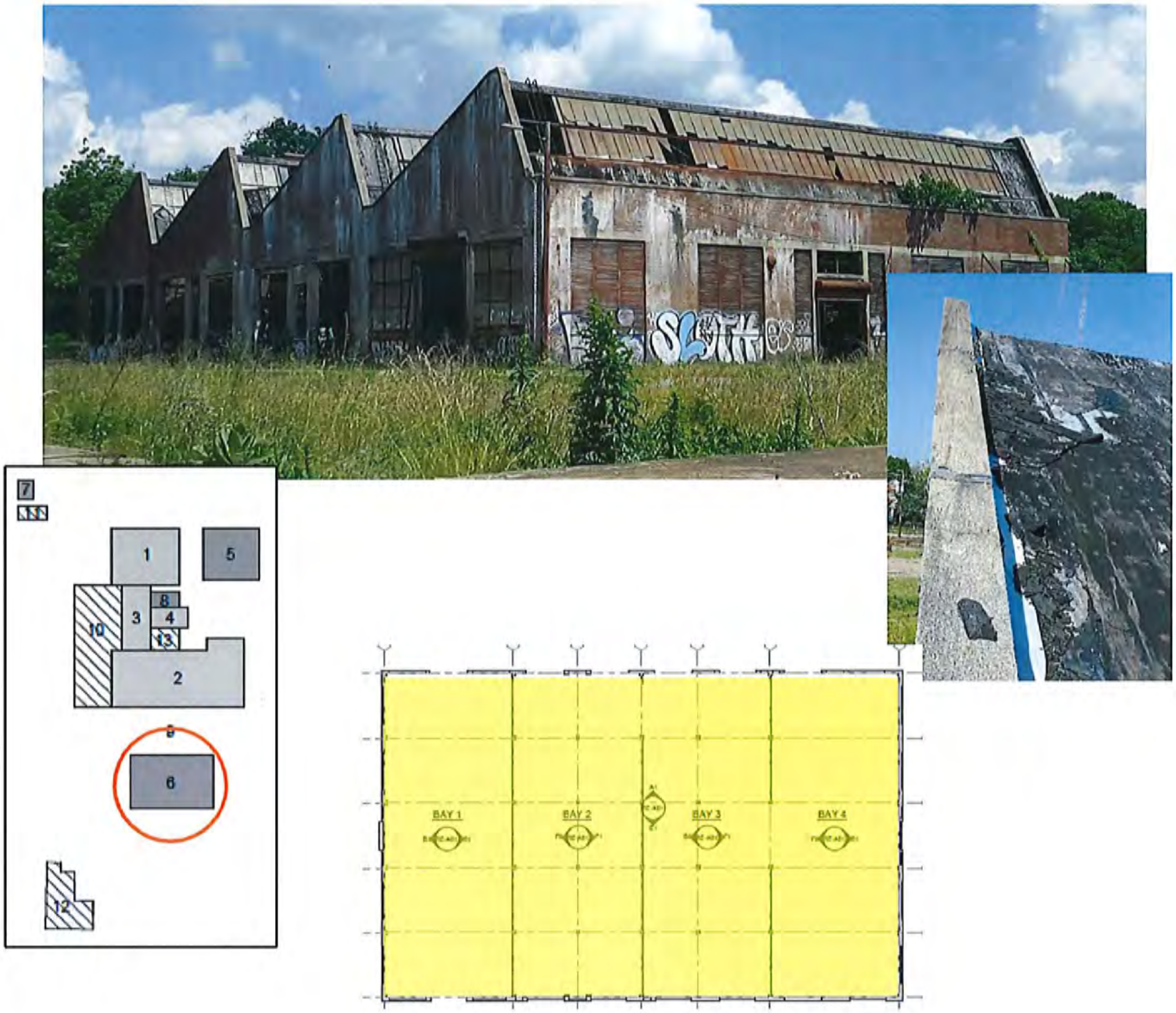
Estimated: ~20 x 22 x 14 feet, ~6,160 sq. ft.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Buildings 6

Asbestos

Roofing Paper and Sealant



Building 6 - Outside

Roofing system is Asbestos – tar paper and black sealant.

Estimated: ~ 17,500 sq. ft.

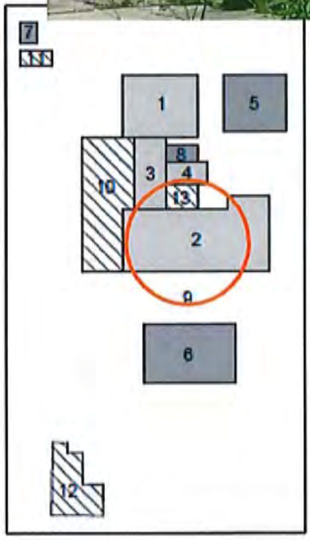
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

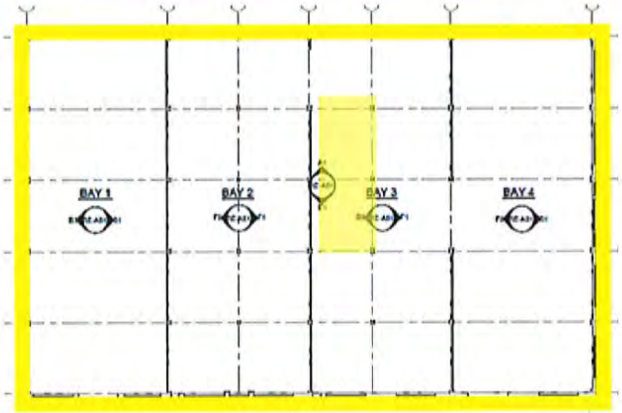
Buildings 6

Asbestos

Window Caulk and Furnace Insulation/Gaskets

Asbestos gasket





Building 6 - Inside

Asbestos window caulk - various amounts in each window.

Asbestos in gaskets and insulation of furnace.

Building Perimeter: ~590

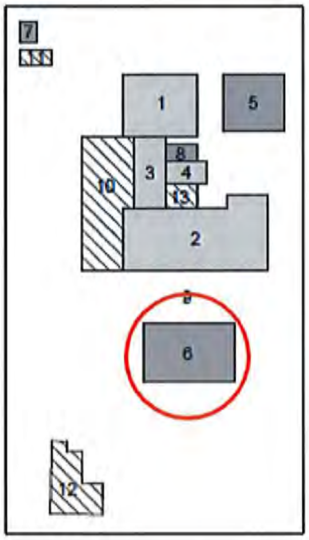
Estimated: Number of window panes 1450

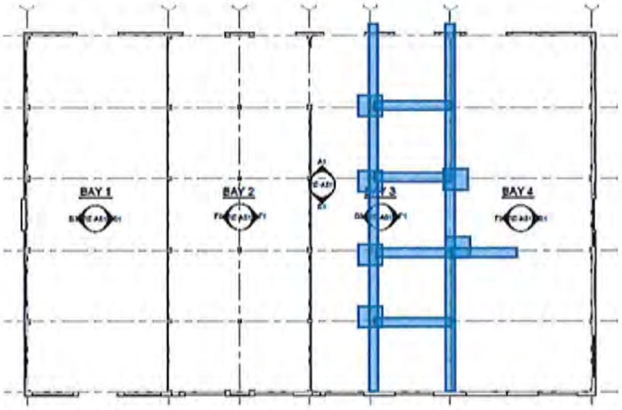
NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

Buildings 6

Lead base paint

Inside – Yellow Steel





Building 6 - Inside

Yellow steel structure LBP.

Estimated: ~450 linear feet

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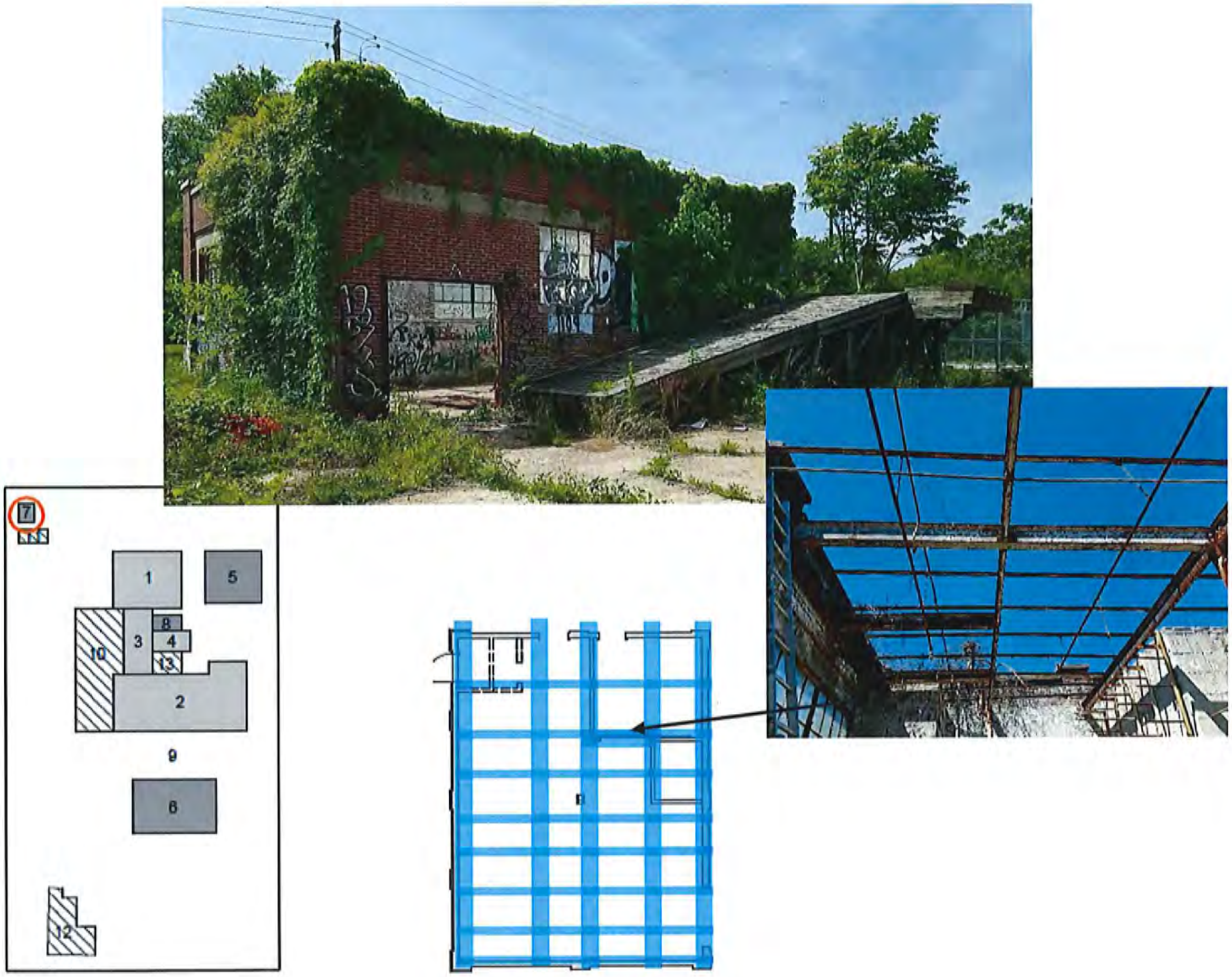
Buildings 7

Lead Base Paint

Cross Beams – No Roof



Building 7 - inside

5 I-beams (40 feet each), 14 smaller cross beams (31 feet each), and 7 minor beams (5-feet each) with white LBP across the open top.

Estimated: ~669 linear feet

Walls are non-LBP.

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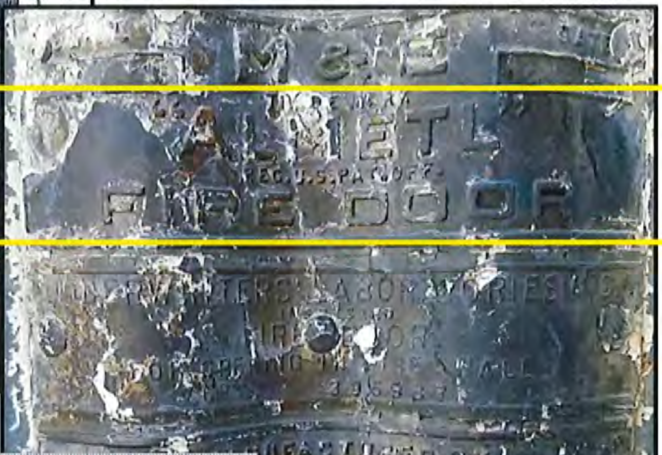
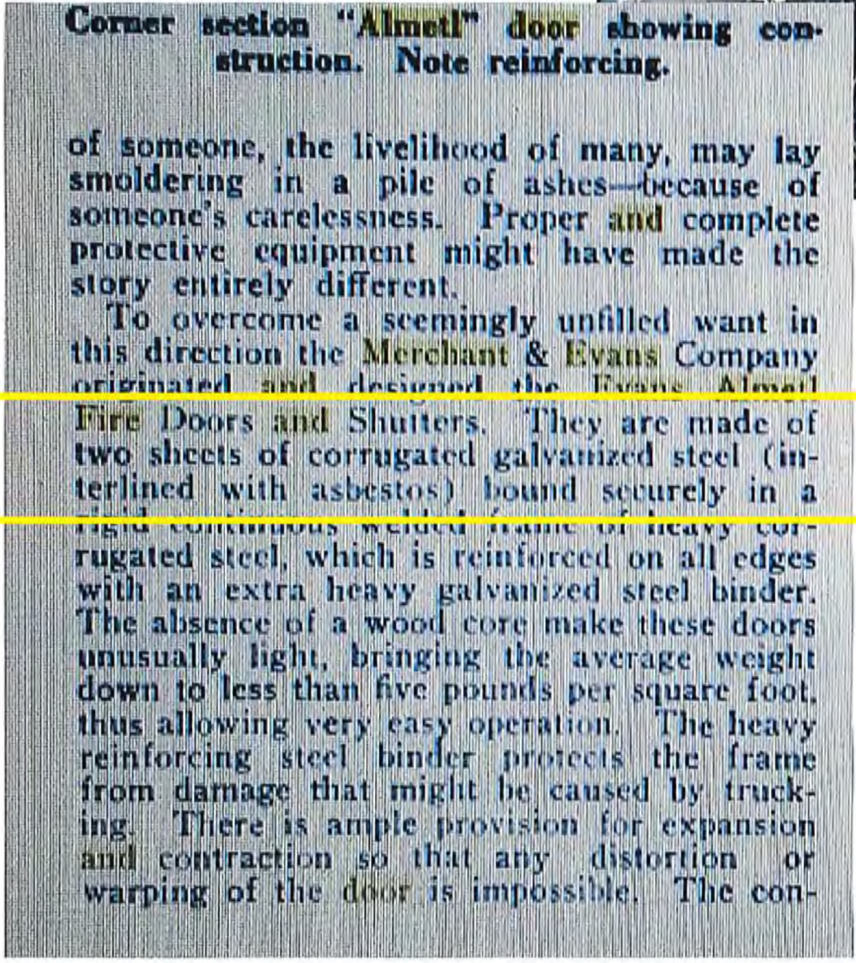
Building 7

Asbestos Fire Door





Corner section "Almetl" door showing construction. Note reinforcing.

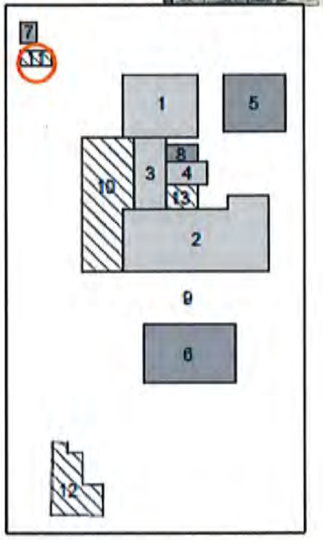
of someone, the livelihood of many, may lay smoldering in a pile of ashes—because of someone's carelessness. Proper and complete protective equipment might have made the story entirely different.

To overcome a seemingly unfilled want in this direction the Merchant & Evans Company originated and designed the Evans Almetl Fire Doors and Shutters. They are made of two sheets of corrugated galvanized steel (interlined with asbestos) bound securely in a rigid continuous welded frame of heavy corrugated steel, which is reinforced on all edges with an extra heavy galvanized steel binder. The absence of a wood core make these doors unusually light, bringing the average weight down to less than five pounds per square foot, thus allowing very easy operation. The heavy reinforcing steel binder protects the frame from damage that might be caused by trucking. There is ample provision for expansion and contraction so that any distortion or warping of the door is impossible. The con-

Buildings 11

Asbestos

Mastic



Building 11 - Outside

ASBESTOS mastic sealant, gray, where the roof meets the building.

Estimated: ~60 linear feet (30 feet each side)

MASTIC IS ON BOTH SIDES, EAST AND WEST.

NOTE ALL ESTIMATES ARE APPROXIMATE. ABATEMENT CONTRACTOR(S) SHOULD VERIFY ALL ESTIMATES PROVIDED.

ATTACHMENT A

Analytical Data

Appendix D

EPA Region 4, Management of Investigation Derived Waste, SESDPROC-202-R3 SOP

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Management of Investigation Derived Waste**

Effective Date: July 3, 2014

Number: SESDPROC-202-R3

Authors

Name: Art Masters
Title: Environmental Scientist, Regional Expert
Signature:
Date: 7/2/14

Approvals

Name: John Deatrick
Title: Acting Chief, Enforcement and Investigations Branch
Signature:
Date: 7/2/14

Name: Mike Bowden
Title: Acting Chief, Ecological Assessment Branch
Signature:
Date: 7/2/14

Name: Bobby Lewis
Title: Field Quality Manager, Science and Ecosystem Support Division
Signature:
Date: 7/2/14

Revision History

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History	Effective Date
SESDPROC-202-R3, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R2. **General:** Corrected typographical, grammatical and/or editorial errors. **Cover Page:** The Enforcement and Investigations Branch Chief was changed from Archie Lee to Acting Chief John Deatrick. The Ecological Assessment Branch Chief was changed from Bill Cosgrove to Acting Chief Mike Bowden. The FQM was changed from Liza Montalvo to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history.	July 3, 2014
SESDPROC-202-R2, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R1.	October 15, 2010
SESDPROC-202-R1, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R0.	November 1, 2007
SESDPROC-202-R0, Management of Investigation Derived Waste, Original Issue	February 05, 2007

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TABLES

Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures and considerations to be used and observed when managing investigation derived waste (IDW) generated during the course of hazardous waste site investigations.

1.2 Scope/Application

The procedures and management options for the different categories of IDW described in this document are to be used by SESD field personnel to manage IDW generated during site investigations. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to manage IDW generated at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when managing IDW. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when managing IDW:

- Due to time limitations and restrictions posed by RCRA regulations on storage of hazardous waste, accumulation start dates should be identified on all drums, buckets or other containers used to hold IDW so that it can be managed in a timely manner.
- During generation of both non-hazardous and hazardous IDW, keep hazardous IDW segregated from non-hazardous IDW to minimize the volume of hazardous IDW that must be properly managed.

2 Types of Investigation Derived Waste

Materials which may become IDW include, but are not limited to:

- Personal protective equipment (PPE) - This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.
- Disposable equipment and items - This includes plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers (COLIWASAs), Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.
- Soil cuttings from drilling or hand augering.
- Drilling mud or water used for mud or water rotary drilling.
- Groundwater obtained through well development or well purging.
- Cleaning fluids such as spent solvents and wash water.
- Packing and shipping materials.

Table 1, found at the end of this procedure, lists the types of IDW commonly generated during field investigations and the current disposal practices for these materials.

For the purpose of determining the ultimate disposition of IDW, it is typically distinguished as being either hazardous or non-hazardous. This determination is based on either clear regulatory guidance or by subsequent analysis. This determination and subsequent management is the responsibility of the program site manager.

3 Management of Non-Hazardous IDW

Disposal of non-hazardous IDW should be addressed in the study plan or QAPP for the investigation. To reduce the volume of any IDW transported back to the Field Equipment Center (FEC), it may be necessary to compact the waste into a reusable container, such as a 55-gallon drum.

If the waste is from an active facility, permission should be sought from the operator of the facility to place the non-hazardous PPE, disposable equipment, and/or paper/cardboard into the facility's dumpsters. If necessary, these materials may be placed into municipal dumpsters, with the permission of the owner. These materials may also be taken to a nearby permitted landfill. On larger studies, waste hauling services may be obtained and a dumpster located at the study site.

Disposal of non-hazardous IDW such as drill cuttings, drilling mud, purge or development water, decontamination wash water, etc., should be specified in the approved study plan or QAPP. It is recommended that these materials be placed into a unit with an environmental permit, such as a landfill or sanitary sewer. These materials must not be placed into dumpsters. If the facility at which the study is being conducted is active, permission should be sought to place these types of IDW into the facility's treatment system. It may be feasible to spread drill cuttings around the borehole, or, if the well is temporary, to place the cuttings back into the borehole. Non-hazardous monitoring well purge or development water may also be poured onto the ground down gradient of the monitoring well when site conditions permit. Purge water from private potable wells which are in service may be discharged directly onto the ground surface.

The minimum requirements for this subsection are:

- Non-hazardous liquid and soil/sediment IDW may be placed on the ground or returned to the source if doing so does not endanger human health or the environment or violate federal or state regulations. Under no circumstances, however, should monitoring well purge water be placed back into the well from which it came.
- Soap and water decontamination fluids and rinsates of such cannot be placed in any water bodies and must be collected and returned to the FEC for disposition.
- The collection, handling and proposed disposal method must be specified in the approved study plan or QAPP.

4 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW must be specified in the approved study plan or QAPP for the study or investigation. Hazardous IDW must be disposed as specified in USEPA regulations. If appropriate, these wastes may be placed back in an active facility waste treatment system. These wastes may also be disposed in the source area from which they originated if doing so does not endanger human health or the environment.

If on-site disposal is not feasible, and if the wastes are suspected to be hazardous, appropriate tests must be conducted to make that determination. If they are determined to be hazardous wastes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to allow arrangements for proper containerization, labeling, transportation and disposal/treatment in accordance with USEPA regulations.

The generation of hazardous IDW should be minimized to conserve Division resources. Most routine studies should not produce any hazardous IDW, with the possible exception of spent solvents and, possibly, purged groundwater. The use of solvents during field cleaning of equipment should be minimized by using solvent-free cleaning procedures for routine cleaning and decontamination (see SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205). If solvents are needed, the volume should be minimized by using only the amount necessary and by capturing the residual solvent separately from the aqueous decontamination fluids (detergent/wash water mixes and water rinses).

At a minimum, the requirements of the management of hazardous IDW are as follows:

- Spent solvents must be left on-site with the permission of site operator and proper disposal arranged.
- All hazardous IDW must be containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

Table 1: Disposal of IDW

TYPE	HAZARDOUS	NON - HAZARDOUS
PPE-Disposable	Containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.
PPE-Reusable	Decontaminate as per SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, if possible. If the equipment cannot be decontaminated, containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Decontaminate as per SESDPROC-205, and return to FEC.
Spent Solvents	Containerize in original containers. Clearly identify contents. Leave on-site with permission of site operator and arrange for proper disposal.	N/A
Soil Cuttings	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in a 55-gallon steel drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Groundwater	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Decontamination Water	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. Decontamination water may also be disposed in a sanitary sewer system, with permission from the wastewater treatment plant representative, and if doing so does not endanger human health or the environment, or violate federal or state regulations.
Disposable Equipment	Containerize in DOT-approved container or 5-gallon plastic bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. If unfeasible, return to FEC for disposal in dumpster.
Trash	N/A	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.

**** These materials may be placed on the ground if doing so does not endanger human health or the environment or violate federal or state regulations.**

Appendix E

EPA, Region 4, Field Sampling Procedures: Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples, SESDPROC-209-R3

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Packing, Marking, Labeling and Shipping of Environmental and Waste Samples**

Effective Date: February 4, 2015

Number: SESDPROC-209-R3

Authors

Name: Art Masters
Title: Environmental Scientist, Regional Expert
Signature:
Date: 2/4/2015

Approvals

Name: John Deatrick
Title: Chief, Field Services Branch
Signature:
Date: 2/2/15

Name: Hunter Johnson
Title: Field Quality Manager, Science and Ecosystem Support Division
Signature:
Date: 2/4/2015

Revision History

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History	Effective Date
SESDPROC-209-R3, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R2. **Cover Page:** Changes made to reflect reorganization of SESD from two field branches to one: John Deatrick listed as the Chief, Field Services Branch. The FQM was changed from Liza Montalvo to Hunter Johnson. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history.	February 4, 2015
SESDPROC-209-R2, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R1.	April 20, 2011
SESDPROC-209-R1, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R0.	November 1, 2007
SESDPROC-209-R0, Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by Department of Transportation under 49 CFR, Subchapter C, Hazardous Materials Regulations, and the International Air Transport Authority (IATA), which is equivalent to United Nations International Civil Aviation Organization (UN/ICAO). Transportation of hazardous materials (dangerous goods) by EPA personnel is covered by EPA Order 1000. This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when packing, marking, labeling and shipping environmental and waste samples to ensure that all shipments are in compliance with the above regulations and guidance.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when packing, marking, labeling, and shipping environmental samples and dangerous goods by air transport. Samples collected during field investigations or in response to a hazardous materials incident must be classified prior to shipment, as either environmental or hazardous materials (dangerous goods) samples.

In general, environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, biological specimens, or any samples not expected to be contaminated with high levels of hazardous materials. Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods.

Government employees transporting samples or hazardous materials (i.e., preservatives or waste samples) in government vehicles are not subject to the requirements of this section in accordance with 49 CFR 171.1(d)(5). EPA contractors, however, are not covered by this exemption and may not transport these materials without full compliance with 49 CFR.

Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

Title 40 Code of Federal Regulations (CFR), Pt. 136.3, Identification of Test Procedures, July 1, 2001. See Table II, Footnote 3.

Title 49 CFR, Pt. 171.1(d)(5), Applicability of Hazardous Materials Regulations (HMR) to Persons and Functions.

United States Department of Transportation (US DOT). 2003. Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No. 02-0093, February 13, 2003.

US Environmental Protection Agency (US EPA) Order 1000.18, February 16, 1979.

US EPA. 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version.

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when packing, marking, labeling, and shipping environmental or waste samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional.

2 Shipment of Dangerous Goods

The project leader is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of the waste sample or highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid is strictly regulated. Consult the IATA Dangerous Goods Regulations for guidance. ***Dangerous goods must not be offered for air transport by any personnel except SESD's dangerous goods shipment designee or other personnel trained and certified by IATA in dangerous goods shipment.***

3 Shipment of Environmental Samples

Guidance for the shipment of environmental laboratory samples by personnel is provided in a memorandum dated March 6, 1981, subject "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples". By this memorandum, the shipment of the following unpreserved samples is not regulated:

- Drinking water
- Treated effluent
- Biological specimens
- Sediment
- Water treatment plant sludge
- POTW sludge

In addition, the shipment of the following preserved samples is not regulated, provided the amount of preservative used does not exceed the amounts found in 40 CFR 136.3 or the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version. This provision is also discussed in correspondence between DOT and EPA (Department of Transportation, Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No.: 02-0093, February 13, 2003). It is the shippers' (individual signing the air waybill) responsibility to ensure that proper amounts of preservative are used:

- Drinking water
- Ambient water
- Treated effluent
- Biological specimens
- Sediment
- Wastewater treatment plant sludge
- Water treatment plant sludge

Samples determined by the project leader to be in these categories are to be shipped using the following protocol, developed jointly between USEPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

Untreated wastewater and sludge from Publicly Owned Treatment Works (POTWs) are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

Environmental samples should be packed prior to shipment by air using the following procedures:

1. Allow sufficient headspace (ullage) in all bottles (except VOA containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).

2. Ensure that the lids on all bottles are tight (will not leak).

3. Place bottles in separate and appropriately sized polyethylene bags and seal the bags. If available, the use of Whirl-Pak bags is preferable, if unavailable seal regular bags with tape (plastic electrical tape).

4. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape inside and outside. Line the cooler with a large heavy duty plastic bag.

5. Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers.

6. Put "blue ice" (or ice that has been "double bagged" in heavy duty polyethylene bags and properly sealed) on top of and/or between the containers. Fill all remaining space between the containers with absorbent material.

7. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

8. Place the Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) into a plastic bag, and tape the bag to the inner side of the cooler lid.

9. Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

Appendix F

EPA, Region 4, Quality System Procedures: Field Sampling Quality Control, SESDPROC-011-R5

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Sampling Quality Control**

Effective Date: April 26, 2017

Number: SESDPROC-011-R5

Authors

Name: Timothy Simpson
Title: Life Scientist
Signature:
Date: 04/20/2017

Approvals

Name: John Deatrick
Title: Chief, Field Services Branch
Signature: John Deatrick
Date: 4/24/17

Name: Hunter Johnson
Title: Field Quality Manager, Science and Ecosystem Support Division
Signature:
Date: 4/20/17

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History	Effective Date
SESDPROC-011-R5, *Field Sampling Quality Control,* replaces SESDPROC-011-R4 **Cover Page:** SESD's reorganization was reflected in the authorization section by making John Deatrick the Chief of the Field Services Branch. The FQM was changed from Bobby Lewis to Hunter Johnson. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **General:** Corrected any typographical, grammatical and/or editorial errors. Changed name of Enforcement and Investigations Branch to Field Services Branch. Removed references to Ecological Assessment Branch. Added Section 2.9 to the Table of Contents. **Section 1.4.6:** Added definition for Organic-Free Water **Section 3.5:** Changed volume needed for soil MS/MSD samples from triple to double volume. **Section 4.1:** Modified statement to read**:** Each lot of chemical preservative will be tested for the appropriate analytes by either FEC Staff or the Branch QAO.	April 26, 2017
SESDPROC-011-R4, *Field Sampling Quality Control,* replaces SESDPROC-011-R3	February 5, 2013
SESDPROC-011-R3, *Field Sampling Quality Control*, replaces SESDPROC-011-R2	October 15, 2010
SESDPROC-011-R2, *Field Sampling Quality Control*, replaces SESDPROC-011-R1	January 28, 2008
SESDPROC-011-R1, *Field Sampling Quality Control*, replaces SESDPROC-011-R0.	October 19, 2007
SESDPROC-011-R0, *Field Sampling Quality Control*, Original Issue	February 5, 2007

TABLE OF CONTENTS

1 General Information

1.1 Purpose

This document describes procedures established to ensure the quality of SESD field sampling activities, including Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations. Collectively, these procedures ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures, resulting in the collection of representative samples. Strict adherence to these procedures forms the basis for an acceptable field sampling quality assurance program.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling samples in the field and when preparing sampling equipment for SESD field investigations. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Sample

A part of a larger lot, usually a volume, area, period or population.

1.4.2 Variability

The range or “distribution” of results around the mean value obtained from samples within a population. There are three types of variability which should be measured or otherwise accounted for in field sampling, depending on the data quality objectives (DQO) for the study:

1. Temporal Variability

   Temporal variability is the range of results due to changes in contaminant concentrations over time. An example would be the range of concentrations obtained for a given parameter in wastewater samples collected at different times from an outfall where contaminant concentrations vary over time.

2. Spatial Variability

   Spatial variability is the range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations obtained for a given parameter in surface soil from a site where discreet "hot spots" are present due to localized releases of contaminants on otherwise uncontaminated soil.

3. Sample Handling Variability

   Sample handling variability is the range of results due to the sample collection and handling techniques used by the sampler. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, etc., or a negative bias due to improper containers or sample preservation.

1.4.3 Grab Sample

An individual sample collected from a single location at a specific time or period of time. Grab samples are generally authoritative in nature.

1.4.4 Composite Sample

A sample collected over a temporal or spatial range that typically consists of a series of discrete, equal samples (or “aliquots”) which are combined or "composited." A composite sample represents the average characteristics of the population under consideration. Four types of composite samples are listed below:

1. Time Composite (TC) – a sample comprised of a varying number of discrete samples or “aliquots” collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.

2. Flow Proportional Composite (FPC) – A sample consisting of discrete samples or “aliquots” collected at a rate proportional to flow. The aliquots are collected during the compositing period by either a time-varying/constant volume (TV/CV) method (“automated flow proportioning”) or a time-constant/varying volume (TC/VV) method (“manual flow proportioning”). The TV/CV method is typically used with automatic samplers that are paced by a flow meter. The TC/VV method is a manual method that individually proportions a series of discretely collected aliquots. The FPC is

typically used when sampling wastewater.

3. Areal Composite – a sample composited from individual, equal aliquots collected on an areal or horizontal cross-sectional basis. Each aliquot is collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.

4. Vertical Composite – a sample composited from individual, equal aliquots collected from a vertical cross section. Each aliquot is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries.

1.4.5 De-ionized Water

Tap water that has been treated by passing it through a standard de-ionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. De-ionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for de-ionized water.

1.4.6 Organic-Free Water

Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.

1.4.7 Branch Field Equipment Manager

Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager will be designated for the Field Services Branch (FSB).

1.5 References

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005-most recent version)

SESD Operating Procedure for Competency and Proficiency Testing, (SESDPROC-006, most recent version)

SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108-most recent version)

SESD Operating Procedure for Sediment Sampling (SESDPROC-200-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206-most recent version)

SESD Operating Procedure for Soil Sampling (SESDPROC-300-most recent version)

SESD Operating Procedure for Waste Sampling (SESDPROC-302-most recent version)

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version

Loan-In Form (SESDFORM-011, most recent version)

SESD Operating Procedure for Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209, most recent version)

SESD Operating Procedure for Training (SESDPROC-007-most recent version)

SESD Operating Procedure for Corrective Action (SESDPROC-009-most recent version)

SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101-most recent version)

2 Field Sampling Quality Control Considerations

This section provides guidelines for establishing quality control procedures for sampling activities. Strict adherence to all of the standard operating procedures outlined in this subsection forms the basis for an acceptable sampling quality assurance program.

2.1 Experience Requirements

There is no substitute for field experience. This field experience will be gained by on-the-job training using the "buddy" system. Each new investigator will accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior investigators. Specific requirements covering experience, competency and proficiency are found in the SESD Operating Procedure for Competency and Proficiency Testing (SESDPROC-006) and SESD Operating Procedure for Training (SESDPROC-007).

2.2 Traceability Requirements

All sample collection and measurement activities will be traceable through field records to the person collecting the sample or making the measurement. All maintenance and calibration records for sampling and measurement equipment (where appropriate) will be kept so that they are similarly traceable. The SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108) contain specific procedures to be followed that ensure traceability.

2.3 Chain-of-Custody

Specific chain-of-custody procedures are included in SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005). These procedures will ensure that evidence collected during an investigation will withstand scrutiny during litigation. To assure that procedures are being followed, it is recommended that field investigators or their designees audit chain-of-custody entries, tags or labels, field notes, and any other recorded information for accuracy. Additionally, the SESD FQM will randomly conduct reviews of project files to ensure that quality procedures are being followed.

2.4 Sampling Equipment Construction Material

Sampling equipment construction materials can affect sample analytical results. Field investigators will ensure the sample equipment construction material will not introduce contaminants to the sample being collected.

2.5 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservatives required for routine analyses of samples collected are found in the USEPA Region 4 Analytical Services Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM). Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the Branch Field Equipment Manager. All samples requiring preservation should be preserved immediately upon collection in the field. Records of sample preservation, including ice, will be documented in the field log books.

Samples that **should not** be preserved in the field are:

1. Those collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials which may be highly reactive. Barrel, drum, closed container, spillage or other source samples from hazardous waste sites are not to be preserved with any chemical.

2. Those that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved according to the ASBLOQAM.

All samples preserved with chemicals will be clearly identified by indication on the sample tag or label that the sample is preserved. If samples normally requiring preservation were not preserved, field records should clearly specify the reason. Samples shipped by air will not be preserved with nitric acid, hydrochloric acid, sodium hydroxide or sulfuric acid in excess of the amount specified in the ASBLOQAM.

2.6 Sample Collection Precautions

In order to prevent cross-contamination during sample collection, the following precautions will be taken:

1. A clean pair of new, non-powdered, disposable latex or nitrile gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come into contact with the media being sampled.

2. Sample containers for source samples or samples suspected of containing high concentrations of contaminants will be placed in separate plastic bags immediately after collecting, tagging, etc.

3. If possible, environmental (low concentration) samples and source or waste samples (high concentration) should be collected by different field teams. If different field teams cannot be used, all environmental samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or

highly contaminated samples should never be placed in the same ice chest as environmental samples. Ice chests or shipping containers for source or waste samples or any samples suspected to contain high concentrations of contaminants will be lined with new, clean, plastic bags.

4. If possible, one member of the field sampling team should record all of the field notes, collect GPS data, etc., while the other members collect the samples.

5. When sampling surface water and sediment at the same location, the water sample should always be collected before the sediment sample is collected.

6. Sample collection activities should proceed progressively from the least suspected contaminated area to the most suspected contaminated area.

7. Investigators should use equipment constructed of Teflon®, stainless steel, or glass that has been properly pre-cleaned according to either the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compounds analyses. Teflon® or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC should <u>not</u> be used to collect samples for trace organic compounds analyses.

8. Field investigators should ensure the sample containers they are using have been verified as suitable for the analyses that will be conducted on the samples through the quality control procedures discussed in Section 4 of this procedure.

Upon returning from the field, un-used sample containers will be examined by project leaders to determine whether bottles should be discarded, recycled or re-shelved for use on other projects. A load-in form (SESDFORM-011) will be completed and signed by project leaders to identify the future use of sample containers returning from the field. Opened boxes of sampling containers that can be re-used, will be segregated from sealed boxes of new containers.

Opened bags of latex or nitrile gloves returning from the field will be segregated from unopened gloves and will not be re-used for sample collection on other projects.

2.7 Sample Handling and Mixing

Once a sample has been collected, it may have to be transferred into separate containers for different analyses. Sample transfer should be done as soon as possible. If necessary, aqueous samples may be collected into a single, larger container for homogenization and transferred into individual sample containers. However, aqueous samples collected for volatile organic compounds, oil and grease, bacteria, sulfides and phenols analyses may not be transferred using this procedure.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is representative of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually.

2. Two quarters should then be mixed to form halves.

3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction and occasionally turning the material over.

2.8 Special Handling of Samples for Volatile Organic Compounds Analysis

Water samples to be analyzed for volatile organic compounds should be stored in 40-ml septum vials with screw cap and Teflon®-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the field investigation.

The vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied and some overflow is lost, but the air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Soil and sediment samples for VOC analyses should be collected and handled as specified in the SESD Operating Procedure for Soil Sampling (SESDPROC-300), Waste Sampling (SESDPROC-302) or the SESD Operating Procedure for Sediment Sampling (SESDPROC-200). Soil and sediment samples collected for VOC analyses should <u>not</u> be mixed.

2.9 Sample Storage and Transport

After collection, sample handling should be minimized. Field investigators should use extreme care to ensure that samples are not contaminated during storage. Environmental and waste samples are typically stored in coolers. To reduce the risk of cross contamination, smaller sample containers such as 8 ounce glass jars, 40 ml VOA vials, and one-liter amber bottles should be placed inside of sealed, plastic bags before being placed in the cooler. If ice is required for preservation of the samples, the ice should be contained in a plastic bag or some equivalent container to prevent the potential for cross contamination of the samples by water produced from melting ice. If ice is used, the coolers should be checked regularly and water should be drained as needed. Custody of samples will be maintained according to the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

Samples will either be transported to the analytical laboratory by field investigators or shipped by common carrier. Shipping of samples will be conducted in accordance with the SESD Operating Procedure for Packing, Marking, labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209).

3 Quality Control Samples

Quality control samples are collected during field studies for various purposes, among which are to isolate site effects (control samples), to define background conditions (background sample), and to evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples, etc.).

3.1 Control Sample

A control sample is typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

3.2 Background Sample

A background sample (usually a grab sample) is collected from an area, water body or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

3.3 Variability Samples

Variability may be defined as a variation in concentrations of compounds or analytes across a site or area of investigation or variations, across time, of waste streams or surface water bodies. Variation can also be introduced during sample handling. The following procedures are used to assess and evaluate variability. When appropriate, spatial duplicate grab and/or composite samples should be collected during investigations and studies in accordance to the project DQOs. In general, no more than ten percent of all samples should be collected as spatial duplicates.

3.3.1 Spatial Variability Duplicate

The following spatial duplicate sampling procedures should be used during the collection of samples as a measure of variability within the area represented by the sample. These samples should be collected at the same time, using the same procedures, the same type of equipment, and in the same types of containers as the original samples. They should also be preserved in the same manner and submitted for the same analyses as the required samples.

Spatial variability duplicate samples are typically collected during investigations where samples are collected from grids that are positioned at fixed intervals over the study area and a sample collection pattern is established within the grids. Spatial variability duplicate samples are collected using the same compositing pattern as the original sample and are collected within the same general area of representativeness, however the pattern is shifted relative to the original aliquot locations. This amount and direction of shift for the duplicate sample is dependent upon the size of the grid or area being sampled and should be specified in the QAPP for the investigation. Data from spatial duplicates will be examined by the investigation project leader to determine if the observed spatial variability is acceptable, based on the investigation or study objectives.

3.3.2 Temporal Variability Duplicate

When appropriate, temporal variability at a given sampling location will be measured by collecting temporal duplicate samples. These samples will be collected from the same sampling location, using the same techniques and the same type of equipment, but at a time different from the original sample. The time selected for the temporal duplicate sample will be similar to the time or span of time specified for the original sample in the project work plan. Data from temporal duplicates will be examined by the project leader to determine if samples represent the time span intended in the project work plan.

3.3.3 Sample Handling Variability

The effectiveness of sample handling techniques will be measured by collecting split and blank samples.

<u>Split Samples</u>

Split samples will be collected by initially collecting twice as much volume as is normally collected. The material will be apportioned, after mixing, if appropriate, into two sets of containers. Both sets of containers will be submitted for analyses with one set designated as an "original sample," the other designated as a "split sample." Data from the split samples will be examined by the project leader to assess sample handling variability. On large studies (more than 20 samples collected), a minimum of 5 percent, but no more than 10 percent, of all samples will be collected as split samples unless required by site data quality objectives.

<u>Blank Samples</u>

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site.

1. Water Sample VOC Trip Blank - A water sample VOC trip blank is required for every study where water samples are collected for VOC analysis. Sealed preserved (or unpreserved, if unpreserved vials were used during the investigation) 40-ml VOC vials will be transported to the field. Two sealed VOC vials will be submitted per trip blank sample. At least one trip blank sample will be submitted per sample shipment. Trip blanks will be prepared by lab personnel. Investigators should submit their request for trip blanks at least one week in advance of scheduled field investigations and inspections and never

(except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up earlier than the morning of departure for the scheduled inspection/investigation. These trip blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample labels and Chain-of-Custody Records as trip blanks.

2. Soil/Sediment Sample VOC Trip Blank - A soil/sediment sample VOC trip blank is required for every study where soil and/or sediment samples are collected for VOC analysis. The required containers are specified the USEPA Region 4 ASBLOQAM. The request and pick up of the soil blank sample will be the same as for the water trip blank. En Core® containers will be transported to the field. These blanks will be handled and treated by field investigators in the same manner as the soil samples collected for VOC analysis on that particular study. These samples will be clearly identified on sample labels and Chain-Of-Custody Records as trip blanks. Two sealed En Core® containers will be submitted per trip blank sample. At least one set of trip blank samples will be submitted per sample shipment.

The following blanks are prepared in the field:

1. Sample Preservative Blanks - SESD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. If preservative blanks are collected, sample containers will be filled with de-ionized water by SESD personnel and transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample labels and the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. In addition, one preservative blank will be collected for each multi-use bottle of preservative used.

   Note: The deionized water will be generated from a water treatment unit provided by the SESD laboratory.

2. Equipment Rinsate Blanks - Equipment rinsate blanks will be collected whenever field decontamination of equipment to be re-used in sampling activities is performed.

   When field cleaning of equipment is required during a sampling investigation, a piece of the field-cleaned equipment will be selected for collection of a rinse blank. At least one rinse blank will be collected during each week of sampling operations. After the piece of equipment has been field cleaned and prior to its being used for sample operations, it will be rinsed with organic-free water. The rinse water will be collected and submitted for analyses of all constituents for which normal samples collected with that piece of equipment are being analyzed.

3. Organic-Free Water System Blanks - When using a portable organic-free water generating system in the field, a sample of the water generated by the system will be collected at least once during each week of operations. Based on the objectives of the study or investigation, it may be appropriate to collect a sample of the raw source water. The collected water sample will be submitted for analyses of all constituents for which normal samples are being analyzed.

4. Material Blanks - When construction materials are being used on a site in such a way as to have a potential impact on constituent concentrations in the sample, a sample of each material will be submitted for analysis.

   Note: For drilling operations where materials are shipped directly to the site from the supplier, see SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101) for material blank collection and reporting requirements.

5. Automatic Sampler Blanks - In general, cleaning procedures outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) should be adequate to ensure sample integrity. However, it is the standard practice of the Field Services Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for organic compounds and metals analyses. Automatic sampler blanks for other standard analyses may be submitted in the event of a special investigation (e.g., criminal or civil).

6. Field Blank - A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample). Organic-free water is taken to the field in sealed containers or generated on-site. The water is poured into the appropriate sample containers at pre-designated locations at the site. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

7. Temperature Blank - A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 6°C (ice). The temperature of the blank is measured at the time of sample receipt by the laboratory. No temperature blank is necessary for waste samples since waste samples do not require ice for preservation.

8. Wipe Sample Blank - A wipe sample blank is a sample of the material and solvent used for collecting wipe samples. The blank is handled, packaged and transported in the same manner as all other wipe samples with the exception that it is not exposed to actual contact with the sample medium.

9. Water Filter Blank - When filters are used for sampling a dissolved constituent, de-ionized water should be run through at least one filter from each lot and the filtered water submitted for the same analyses. When filters are used for chlorophyll sampling, the filter should be prepared using de-ionized water and submitted for the same chlorophyll analysis.

3.4 Spikes

Spike samples are used to measure bias due to sample handling or analytical procedures. Spike samples are typically used by SESD to evaluate the performance of contract laboratories and are shipped directly to the CLP laboratory by the ESAT contractor.

3.5 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Organic Compounds Analyses

Matrix spike and matrix spike duplicate (MS/MSD) samples will be submitted to the laboratory for volatile organic compounds, extractable organic compounds, pesticides/PCBs and/or herbicides analyses from at least one sampling location per project and laboratory used. One MS/MSD sample should be collected per 20 samples per media collected.

Additional volume will be required for the soil MS/MSD samples. Semi-volatile organic compounds, pesticides, and PCB analyses of soil/sediment samples require the collection of one additional eight ounce glass jar. For VOC soil/sediment samples, double volume, i.e., six En Cores® or six 40 ml vials with syringe collected sample, is needed for the MS/MSD samples.

Additional volume will be required for the water MS/MSD samples. For routine full scan analysis, i.e., extractable organic compounds, pesticides and PCBs, four one-liter amber containers provide the required sample volume. Eight containers, therefore, should be submitted for the MS/MSD sample. For VOC water samples, a total of six 40 ml vials should be collected.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. The duplicate samples should be clearly identified as "Duplicate Sample for Matrix Spike" or “MS/MSD” on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.6 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Inorganic Analyses

A matrix spike sample and a duplicate sample (MS/MSD) will be submitted to the laboratory for inorganic analyses from at least one sampling location per project and laboratory used. One matrix spike and duplicate sample should be collected per 20 samples per media collected per laboratory.

Soil/sediment and water samples collected for inorganic analyses will normally have sufficient sample volume to perform the matrix spike analyses without requiring the collection of extra sample volume. The project leader should designate a sample, typically one considered to be representative of background or relatively uncontaminated conditions, as the matrix spike sample. For water samples, the sample volume collected will normally provide adequate volume for the MS/MSD analyses.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. MS/MSD samples should be clearly identified as "Duplicate Sample for Matrix Spike" or “MS/MSD” on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.7 Special Quality Control Procedures for EPA Contract Laboratories

On a case-by-case basis, field investigators may be required to collect split samples (or duplicate samples if appropriate) for analyses by either the Region 4 SESD laboratory or contract laboratories. The split samples are to be submitted to the Region 4 laboratory using established procedures. The contract laboratory involved <u>will not be notified</u> that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the Region 4 SESD laboratory.

3.8 Special Quality Control Procedures for Dioxins and Furans

The Region 4 laboratory does <u>not</u> conduct in-house analyses for dioxins and furans. Dioxin and furans analyses are conducted by contract laboratories. The Region 4 laboratory may accept environmental samples (soil, sediment, groundwater and surface water) suspected of being contaminated with polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF), as long as suspected PCDD and PCDF contamination is not due to RCRA hazardous waste classified as F020-023 and/or F026-

028. If these environmental samples are not contaminated with an F020-023 and/or F026-028 waste, it may be analyzed for parameters other than dioxin and furans. Environmental samples known or suspected to be contaminated with the RCRA hazardous waste F020-023 and or F026-028 will not be accepted.

NOTE: Environmental samples suspected of being contaminated with RCRA hazardous waste classified in 40 CFR, 261.31 as F032 will be accepted. The F032 waste is defined as wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that currently use or have previously used chlorophenolic formulations. The F032 listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and or pentachlorophenol. Prior to a sampling event, the project leaders should consult with the Analytical Services Branch Sample Control Coordinator to determine if the Region 4 laboratory can accept the samples. The Region 4 SESD laboratory should also be consulted for the current quality control procedures for dioxins and furans samples prior to a sampling investigation.

4 Internal Quality Control Procedures

The focus of this section is on Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations, as well as, field data generated under the specific sample collection quality control procedures discussed in Section 2. Quality control checks of these operations ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures.

4.1 Traceability Requirements

Records, will be kept by designated SESD staff or FEC personnel documenting the dates of operations and the person performing operations for the following:

1. Organic-Free Water System Maintenance (FEC System) - Maintenance on the FEC organic-free water system will be performed at least once per 180 days.

2. Air Monitoring Safety Instrumentation Checkouts - Pre-loadout checks on safety monitoring instrumentation will be recorded each time they are performed. Discrepancies will be immediately reported to the Branch Safety Officer.

3. Self Contained Breathing Apparatus (SCBA) Checkouts - Pre-loadout checks on SCBAs will be recorded when they are performed. SCBA checkouts will be performed at least once per calendar quarter in the absence of loadout requests. Any discrepancies will be reported immediately to the Branch Safety Officer.

4. Other Equipment Maintenance - Maintenance performed on equipment other than that listed above will be accordance to the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). All required repairs will be reported to appropriate Branch Field Equipment Manager.

5. Tubing, Sampling Containers and Latex Gloves - The Field Services Branch Quality Assurance Officer (FSB QAO) is responsible for conducting verification sampling for tubing, sample containers, and latex gloves that are used during field investigations. Upon receipt, the tubing, containers and gloves are placed in the quarantine room at the FEC. A record is kept of the lot numbers for each shipment received. The FSB QAO, or designee, will collect blank samples from tubing, containers and gloves within each lot received and will review the results to ensure the sample containers and gloves are suitable for use during field investigations. Once the supplies are deemed suitable, the FSB QAO will release the items for use.

6. Chemical preservatives commercially purchased will be tested prior to use. Each lot of chemical preservative will be tested for the appropriate analytes by either the

Branch Field Equipment Manager or the FSB QAO. Once released by FSB QAO, the preservatives can be used in the field.

7. Equipment - All equipment cleaned and wrapped for field use will be marked with the date on which preparation was completed. Equipment will be stored at the FEC in specified areas to minimize the risk of contamination while awaiting use.

4.2 Specific Quality Control Checks

When collecting samples during field investigations, it is necessary to take measures to prevent cross contamination to ensure the integrity of the data generated. The field branches conduct verification sampling of sample containers, gloves, sampling equipment, tubing and water utilized during field investigations as one of these measures. At least once per calendar quarter, the FSB QAO will conduct the following checks and issue a written report to the Field Quality Manager with the results.

1. Collect and submit for analyses samples of each new lot of containers received, Bottles from each lot will be tagged and sealed, then submitted for the following analyses:

    1. 1-liter Amber – extractable organics, pesticides, and PCBs.

    2. 8-oz. Clear Glass – metals, cyanide, extractable organics, pesticides, PCBs, and volatile organic compounds.

    3. 1-Liter Polyethylene – metals and cyanide.

    NOTE: In addition to the quality control checks listed above, samples may be collected during field investigations for classical inorganic parameters such as nitrates, nitrites, sulfides, etc. Due to the detection levels generally required for these parameters, it is unlikely that cross contamination may occur in association with the sample containers and sampling equipment used during sample collection. Therefore, classical inorganic analyses are not conducted as part of the routine quality control checks. If the data quality objectives require additional quality control checks, bottles will be submitted to the laboratory for analyses.

2. Collect and submit for analyses a rinsate blank for each new lot of latex or nitrile gloves received during the calendar quarter. Samples will be collected as rinse blanks using organic-free water. The rinsate will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. A new glove will be rinsed for each parameter (e.g., one glove for the VOC sample, another glove for metals, etc.) to avoid dilution of potential contaminants on the gloves. Water for the VOC samples should be provided by the ASB laboratory.

3. Collect and submit for analyses a sample of water from the FEC organic-free water system. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs.

4. Collect and submit for analyses a rinsate blank of at least one piece of sampling or sample related equipment stored at the FEC. The sample will submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

5. Collect and submit for analyses a rinsate blank for each new lot of Silastic® or Tygon® tubing used in peristaltic pump head. The sample will be submitted for metals and cyanide analysis.

6. Teflon® tubing – Collect and submit for analyses a rinsate blank for each new lot of Teflon® tubing received. Rinse blanks will be collected through the Teflon tubing. The sample will be submitted for metals, cyanide, extractable organics, volatile organic compounds, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

4.3 Quality Control for Special Order Equipment and Supplies

Some equipment and supplies ordered for specific projects are received in what can be considered ready to use condition. In order to ensure the integrity of these materials, an equipment rinsate blank will be collected from at least one item in each lot. The equipment and supplies will not be used until the QAO has reviewed the analytical data for the blanks and released the items.

4.4 Quality Control Evaluation and Corrective Action

All field investigation reports will contain a clearly identified section where the results for all field generated quality control (QC) samples are discussed and reported. Quality control data review includes but is not limited to detections of organic and inorganic compounds at any concentration in quality control blanks (i.e., trip blanks, equipment rinsate blanks, portable organic-free water system blanks, etc.).

All detections of organic and inorganic compounds will be immediately reported to the FSB QAO. The project leader will analyze of the results to determine if the source of contamination can be identified. If the source of contamination cannot be determined by the project leader, the branch QAO will conduct an additional review of the results to assess the source of contamination. If the source of contamination cannot be determined, the QAO will monitor all quality control results generated by the branch and assess the data for trends of contamination.

If it is determined by the project leader and the FSB QAO that the contamination adversely impacts the data collected during the investigation, the project leader will report the results to their Section Chief and the FQM. The project leader, in consultation with management, will determine whether the impacted data are usable or should be rejected. If data are rejected, the project leader and their management will determine whether samples must be recollected.

Data reported to the FQM will be analyzed to determine if the contamination is due to non-conforming work. If it is determined by the FQM, in consultation with management, that the contamination is due to non-conforming work, a corrective action is warranted and will be selected and implemented in a timely manner. If a corrective action is required, it must be implemented and reported according to the SESD Operating Procedure for Corrective Action (SESDPROC-009). If contamination is not due to non-conforming field work, then the source of contamination will be identified, if possible, and documented by the FQM. If the source of contamination cannot be determined, FQM will monitor all quality control results generated by SESD and assess the data for trends of contamination.

4.4.1 Quality Assurance Reports

It is each project leader's responsibility to ensure that a copy of the quality assurance data from each field investigation report is provided to the FSB QAO, who will compile a quarterly report of field quality assurance data and forward the report to the FQM.

The FQM will prepare an annual quality assurance report based on the quarterly reports. This report will be distributed to all field investigators each year and will document and discuss all quality control issues or trends identified during the data review. This report will be retained by the FQM to document that QC measures have been taken, that the QC measures are appropriate, that the QC results are acceptable or, if not, that corrective actions were taken.

Appendix G

AES Laboratory Asbestos QAM and Excerpts of QAM for Lead

Analytical Environmental Services, Inc.
3080 Presidential Drive
Atlanta, GA 30340-0370

SOP No.: QA-01014
Date Initiated: 06/92
Date Revised: 7/16/18
Revision No. 26

Standard Operating Procedure for the
Asbestos
Quality Assurance Manual
(NVLAP PLM and TEM)

Analytical Environmental Services, Inc.
3080 Presidential Drive
Atlanta, Georgia 30340-3704
(770) 457-8177
FAX (770) 457-8188

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"2018 Asbestos QA Manual"

Mehmet Yildirim 7/31/18
Mehmet Yildirim, Laboratory Manager **Date**

7/30/18
Yelena Khanina, Bulk Asbestos Lab QA Officer **Date**

7/31/18
Mort Soltani, Air Asbestos Lab Asbestos Manager **Date**

7/30/18
Douglas Mendrala, Quality Assurance Manager **Date**

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1.0 - INTRODUCTION

Analytical Environmental Services, Incorporated (AES) specializes in the analytical testing sectors of the asbestos industry. AES has implemented a quality control and quality assurance (QAQC) program. This program was developed by management to establish a baseline of standards insuring routine performance satisfactory to governmental agencies and our clients.

This quality control/assurance manual summarized the routine employed by AES's laboratory. The person in charge of the laboratory, as well as the implementation of this manual, is the Laboratory Manager. On a yearly basis, the Laboratory Manager insures this manual is reviewed and updated.

The quality of the laboratory service provided to the clients of AES directly affects their competitive and profit position in the marketplace. Therefore, it is in the best interest of AES and its clients that the services provided are of the highest quality and analytically defensible to current state-of- the art standards.

To achieve these necessary goals AES will provide effective quality and management systems. To this end AES will provide the proper tools, training and operating standards to promote an environment where the talents and energies of all AES associates are directed toward continued quality improvement. Only by constant attention to quality improvement and variation reduction can we ensure that client expectations for quality will be met.

The purpose of the Quality Assurance Program undertaken by AES is to provide analytical data of known and supportable quality. This program is also to insure a high professional standard in the date generated for said analysis by its technical staff. This is accomplished by the procedures and policies found herein.

2.0- QUALITY CONTROL/QUALITY CONTROL ELEMENTS

2.1 - CHAIN OF CUSTODY

AES has implemented sample chain-of-custody procedures to provide accurate, verified, and traceable records of sample possession and handling, from sample container shipment through laboratory receipt and sample disposition.

Documentation of sample collection, shipment, laboratory receipt and custody is accomplished utilizing a chain-of-custody record. A sample is considered in custody if the following conditions have been met:

1. The sample(s) are in the physical possession of the sampler or courier.

2. The sample bottle(s) are labeled and sealed, so that sample integrity is maintained, while in the possession of the sampler or transferee.

3. The sample bottle(s) are in a secured area restricted to authorized personnel.

Laboratory records, including copies of the chain-of-custody forms and any associated documentation, are maintained in a secure area with any associated project records.
The sample custodian receives all samples. Legal custody is a special type of sample custody in which all events associated with a specific sample are documented in writing.

For each batch of samples received in the lab, a PLM ‘Chain of Custody’ form (Exhibit #1) is filled out. The client is responsible for filling out all applicable information, as well as, listing the samples being submitted. The client then signs and notes the date and time the samples are submitted. The AES representative receiving the samples also signs the Chain of Custody form and notes the date and time. The condition of the package and the condition of the samples is observed, also. The samples are then checked to verify they are consistent with the information provided by the client on the Chain of Custody. The client, project information and samples are then entered into the Laboratory Information Management System (LIMS) and the computer automatically assigns a project number. Each sample is assigned a unique AES ID number and labeled accordingly. A ‘Chain of Custody Record (COCR)’ (Exhibit #2) is used to track the project progress within the laboratory. It is also used to document receipt of rush samples. A ‘Sample Receipt Check List’ (Exhibit #2) is used to document observations at sample receipt. If a discrepancy is found, the variance is noted on this form and the client is contacted to clarify the problem. Initial results are entered on a ‘Preliminary Bulk Asbestos Report’ (Exhibit #3).

2.2 - CRITERIA FOR ACCEPTANCE / REJECTION OF PLM SAMPLES

Upon the receipt of the package in the laboratory, the package is examined to see if it has been damaged in such a way that any container of the asbestos sample has been broken or deformed, the lid has been opened, etc. In any of the above cases, some of the samples may not be acceptable for testing. The client I.D. number on each sample container must be legible and not ambiguous, and verified against the transmittal form. In the case of any discrepancy, the sample could be rejected for analysis and noted on the checklist.

2.3 - PLM ANALYTICAL PROCEDURES

The analytical method employed by AES to analyze bulk samples for the presence of asbestos is the EPA method entitled *Method for the Determination of Asbestos in Bulk Building Materials* (EPA/600/R-93/116); hereinafter referred to as the "method." EPA PLM method ‘*Interim Method for the Determination of Asbestos in Bulk Insulation Samples* /EPA-600/M4-82-020’ (and subsequent clarifications for the use of "equivalent volume estimation techniques") is maintained in this document. In that the EPA allows for volume estimation, the laboratory employs techniques for its day-to-day analysis from; *Analytical Methods for Asbestos Products* by W. C.

McCrone of the McCrone Research Institute, Chicago, Illinois. This method serves as the industry benchmark for stereoscopic and volume estimation basic analysis routine and serves as the basis for the current EPA method.

The 1994 EPA PLM method is an excellent improvement over previous versions in that it includes a much more through treatment for other analysis methods including; point counting, gravimetry and diffraction. Many nonfriable samples (such as floor tiles, roofing materials etc.) require additional preparation techniques and/or analysis techniques because of extremely small fiber size or masking matrices. This revised method is the best comprehensive reference currently available from a regulatory body that addresses these analysis issues. If necessary or requested, AES uses gravimetry / ashing techniques found in the method for non-friable samples (E.g. floor tile and roofing materials). Additionally, AES uses techniques described in *Determination of Asbestos in Resilient Floor Tile,* by Dr. Eric Chatfield. Diffraction techniques (TEM) are available if needed or if requested by the client.

The above will serve as a base for all standard analytical methodology employed by AES. Any analysis technique outside that of the standard visual estimation technique will be so noted on the final client report. Various reference articles, regulatory clarifications that assist in this can be found in the appendices. Analysts should all have a demonstrable knowledge of the method and related reference materials.

It is required that all analysts performing bulk sample analysis at AES fulfill the background and training requirements set forth in this document. Therefore, specific methods pertaining to the proper use of the polarized light microscope for the determination of optical properties will not be provided here. Instead, the reader is referred to standard optical mineralogy texts (Bloss, Kerr, Phillips, D-H-Z, McCrone, etc.), which are vastly superior to any explanations that could be given within the scope of this document. These references provide detailed and accurate presentation of the theory and applications of the PLM. They also serve as an excellent source of reference information for the determination of mineral species.

2.3.1 - Analysis Sequence

In order to perform the analysis, samples are transferred from their storage containers into the freshly cleaned sampling pan with caution. This transfer takes place under a negative air environment provided by a HEPA vacuum and blower unit. This procedure protects our microanalysts from being exposed to asbestos during the transfer. The microanalyst insures that a representative subsample is analyzed from the sample container. This negative air system pulls all fibers released during analysis away from the microscopist, trapping them in the HEPA filter.

If the sample is wet it will be placed in an oven and heated at 150 degrees Fahrenheit until it is dried. The purpose of this action is to avoid change of optical characteristics of fibers due to absorbed water on the fiber surface. If the sample is hardened and physical abrasion will be required to analyze internal components, the microanalyst will do it safely under the negative vacuum system.

Once the sample is prepared and dried, if necessary, an initial gross stereomicroscope examination will be performed. Sample analysis should proceed by using the lowest magnification to observe the general features of the sample, such as: color, homogeneity, or inhomogeneity, layering, structures, etc. If necessary, higher magnification may be used to determine the characteristics of each component present in the sample. The visual volumetric percentage estimates should be done at lower magnification.

When suspect fibers are found, they are mounted in the refractive index liquid and examined under the polarized light microscope for more detailed study. Using PLM techniques and measuring optical properties including indices of refraction, pleochroism, color, birefringence, extinction angle, and sign of elongation accomplish final identification of each component. The above properties for each asbestos fibrous component are recorded on a ‘Preliminary Bulk Asbestos Report’ (Exhibit # 3). For non-asbestos fibrous components, only the predominant optical property is noted. A comprehensive list of optical properties for asbestos and non-asbestos fibers can be found in the “method” on pages 19-20 (Tables 2-2, 2-3). AES utilizes Dr. Shu-Chun Su’s *Rapidly and Accurately Determine Refractive Indices of*

Asbestos Fibers by Using Dispersion Staining Method (Rev. 2010-07-11) for determining refractive index of all asbestos species (see appendices). All analysts will demonstrate proficiency with this method during in house training (See Training Program for PLM Method attachment). Room temperature is recorded to identify the refractive index of fibers. Analysts will also be able to use the Becke line technique as necessary to determine refractive index measurements.

After all components have been identified, a visual volumetric percentage relative to the volume of the sample must be given by using both stereomicroscopic study and PLM information. All materials are separated into four individual categories including asbestos fibers, non-asbestos fibers, non-fibrous components and others. The percentage is adjusted to 100%.

After analysis, the samples are returned to their storage canisters under the HEPA hood system. The canisters are cleaned to insure that there are no fibers on the exterior. The canisters are then placed into either an asbestos or non-asbestos bag (sorted by month) and placed in a storage room where they are kept for a period of three months.

The allowable tolerance limits for refractive index measurements are currently +/- 0.004. If any fluid is outside of these values it is to be replaced. Dr. Su's refractive index determination method may be used in this endeavor.

2.3.2 - Calibration of Refractive Index

Procedure for Determination of Refractive Index by Using Dispersion Staining Methods

1. Select the proper immersion oil for the suspected fiber (e.g. chrysotile 1.550, tremolite, actinolite and anthophyllite 1.620, amosite and cricidiolite 1.700).
2. Measure the temperature of the immersion oil. It can be reasonably assumed that the oil temperature is the same as the room temperature.
3. Check alignment of the Polarized Light Microscope.
4. Observe the dispersion staining (DS) color associated with α of the asbestos fibers. Make sure the DS color that gives the lowest refractive index is observed.
5. Convert the observed DS color and the corresponding matching wavelength between the asbestos fiber and the immersion oil used (See attached dispersion staining color chart (McCrone, 1987).
6. Find the numerical value of α corresponding to the observed wavelength and temperature. Refer to Dr. Su’s tables to convert to the corresponding refractive index.
7. Observe the DS colors associated with γ of the asbestos fibers. Rotate the microscope stage 90° and then repeat steps 4-6 to determineγ.

2.3.3 - Dealing with Problem Binders

Commonly, asbestos fibers may be obscured by powdered calcium carbonate. The use of a weak HCL solution will suffice to remove enough interfering material for fiber identification. The slide must be dried before refractive index fluid is used.

2.3.4 - Dealing with Non-Friable Samples

For other interfering materials, the ashing technique found in the gravimetric methods referred to in this document is the most probable solution to allow for minor identification. TEM will be used if requested. Notes should be made for any sample using ashing or diffraction techniques and a reference should be made on the report to the client.
Other fibers (polyethylene, wollastonite, etc.) can be misidentified as asbestos. Various charts and references are found in this document that describes these. The most comprehensive list that also details optical properties is found in the method on page 20-21 (Tables 2-4, 2-5).

STANDARD OPERATING PROCEDURE FOR DEALING WITH
SEMI- OR NON-FRIABLE SAMPLES

1. Fracture the sample and examine it using the stereo-binocular microscope.

2. Use a clean scalpel to shave off thin layer of the sample. The shaving shall be taken from the full cross-section of the sample by cutting perpendicular to the plane of the sample, avoiding any mastic if present on under-side. This insures that the sample is representative of all of the layers. Approximately 100-500 mg of shaving required.

3. Weigh a crucible, place the sample in it and re-weigh.

4. Place the lid on the crucible containing the sample and place it in a muffle furnace with temperature 350-450°C. Leave in the furnace for 6 hours.

5. Remove the crucible from the furnace, and allow cooling to room temperature.

6. Remove the lid and weigh the crucible & residual ash.

7. Weigh the cellulose membrane filter, 0.45um.

8. Set up a glass filtration assembly, using a vacuum flask. Install the filter in the filtration assembly.

9. Add approximately 0.5ml of distilled water in to the crucible w/ ash and grind the material to disperse it.

10. Slow add approximately 2ml. of concentrated hydrochloric acid. When no more evolution of gas is observed, dilute the suspension with more water.

11. Apply the vacuum to the filtration assembly, pour the suspension into the filter reservoir, and add about 50 ml. of distilled water. Allow the filtration to proceed to completion. Rinse with approx. 10 ml. of water twice.

12. Transfer the filter w/ residue in Petri-dish. Put small piece of paper towel under the filter to prevent sticking as it dries.

13. Allow the filter to dry using heat lamp or slide warmer.

14. Weigh the filter w/ residue.

15. Analyze residue using PLM or TEM Method.

16. Calculate relative weight % asbestos in sample.
    a) Subtract weight of filter from total weight (crucible + residue)
    b) Divide residue weight by starting weight and multiply by 100 to determine weight % residue.
    c) Wt% asbestos in sample = % asbestos in residue x wt % residue÷100. For samples, if the ashing procedure was used first, multiply the weight % of asbestos in the sample, as determined above, by weight percent of residue from ashing procedure, the divide by 100.

Example:

A = wt% residue from ashing = 70%
B = wt% residue from HCl = 20%
C = wt% of asbestos in HCl residue = 50%

Wt% asbestos after HCl dissolution = B x C ÷ 100 = 20 x 50 ÷100 = 10%
Wt% asbestos in sample = (B x C ÷ 100) x A ÷ 100= 10x70 ÷100 = 7%

2.4 - CONTAMINATION CONTROL

A cleaning procedure for all analytical stations is sustained. However, it remains the responsibility of the individual analyst to insure that the glassware and sampling instruments used in the performance of the analytical procedure are properly cleaned. Dispersion liquids, glass slides, cover slips and sampling instruments analyzed daily are to check for contamination. Contamination has to be checked daily prior to any analysis. If a contaminated liquid is found, it is to be disposed of after thoroughly checking that the source of the contamination is not on the slides or cover slips. Blank samples are analyzed weekly. The results are recorded and tracked on a 'Contamination Control Testing' sheet (Exhibit #4).

2.5 - INSTRUMENT AND MATERIAL PERFORMANCE

To check the instrument and material performance the calibration and its frequency are conducted as follows:

- The entire instrumentation system is checked daily.

- A daily microscope alignment is performed by the PLM Laboratory QA Manager (Appendix 1) and recorded on the 'Microscope Alignment Sheet' (Exhibit #5). In the case that the microanalyst cannot fix or make alignment, or if any problem arises that he or she cannot solve, the Laboratory Manager will be informed. In the case that the Laboratory Manager cannot solve the problem, the microscope should be out of service until the service personnel resolve the problem.

- Cargille glass slides used for calibrating refractive index liquids when a new bottle is opened or every six months**.**

- An outside service company performs a yearly check of the entire optical system.

2.6 - SAFETY

Safety in the laboratory must be practiced on a continual basis, (See Appendix 2).

1. All samples are treated as hazardous.

2. Routine laboratory safety procedures are followed as necessary. These include wearing proper personal protective equipment as laboratory coats, gloves, etc.

3. Housekeeping is an important aspect to insure a safe environment. Analysts are required to keep their work areas clean and free of clutter.

4. Proper sample storage and disposal procedures are followed.

5. Internal audits monitor safety in the laboratory.

6. Corrective actions are submitted to management for review.

2.7 - DISPOSAL OF ASBESTOS

- Dispose of slide, cover slips, paper towel, and any other material contaminated by asbestos as follows:

- During the analysis of bulk samples, a zip lock bag should be kept under the working hood, under continuous HEPA ventilation.

- At the end of the day these bags shall be disposed of in a central bag labeled “Asbestos Waste” and sealed.

- Asbestos waste is regularly removed and transported to a landfill. An asbestos removal company shall perform the handling and transportation of the waste.

2.8 - Quality Control

Quality Policy

The objective of Analytical Environmental Services, Inc. is to generate high quality data in a cost-effective manner, which is accurate, impartial, reliable, and adequate for its intended use. The management of AES is committed to following accepted laboratory practices to achieve high quality of testing services, and strives to ensure both the analytical validity and legal defensibility of all reported data.

AES management is committed to establish, implement, and maintain a quality system appropriate to the scope of all laboratory activities, including the type, range, and volume of testing.

Management shall document the policies, systems, programs, procedures, and instructions to the extent necessary (*compliance with NVLAP Handbook 150, 150-3, and 150-13*) and International Standard (ISO/IEC 17025) to enable AES to assure the quality of the test results generated. Quality system documentation is communicated to, understood by, and made available to personnel through AES management by means of training and educational instruction.

All laboratory staff concerned with analytical testing activities must familiarize themselves with the quality documentation and implement the policies and principles in their work.

It is the policy of AES to continually improve quality systems and provide support to improvement efforts.

Estimation of Uncertainty
A reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data. It is defined by the monthly checks, proficiency exam results and error rates.

2.8.1 - INTERNAL QUALITY CONTROL PROCEDURE FOR DETERMINATION OF PRECISION

The closeness of agreement between randomly selected individual measurements of test results is defined as precision. It is also defined as repeatability of the results of a particular experiment or to the agreement among repeated measurements of the same quantity. The precision of each PLM analyst is determined by sample reanalysis (replicate/duplicate analysis).

To insure quality, AES has developed the following system of checks and balances to evaluate analytical performances.

1. The Quality Assurance Program tracks monthly precision via a ‘Monthly Precision Summary Report’ (Exhibit #7) for each analyst. Two analysts view each sample. The primary analyst generates the sample report and the secondary analyst rechecks 20% of the samples against the original analysis.

   The R value in percent (a.k.a. % RPD) is calculated for each pair of results. This value is the precision and is expressed as follows:

$$\text{R Value, \%} = \frac{A - B}{(A + B) / 2} * 100$$

where: A = original analyst result
B = second analyst result

The standard deviation (S) is then generated from analyst R values as follows:

$$s = \sqrt{\frac{\sum(x - \overline{x})^2}{N - 1}}$$

x = each R value

$\bar{x}$ = the mean of R values

N = the total number of values

Upper and lower warning limits are established using ± 2 standard deviations from the centerline. In addition, upper and lower control limits established at ± 3 standard deviations from the centerline. After the data has been tabulated for each analyst, the information is used to create control charts using EXCEL spreadsheets. These control charts are updated periodically (usually monthly) when sufficient additional data points are available. Charts are reviewed for trends by the Department Quality Assurance Manager and the Corporate Quality Assurance Manager. In addition, false positives, false negative, and misidentification of asbestos types are recorded in the 'Monthly Precision Summary Report'.

2. Each analyst reanalyzes no less than 5% of the samples analyzed as blind samples. Blind samples are selected by the QC Officer and re-analyzed as unknowns. Separate sample sheets will be completed for these QC samples in the same way they are for the original samples. The results of the analyses are compared against those originally determined by the same analyst for conformity of results. Data is tabulated and reflected on a 'Blind Recount Form' (Exhibit #6).

3. The current EPA method has been demonstrated (published papers and professional presentations; Perkins & Harvey, various) to be equivalent to point counts in most cases. AES will use the point counting technique as needed or when requested by the client.

4. An internal audit program has been implemented to check activities at every stage of our laboratory's analysis and results reporting process.

5. Internal checks to the laboratory are conducted and documented periodically to insure that all laboratory supplies and reagents meet specifications and that the cleanliness of the laboratory remains at a level acceptable for the type of work performed.

6. AES maintains a reference collection of samples, which are, used by the analyst to insure that their mineral identification and estimation techniques remain consistent with standards established by current practice. In order to evaluate the precision and accuracy of our analysis, we have implemented a project in which standard samples with known asbestos concentrations are analyzed on a monthly basis. These samples are past NIST rounds, round robin samples and purchased standards. The results are used to determine the precision and/or accuracy of each microanalyst, and also to see if the results are in the acceptable range announced by NVLAP.

7. WORKING DEFINITION OF TRACE
The working definition of "Trace" distinguishes between trace concentrations of asbestos and those near 1 %. "Trace" refers to a concentration of asbestos fibers far enough below 1 %, as determined by calibrated visual estimation, that the analyst is confident it is not necessary to pursue additional verification. Whereas asbestos reported as <1 % indicates

it is nearer to but less than 1 % even though the analyst ascertains that the asbestos concentration is not equal to or higher than 1% by calibrated visual estimation. Point counting or TEM confirmation recommended in these instances.

Using the point count method: If asbestos appears in the field of view but does not lie directly under a point, the analyst must note this on the analysis sheet. The sample is reported to be trace for that type of asbestos.

At trace (≤ 1 %) percentages, it may be possible that such an amount may not be detected in all cases of reanalysis. Volume estimation percentage determination is determined from conventional comparison charts.

Expertise is gained from ongoing training with low percentage reference samples and comparison with point counted samples. If there is any question in the re-analysis process, if such samples are indeed > or < 1%, point-counting techniques as described in the EPA PLM method should be employed. Any asbestos content >1% must be detected. The PLM Laboratory QA Manager should document the identification or quantitative difference as a major or minor error as defined below. Any corrective actions taken should be documented to the analysts file.

2.8.2 - EXTERNAL QUALITY CONTROL PROCEDURE FOR DETERMINATION OF ACCURACY

The accuracy of measurement is the difference between the average of several measurements and the true value or the degree of agreement of a measured value with the true or expected value of the quantity of concern. The accuracy of asbestos identification and counting of each PLM analysts and the laboratory is determined by analysis of reference materials and NIST proficiency testing.

The laboratory will participate in an external testing program with no less than 2 other laboratories. This program consists of one of the participating laboratories generating no less than 4 samples that are sent “round-robin” to the other participants for full PLM analysis. The samples should be a combination of: past NIST rounds or purchased standards (repackaged), typical samples seen in day-to-day analyses and point counted samples.

Upon completion of analyses by all laboratories, the originating laboratory will gather all data and provide a numeric summary for all participating laboratories for their study. It will be the responsibility of the next laboratory in line to generate the next round and summaries. The samples from each round are returned to the originator. This activity will occur no less than semiannually, and it will be the responsibility of the Laboratory Manager to insure its scheduling and completion.

This is valuable information to all laboratories in that it provides for input on the overall accuracy and mineral identification ability of each laboratory and analyst. These samples are extremely valuable as they are now characterized by multiple analysts and will serve as important additions to the set of standards used for all internal testing. These and NIST samples should be used as “spikes” for duplicate analysis. This data will be invaluable for the measurement of precision and accuracy for each analyst and the laboratory as a whole. These results are recorded in the PLM ‘Summary of Monthly Accuracy’ Sheet (Exhibit #10).

2.9 - JOB RESPONSIBILITIES FOR TECHNICAL STAFF

Organization

Analytical Environmental Services, Inc. is a locally owned, permanent laboratory facility that performs asbestos, chemical and biological testing on a variety of environmental samples.

Management System and Organizational Structure

The relationship between management, technical operations, support services and quality system is as follows: Laboratory Operations, Quality Assurance Department, Technical Director, and Customer Service Department report to the Vice President of Operations, who in turn reports to the company President. The asbestos departments also report directly to the Vice President of Operations with the PLM and TEM QA Officers reporting to the (corporate) Quality Assurance Manager. The organizational structure of AES provides for an independent Quality Assurance Department with the overall responsibility of developing and auditing for compliance to a comprehensive Quality Assurance Program. The QA Department has the authority and organizational freedom to ensure that QA activities are implemented and accomplished. The Quality Assurance Manager reports directly to the Vice-President of Operations of AES. PLM and TEM sections each have a Department Manager to which analysts report.

At AES, Inc. analysts and technicians are insulated from work-related undue pressures that would compromise the quality of their work. Management is aware and considerate of these internal pressures such as management burdens and project deadlines, and of external stresses such as customer complaints and priority requests for analysis.
Management policy is to remain supportive of laboratory personnel and aware of their workloads and the demands placed upon them. Precautions are taken to ensure that there are no conflicts of interest between staff and clients. For example, priority requests, complaints, or status of work inquiries are directed through supervisors, managers, or administrative personnel.

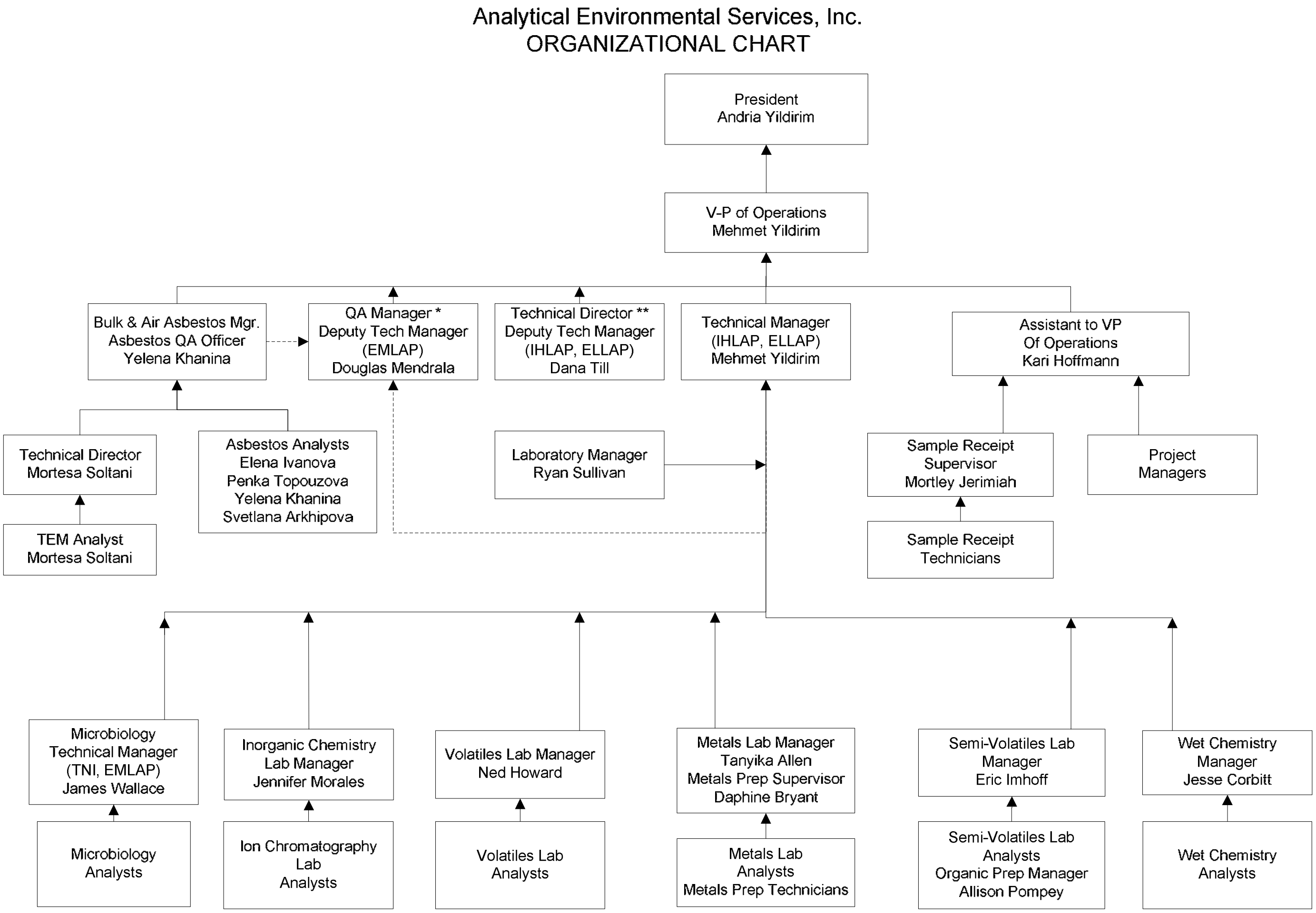
Legal and Ethical Policy

For a quality assurance program to succeed, it is imperative that all employees adhere to procedures which detect and prevent improper, unethical, or illegal actions which could in any way compromise the reliability and integrity of the laboratory. Training in legal and ethical responsibilities is mandatory and is performed using the company Standard Operating Procedure (SOP AD-02008). Records are maintained that document, through individual signatures, that every employee understands the consequences of improper, unethical, or illegal actions.

Structure

The organizational structure at AES is documented in the form of an organizational chart, Figure 2-1, which identifies the personnel involved in the production of quality data and depicts the lines of communication and responsibility throughout the entire organization.

Figure 2-1, ORGANIZATIONAL CHART



* The QA Manager will serve as deputy in the event of the Technical Director's absence.
** The Technical Director will serve as deputy in the event of the QA Manager's absence.

2.9.1 - Chain of Custody Officer

A. <u>Requirements</u>:

Employee works in conjunction with that asbestos analysts and is familiar with the (EPA-600/R-93/116) requirements of asbestos bulk sample testing.

B. <u>Job Description:</u>

1. Preparation of chain of custody form for each project.

2. Checking the condition of arriving packages.

3. Acceptance or rejection of each sample based on the criteria mentioned in the Quality Control / Quality Assurance Procedure Manual.

4. Sample preparation, including:

    a. Checking the condition of each sample.
    b. Verification of client's sample I.D. number with transmittal form.
    c. Assign AES's serial number for each sample.
    d. Preparation of Analytical Data Sheet.

5. Checking the typed reports and making the corrections.

2.9.2 - Microanalyst

All new analysts will either attend a 5-day McCrone PLM training program (or equivalent) or be instructed in the proper in-house techniques by someone who has been qualified. Specific requirements for competency are outlined in the Training Program (Appendix 2) and this section. Each analyst signs a ‘Quality Assurance Manual Training Summary’ upon completion. Training files are maintained for each analyst.

AES will promote the ongoing education of its analysts through various mechanisms such as technical meetings, industry journals, and supplementary training programs.

A. Requirements:

1. A degree in Natural Sciences, Applied Sciences, or Physical Sciences and/or one or more years experience in the testing of asbestos under the NVLAP program.

2. Demonstration of proficiency analysis for 20 test sample submitted by the end of the training program.

B. Job Description:

1. Checking the instrument performance.

2. Sample Analysis (All steps must be conducted in accordance with published methodologies.

    a. Transferring the samples to analytical dish under HEPA vacuum.

    b. Stereomicroscopic study including: homogeneity or heterogeneity of samples, color, texture, presence or absence of fibrous components, preliminary fiber identification, non-fibrous component determination.

    c. PLM studies including: morphology, index of refraction, color birefringence, extinction angle, sign of elongation and dispersion of color.

    d. Visual volumetric percentage for each component.

    e. Recording of analytical result.

3. Safety consideration.

   a. Checking on HEPA vacuum system.

   b. Checking on stereoscope hood system.

   c. Careful handling of the asbestos samples.

   d. Using all facilities in a safe manner.

4. Contamination consideration.
   a. Daily cleaning of analytical station.

   b. Cleaning of glassware and sampling instruments.

   d. Checking contamination of dispersion liquid, glass slides, cover slips and sampling instruments by preparing slides.

5. To determine the precision and accuracy of each microanalyst, he or she should analyze the 12 internal standard samples per month.

6. Each microanalyst should reanalyze 5% of the total samples analyzed by each analyst for that month.

2.9.3 - PLM Laboratory Quality Assurance Manager

A. Requirements:

1. At least six months of experience in analysis of bulk samples and quality assurance procedures.

2. At least 1,000 samples analyzed by PLM technique.

B. Job Description:

1. To implement a Quality Control/Quality Assurance program insuring satisfactory performance to governmental agencies and our clients.

2. Checking the performance of all instruments.
3. Quality control on the chain of custody form and the quality of the work that has been done by the chain of custody officer.

4. Quality control on all samples analyzed by microanalyst and instruction of quality and quantity corrections of their analysis.

5. To instruct microanalyst about contamination and safety considerations. Also, to determine the precision and accuracy of the microanalyst.

6. Periodically checking the calibration of the analytical instruments.

2.9.4 – Bulk & Air Asbestos Manager

A. Requirements:

1. A degree in the Physical Sciences.

2. At least 1,000 samples analyzed by PLM technique.

B. Job Description:

1. Responsible for the day to day activities concerning the PLM Lab.
2. Quality control on the chain of custody form and the quality of the work that has been done by the chain of custody officer.
3. Instructing the chain of custody officer to make deficiency corrections.
4. Making sure that all samples are analyzed in time frame specified by the client.
5. Checking the laboratory report system.

The laboratory affords its clients and representatives cooperation by clarifying their request and monitoring the laboratory's performance relative to the work performed.

The Bulk & Air Asbestos Manager accomplishes this the following way:

- Ensures effective and accurate communication between the client and the laboratory.
- Handles all client requests and needs.
- Utilizes any corporate documents to consult with clients about client questions or concerns.
- Responsible for notifying the Customer Service Manager of any client activities that entail services that are not currently performed by AES.
- Assesses client requests in light of current workload with consultation with the Customer Service Manager.
- Develops and maintains client records and requirements.
- Ensures that the laboratory is aware of, and completes, all client requests and requirements.
- Responsible for meeting with the Marketing Manager, Customer Service Manager, and President on a periodic basis for marketing purposes.
- Communicates proper sampling, shipping, and receiving procedures to clients.
- Documents all client interaction and maintains all client information in the Project Management System.
- Reviews and approves data reports prior to their release to the clients.
- Ensures client specific reporting and quality control requirements are met.

2.10 – Review of Requests, Tenders and Contracts

Review of New Work

The Laboratory Manager is primarily responsible for determining the capacity of the facility and its resources to handle new work, although other senior members of management may be called upon to provide expertise and input as needed. This determination consists of a comprehensive appraisal of the client's projected needs. Factors assessed are the ability of the laboratory to comply with the requirements of its accreditations while maintaining the expected level of legal defensibility and analytical validity of all reported data.

Prior to the acceptance of any new requests, tenders, or contracts by Analytical Environmental Services, Inc., the appropriateness of facilities and resources is considered utilizing the information in the following sections. If the facility and/or resources are inadequate to perform the work, the Laboratory Manager may exercise his discretion to refuse to perform all or part of a particular project. The Client Services Manager will be informed of this decision and the Project Managers will inform the client.

1. Facilities - The facility must be suitable for the proper receipt and storage of the number and type of samples proposed to be accepted.

2. Methods - The specified methods must be adequately defined, documented, and understood.

3. Resources - Stipulated methods, sample preparations, final reports, data packages, and deliverables are reviewed to determine the availability of suitable instrumentation and personnel.

The laboratory shall advise and obtain approval from the client before subcontracting work to another laboratory.

4. Technical and Management Capability - The review of capability must establish that the laboratory possesses the necessary physical personnel, information, and resources to perform the tests in question. Additionally, the laboratory personnel must have the skills and expertise required for performing these tests.

The laboratory shall have adequate personnel at all times during the performance of analytical testing to ensure that clients receive data which meets the terms and conditions of the work agreement.

The review may consider the results of previous work of a similar nature or, where new testing is being implemented, the results of interlaboratory testing, trial tests, proficiency samples, MDL studies, etc.

5. Discrepancies - Any differences between the request or tender and the capability of the laboratory to fulfill the proposed work is resolved before any testing begins.

Modifications are allowed upon consent of the client. Each contract shall be acceptable to both the laboratory and the client.
Problems encountered during any stage of reviewing the testing are addressed and resolved to the satisfaction of both the laboratory and the client.

6. Records - The laboratory maintains any records for the initial review of new work entering the laboratory, including any significant changes in the proposed work plan.

Communication logs (telephone calls, on-site visits, meetings, e-mails, etc.) are used to record all pertinent discussions concerning the client’s requirements. Logs must include the date, time, brief details of the exchange, resolution of any complaints, and identification of the parties involved.

Subcontracted work is fully described & documented in advance of receipt of the work from client.

Once work has been accepted, the Customer Service Manager is responsible for setting up the client in the LIMS system, setting up an account with the client, and monitoring the project to ensure that all of the client's requirements are met.

2.11 - PROCEDURES FOR AUDIT AND REVIEW

The Laboratory Manager shall conduct audits annually.

Internal performance audits are a means for the Quality Assurance Department to determine the applicability, effectiveness, and utilization of procedures by all sections. Designated personnel perform the performance audits. At the beginning of each new year, and on an on-going basis, a schedule of audits and surveillance is developed and updated by the Quality Assurance Section. Surveillance is performed on an unannounced basis with the sections so that objectively may be maintained. Findings from audits and surveillance are documented and corrective actions are implemented. Additional surveillance is scheduled to ensure that all deficiencies are corrected.

An audit is performed using the 'Bulk Laboratory Audit Form' (Exhibit #8). The results of the audits are discussed with the QA/QC Officer and if necessary corrective actions are recommended. Corrective action shall be performed immediately.

The Laboratory Manager, following the QA/QC performs review of all analytical data. The Laboratory Manager is responsible for checking the data integrity, correct petrographic description, usage of the appropriate methods, etc.

2.11.1 - Annual Review

The annual report to management includes the following information:

1. SOP. The report indicates any changes to existing SOPs or any new SOPs.

2. Corrective action reports. The report contains information about any corrective action reports that may have been written during the time period since the last QA report. (See SOP QA-01000 General Quality Assurance Manual for the procedures for Corrective and Preventive Actions.)

    i. Audits. The QA report includes the results of any audits performed during the time period since the last report.

    ii. Proficiency Testing (PT) samples. The report includes the results of PT samples analyzed since the last report. The PT report indicates the status of performance as it relates to NVLAP (laboratory needs to satisfactorily complete the most recent two out of three PT samples for any given accredited category).

    iii. Certifications. Any changes or additions to the laboratory's certifications are addressed in the reports.

    iv. The annual report is reviewed and signed by the President Laboratory Manager, and the Bulk & Air Asbestos Manager. It includes elements of HB 150, HB 150-3, and HB 150-13 checklists. A copy of this report is kept for 5 years.

2.12 - CORRECTIVE ACTIONS

In the cases where QC data fails to meet the required limits, or the overall QA of the project is suspect; the Laboratory Manager should be notified immediately. Corrective actions shall be determined based on the severity of the breakdown and the requirements of the customer or in-house field department.

In general, major QC failures in asbestos mineral identification, precision and/or accuracy will require repeat measurements and/or sample preparation. A detected deficiency of the QA/QC plan will be dealt with on a project-by-project basis depending on the severity of the problem and/or the requirements of the customer or field department. Any QA/QC problem that has been detected by sources outside the daily operation of the laboratory should be cause for the Laboratory Manager to consider a general system analysis. In all cases the Laboratory Manager will make the final decision in the QA/QC failure.

Deficiencies or nonconformities in analytical procedures, materials, components or methodology may lead to the release of incorrect analytical results to the customer. Once a deficiency or non-conformance has been identified, corrective actions must be implemented to insure proper data qualification and narration on the final client report and, when possible, prevent the deficiency being repeated. To document and track the non-conformance, a Corrective Action Report (CAR) is issued through the LIMS system. An example of a Corrective Action Report is at the end of this section.

Standard Procedure for Defining, Implementing, and Closing a Corrective Action Report (CAR):

Non-conformance: A non-conformance is defined as any situation that is either outside acceptable limits (data) or does not comply with the procedure/method in some way. The following situations are considered non-conformances and the completion of a CAR report is required. (E.g. Contamination in the Blank)

Procedure for the issuing, completing, and closing of an analytical or technical systems related CAR:

When a non-conformance occurs, the employee performing the work or the initial data reviewer must complete a CAR in the LIMS system as indicated below. When completing a CAR, include all accompanying data, information, etc in a "Data Package" along with the NCR and submit this to the Technical Director or Quality Assurance Manager for review.

General Procedures and Responsibilities for Corrective Action Reports Involving Deficiencies:

1. When the PLM Laboratory Quality Assurance Manager or Bulk Air & Asbestos Manager issues a corrective action report (CAR) for a non-conformance classified as a deficiency, the Laboratory Manager, Assistant Laboratory Manager, or Technical Director will be informed immediately.

2. The QA Manager will track the completion of the corrective actions required to correct the deficiency. The assigned personnel are responsible for completing the corrective action within the specified time frame.

3. The chain of custody and the Sample Receipt Forms are used to document non-conformance during log-in.

4. If the non-conformance is determined to be a deficiency, full QA review and documented corrective action to prevent re-occurrence is required. A root cause will be identified by the Quality Assurance Manager for deficiencies. Root causes will be categorized as one of the following: personnel, (LIMS) database, Quality Control, procedure, or laboratory controls.
    a. Personnel: Root causes attributed to personnel may require training or retraining to insure individuals understand their responsibilities in the process.

    b. Database: A Root cause from a database issue primarily refers to the Laboratory Information Management System (LIMS). This type of nonconformance will require the database to be updated. This may include method information (test codes), client information, project information, login entries, calculations, audit trail, and reports among others. Database root cause will also include individual instrument databases and software (GCs, ICPs, AA, Lachat autoanalyzers, etc.)

    c. Quality Control: QC root causes result from incorrect QC acceptance ranges in logbooks, LIMS or are the result of trend changes. These will be reviewed and updated as necessary.

    d. Procedure: This root cause covers procedures, policies, checklists, standard operating procedures (SOPs) that will be reviewed for modifications.

    e. Laboratory Controls: Root causes from instrumentation, software and equipment will be investigated. These may require maintenance, repair, or updates.

Preventive Actions
The Technical Director or QA Manager may determine that the nonconformance require a certain type of corrective active report called a preventive action report. Internal audits, management reviews and input from laboratory personnel shall be used to identify potential sources of nonconformance and needed improvements or preventive actions. If appropriate, a preventive action plan shall be developed, implemented and monitored that will reduce the likelihood of nonconformance. Preventive action shall include the application of controls to ensure that actions taken are effective, and may involve the analysis of data, additional auditing, control charts and trends, proficiency test results, and client complaint letters.

Method Suspension or Restriction:
In some cases, it may be necessary to suspend or restrict the use of a method that constitutes significant risk and or liability to AES. Suspension or restriction procedures can be initiated by the Quality Assurance Manager, Technical Director, Laboratory Manager, or VP of Operations.

Prior to suspension or restriction, confidentiality is respected, the problem and the required corrective action is stated in writing on the associated CAR and presented to the Laboratory Manager.

The Laboratory Manager, Technical Director, Quality Assurance Manager, and the affected supervisor are notified.

The Laboratory Manager arranges for the appropriate operations people to speak with the Quality Assurance Manager or Technical Director the day of notification. This meeting is held to confirm that there is a problem, and that suspension or restriction of the method is required.

The suspension or restriction meeting will conclude with a discussion of the steps necessary to bring the method or test fully back on line if the method is suspended or restricted. The Quality Assurance Manager will also specify any documentation necessary to verify that corrective action has occurred.

After suspension or restriction, the laboratory will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. It is the responsibility of the Laboratory Manager to hold all reports. Clients will not generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Upon completion of the required corrective actions per the CAR, laboratory management will determine if the affected systems are back in control and reports can be released. If systems are still deemed out of control, further corrective actions are required. A team, with all principals involved can devise a start up plan to cover all steps from client notification through compliance of method and release of reports.

If the PLM Laboratory Quality Assurance Manager or Bulk Air & Asbestos Manager recommends client notification regarding affects on past or current data quality, all associated information is forwarded to the Laboratory Manager and VP of Operations. These will review the data and determine appropriate actions.

Client notifications are the responsibility of the Laboratory Manager and VP of Operations.

The greatest overall priority of producing quality data is to reduce error rates of less than 1% on qualitative analysis. This is defined in this section as Major Errors. AES endeavors to produce data to meet this goal by monitoring these errors on a regular basis.

Errors will be classified as follows:
MAJOR ERRORS: False positive or false negative if asbestos is >1%, incorrect identification of an asbestos mineral species, a percentage estimation error (including precision and accuracy measurements for testing rounds) > +/- 25% of the mean data for a specific QC sample (for samples between 5-10%, > +/- 10%; for those samples < 5%, > +/- 1%).
MINOR ERRORS: False negative if asbestos percentage <1% or trace, percentage estimation error > +/- 15% of the mean data for a specific sample (> +/- 5% for samples between 5-10%), an incomplete lab bench sheet. Repeated omissions equal a major error for the sake of individual analyst QC summarization.
Corrective actions for errors are as follows:
MAJOR ERRORS - Immediate Action

1) Review all paperwork for numbering system or paperwork error.

2) Review sample, especially sample description, to insure that the sample analyzed matches the description in order to confirm the sample is the same material.

3) Reanalyze the sample with at least 3 slide mounts. Submit to other laboratories if necessary. Other techniques such as enhanced sample preparation techniques or diffraction techniques may be required.

4) Check for equipment contamination and equipment alignment

5) For identification error of asbestos species the analyst should review the appropriate literature as needed and review standard materials of similar make-up. The Laboratory Manager may choose to initiate a testing round so that the analyst can show proficiency. This should be required for any analyst that shows any tendency for this type of error. The Laboratory Manager should be prepared to suspend the analyst from the normal flow of sample analysis until proficiency is assured.

6) If needed, issue a corrected report as a supplement to the original report, if already sent.

MINOR ERRORS - 1 to 2 weeks after monthly summary

1) Repeat steps 1 to 4 as deemed necessary

2) Review procedures to ascertain if the error was based on a systematic bias.

3) Review lab bench sheet completion procedure and emphasize the importance of accuracy regarding this responsibility.

4) In general consider having the analyst practice with archived samples including non-friable materials (floor tiles etc.) until the overall accuracy measurements fall within with guidelines. Emphasis should be placed on more difficult mineral identification such as tremolite/actinolite. Review appropriate literature and methods as needed. If repeated errors occur, retraining in estimation should be considered.

In general, following the procedures and protocols set forth in this document can minimize the need for corrective actions.
Other General Corrective Actions for PLM Analysis of Bulk Samples:

1) When original analysis and QC analysis (replicate) of asbestiform minerals varies by 1% or more in samples containing less than or equal to 5% asbestos, the sample must be reanalyzed by a second analyst observing no less than 3 PLM mounts. Once a decision has been made as to the corrected percentage, amendments should be appropriately made if the ample results have not yet been sent to word processing. If found at a later date, a completed bench sheet will be added to the original job folder and attached

to the original bench sheet. In either case, this information will then be recorded in the QC logbook with appropriate notations made in the comments section to show that corrective actions have taken place.

2) When a client has questions regarding analytical results the sample will be pulled from the archives and all numbering information will be checked to insure that both the client and the laboratory correctly performed labeling. If the numbers appear to be correct the process of reanalysis will take place as enumerated above.

- If the results of the reanalysis match the original analysis results, the client is informed and will be asked to submit a duplicate sample for analysis. This will be necessary in the case of another laboratory receiving a split of the original sample. The requested duplicate sample is to insure that this laboratory received the same material as the other lab for the original analysis

- If the reanalysis does not match the original analysis, double check that all samples were analyzed in the proper numeric order. If in disorder, inform client and provide corrected results. If reanalysis shows that the error was on the part of AES, the error will be managed as described in above sections. A corrected report will be issued to the client.

2.13 - COMPLAINTS FROM CLIENTS

- Addressing complaints is a normal function of conducting business and a valuable tool to improve services to and relationships with clients. The goal of AES is to provide expeditious resolution of complaints. At AES, the supervisor and the management team handle complaints related to sample results. Client Services resolves specific complaints concerning container orders, shipping, expected report dates, and results. This information is documented in LIMS.

- In the event that a complaint is related to the laboratory's compliance with its own policies and procedures, the rules of an accrediting agency, or the validity of data, the PLM Laboratory Quality Assurance Manager and or Laboratory Manager initiate an internal audit of the areas involved. These personnel document the complaint, audit findings and recommendations.

2.14 - RECORD KEEPING SYSTEM

ANALYTICAL ENVIRONMENTAL SERVICES, INC. maintains all the original records in its archives. Records are saved as hardcopies as well as backup files on the tapes.

The record keeping system was developed by AES in consideration of the following:

1. Records have to be continuously maintained during the data entry process.
2. Records must be kept in a database capable of providing QA/QC elements. Records shall be backed up monthly.

3. All final reports receive a secondary review. The last person to review the final reports shall be the Laboratory Manager.

All computer applications that affect the system (from log-in to final reports) shall be backed-up on a regular basis. Duplicate back-up tapes and/or disks should be maintained off site to protect against catastrophic data loss from computer systems. All software applications that could be altered to change client information or analysis results should have sufficient password protection to prevent tampering. Appropriate "hard-copy' print outs of data based information should be reviewed periodically to ensure that transcription errors are <1% of the workload.

Document Control
Regardless of which analytical procedures are used in the laboratory, the methodology shall consist of carefully documented Standard Operating Procedures

(SOPs) and approved methods which may be periodically modified, updated or replaced entirely due to advances in technology or changes in regulatory protocols. Some clients may require pre-approval of method revisions before modifications are used to generate data. Documentation of analytical procedures for generating laboratory data shall be clear, concise, adequately referenced, and reflect the actual steps employed by the analyst.

Procedures
Methodologies employed in the laboratory are documented by the creation of an SOP. This document provides the analyst with the information necessary to perform the analysis. Every SOP is created in accordance with this QA document. It follows the intent of the method it is patterned after, but provides any additional information essential to the specific instrument instructions, specific quality concerns, etc.

If an SOP is not available for a specific analysis, the analyst will follow EPA, Standard Methods, NIOSH, recognized industry standards, or other regulatory methodology as required. No deviations of any kind are allowed.

All analytical procedures must provide documentation so that the complete process used to produce data can be reconstructed.

All deviations from an approved analytical procedure are authorized and documented by the Technical Director.
All changes to an approved procedure require, at a minimum, an Interim Change Notice. A complete revision and re-issuance of the SOP may be required. SOPs are reviewed at least annually.

A list of all current SOPs including their review and revision status is maintained electronically on AES_server\L\Current SOP\SOP Masterlist. Current SOPs are maintained electronically in the AES_server\L\Current SOP Laboratory SOP\Current Revisions\. These documents are also available from the AES Portal Server in the Technical Management folder. All controlled documents are in “Read Only” format and password protected. The Vice-President of Operations, QA Manager, Technical Director and their appointees are the only laboratory employees with edit access to these folders.

2.15 - PROCEDURES FOR CONFIDENTIALITY AND PROPRIATORY RESULTS

The samples and the data generated from them are considered to be the property of the client.

NVLAP logo shall only be used on the final report. The analyst's signature on the final report constitutes acknowledgement of NVLAP accreditation.

AES, Inc., will not intentionally divulge to any person (other than a client or person designated by a client in writing) any information regarding the services provided by AES or any information disclosed to AES by the client. Any information *known* to be potentially endangering to national security or any entity's proprietary rights will NOT be released.

Test results are reported according to client requirements. If a client requests to have reports or information sent by fax, the client is notified in advance of the transmission, whenever possible, and all documents include a cover sheet with the following statement:

NOTICE OF CONFIDENTIALITY

The information contained in this facsimile message may be legally privileged and is confidential information intended only for the use of the individual or entity named above. If the reader of this message is not the intended recipient, you are hereby notified that any use, dissemination, distribution or copy of this facsimile message is strictly prohibited. If you have received this facsimile message in error, please contact us by telephone at (770) 457-8177 and return the facsimile message to us at the address above via the US postal service.

2.16 - RECORD RETENTION

Analytical Environmental Services, Inc. shall retain all original observations, calculations and derived data, and test reports for a period of five years, unless otherwise instructed by the client. [Note: Attachment 1 (40CFR763 page 120), shows record storage dates of 3 years, which is not the laboratory policy. The laboratory's policy for record storage is 5 years. This attachment is included for reference.] If a client requires a longer retention time AES will impose a storage charge.

2.17 - SAMPLE RETENTION

Analytical Environmental Services, Inc. shall retain samples for a period of three months. After this time the samples will be disposed of unless otherwise directed by the client.

2.18 - NVLAP LOGO

NVLAP logo shall only be used on the final report. The analysts' signature on the final report constitutes acknowledgement of NVLAP certification.

1. The term and logo shall not be used in a manner that brings NVLAP into disrepute or misrepresents a laboratory's scope of accreditation or accredited status.

2. When the term *NVLAP* is used to reference a laboratory's accredited status, it shall be accompanied by the NVLAP Lab Code.

3. When the NVLAP logo is used to reference a laboratory's accredited status, it shall be accompanied by the NVLAP Lab Code in an approved caption. The caption shall appear below and in close proximity to the logo.

4. The form of the NVLAP logo must conform to the following guidelines:
   - The logo shall stand by itself and shall not be combined with any other logo, symbol, or graphic.
   - The aspect ratio (height to width) shall be 1 to 2.25.
   - The logo and caption shall be of a size that allows the caption to be easily read. The size of the caption shall not exceed the size of the logo itself.
   - The logo shall appear in black, blue or other color approved by NVLAP, and may be filled or unfilled. In the case of a filled logo, the same color shall be used for the outline and the fill.

5. The name of at least one Approved Signatory shall appear on a test or calibration report that displays the NVLAP logo or references NVLAP accreditation.

6. When the term and logo are used on test or calibration reports, such use shall be limited to reports in which some or all of the data are from tests or calibrations performed by the laboratory under its scope of accreditation.
   - A test or calibration report that contains both data covered by the accreditation and data not covered by the accreditation shall clearly identify the data that are not covered by the accreditation.
   - The report must prominently display the following statement at the beginning of the report: "This report contains data that are not covered by the NVLAP accreditation."

7. When the term and logo are used on test or calibration reports that also include work done by subcontracted laboratories, such use shall be limited to reports in which some or all of the data are from tests or calibrations performed by the laboratory under its scope of accreditation.
   - A test or calibration report that contains both data covered by the accreditation and data provided by a subcontractor shall clearly identify the data that were provided by the subcontracted laboratory.
   - The report must prominently display the following statement at the beginning of the report: "This report contains data that were produced under subcontract by

     Laboratory X." If the subcontracted laboratory is accredited by NVLAP, then its Lab Code should also be stated.
   - If the subcontracted laboratory is accredited by a body other than NVLAP, then the name of the accreditation body and the laboratory's number will be stated. If the subcontracted laboratory is not accredited, then this must be stated.

8. Each test and calibration report bearing the term or logo shall include a statement that the report must not be used by the client to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the federal government.

9. When used in a contract or proposal, the term and logo shall be accompanied by a description of the laboratory's scope of accreditation and current accreditation status.

AES will notify its clients in its final reports if subcontracted work is performed. *A statement will be included as follows if NVLAP accredited:*
"This report contains data which were produced by a subcontracted laboratory **ACCREDITED (NVLAP LAB CODE)** for the calibration or test methods performed"

If not NVLAP- accredited:
"This report contains data which were produced by a subcontracted laboratory **NOT ACCREDITED** for the calibration or test methods performed.

AES will issue a separate report for any analysis subcontracted or performed that does not fall within the NVLAP accreditation. (Lead, soil etc.)

2.19 - Subcontracting Analyses

If mineral identification or quantitation is beyond the training of the attending analysts and the Laboratory Manager, an experience opinion should be sought from another NVLAP accredited laboratory. In the case of non-friable material, the EPA and many industry professionals are suggesting various diffraction techniques (gravimetry, TEM, XRD) as mineral identification options. These options should be explored if asbestos mineral identification is in doubt. A statement of uncertainty should be forwarded to the client if additional techniques are not employed for any reason. If analysis is subcontracted AES will keep in its files, evidence of laboratory competence. This will include a copy of the hired laboratories NVLAP certificate and statement of qualifications. AES will place in its files evidence that it has investigated the qualifications, competence and compliance of all laboratories that receive subcontracted work.

All subcontract laboratories are required to supply the Quality Assurance Manager, upon written request, with adequate proof of accreditation in applicable state, NVLAP, or other programs, depending upon the client and origination of the samples. Documents shall be requested from all subcontract laboratories. The requested documents will include, but may not be limited to, a current Quality Assurance Manual, the scope of approved testing, proof of insurance, and NVLAP and/or applicable state accrediting authority certificates.

The client must be contacted in writing of the intent to subtract any portion of the testing to another party.

When samples are received which have testing requirements that cannot be performed in-house, the samples must be sub-contracted to another laboratory. The subcontract laboratory's scope will be checked to make sure it possess the proper accreditation for the requested test. The results from the subcontracted laboratory must be reported utilizing a copy of the original report received from the subcontract laboratory. By approving the subcontractor, the client has made the selection therefore assumes responsibility.

The following is a list of laboratories which may be utilized for the subcontracting of AES samples:

Material Analytical Services (MAS) 770-866-3200
3945 Lakefield Ct
Suwanee, GA 30024

The results from the subcontracted laboratory must be reported utilizing a copy of the original report received from the subcontract laboratory.

2.20 - Purchasing Services and Supplies

Procurement Document Control

Vendors of analytical material supplied to AES Inc. are regarded as a resource to and an extension of the laboratory organization. The standards for quality identified in this document shall be applicable to vendors.

The purpose of the procurement control document is to assure the quality and traceability of procured items (equipment, materials, or services) in instances in which the specifications could affect the quality of the services provided by AES, Inc. This includes such quality related items as the calibration of instruments by outside laboratories, purchase of standards, subcontracted services, and materials requiring testing before use.

Control of purchased materials, equipment, and services is a system designed to insure that these products and services conform to the procurement requirements. This system includes provisions for vendor evaluation and selection, objective evidence of quality furnished by the vendor, inspection of the vendor's or sub-vendor's facilities, and examination of products or services upon delivery. Prior to the use of such products and services, documented evidence of conformation to the procurement requirements must be provided. This evidence is maintained in the Laboratory Information Management System (LIMS).

It is the responsibility of the Accounting Department to insure the development and implementation of procedures to control purchased products and services. It is the responsibility of the purchasing agent to specify quality objectives for procured items and services. Purchased materials that fail to meet established criteria are documented by Non-conformance reports issued by the purchaser.

Procedures and Responsibilities

1. It is the responsibility of the purchasing agent to provide assurance, when required, that all applicable regulatory requirements, industry codes, and standards appear with the purchase documentation for the affected services and products.
2. The Purchasing Department retains Purchase Orders for control purposes.
3. Purchased items which do not meet the minimum standards set forth by the purchasing agent are processed according to procedures set forth in Section 2.12, "Corrective Action."
4. The appropriate manager or supervisor and QA Manager review purchase orders to insure that quality related services or products meet the criteria of the laboratory's accreditations.

2.21 - Facility / Environmental Conditions

The facility must be suitable for the proper receipt and storage of the number and type of samples proposed to be accepted.

Stipulated methods, sample preparations, final reports, data packages, and deliverables are reviewed to determine the availability of suitable instrumentation and personnel.

The Laboratory Manager is primarily responsible for determining the capacity of the facility and its resources to handle new work, although other senior members of management may be called upon to provide expertise and input as needed.

The laboratory controls and records environmental conditions as required by methods and procedures or where they influence the quality of results. Due attention shall be paid to dust, electromagnetic disturbances, humidity, electrical supply, temperature, and sound and vibration levels. Tests shall be stopped when environmental conditions jeopardize these results.

There shall be separation between areas in which there are incompatible activities. Measures shall be taken to prevent cross contamination.

There shall be good housekeeping in the laboratory.

Exhibit #1
Chain of Custody (COC)

Work Order: _______________

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ANALYTICAL ENVIRONMENTAL SERVICES, INC.
3080 Presidential Drive Atlanta, GA 30340-3704
Phone: (770) 457-8177 / Toll-Free: (800) 972-4889 / Fax: (770) 457-8188

CHAIN OF CUSTODY
BULK ASBESTOS ANALYSIS

Client Name: ____________________
Address: ____________________
City, State, Zip: ____________________
Contact: ____________________
Sampler's Name: ____________________
Report To: ____________________

Phone: ( ) ____________________
Email: ____________________
Project Name: ____________________
Project Number: ____________________
Sampling Date: ____________________
Invoice To: ____________________

	Sample ID	Sample Location/Description	Analysis Requested	Turnaround Time (TAT)	Comments
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Relinquished by: ____________________ Date/Time: ____________________
Received by: ____________________ Date/Time: ____________________
Relinquished by: ____________________ Date/Time: ____________________
Received by: ____________________ Date/Time: ____________________

Submission of samples to the laboratory constitutes acceptance of AES's Terms & Conditions. Samples received after 3PM or on Saturday are considered as received the following business day. If no TAT is marked on COC, AES will proceed with standard TAT.

FOR LAB USE ONLY

Lab Recipient: ____________________ Date/Time: ____________________ Method of Shipment: ____________________

Exhibit #2
'COC Record' / Sample Receipt Checklist

Analytical Environmental Services, Inc.
3080 Presidential Dr.
Atlanta, GA 30340

CHAIN OF CUSTODY RECORD

Company Name: ______________________ Turnaround: ______________
Project Name: ______________________ Project Number: ______________
AES Project ID: __________ Date In: __________ Due Date: ______________
Total Samples: __________ Range: __________ Total Analyzed: ______________

Shipping Information

Samples delivered to lab via:	Ground UPS courier	FedEx Std. client	FedEx Priority Overnight other	FedEx 2-Day
Time in:	AM PM			

Sample Information

Requested Analysis:	PLM	TEM	PCM	Gravimetry	Point Count

Received By:	**Time:**	AM PM
Logged In By:	**Time:**	AM PM

Reported By:	To:	Date:	**Time:** AM PM
	Reported via: telephone fax e-mail		
Reported By:	To:	Date:	**Time:** AM PM
	Reported via: telephone fax e-mail		
Comments:			

	Initials	Date	Comments
Analyzed by:			
QC'd by:			
Typed by:			
Login Reviewed by:			
Report Reviewer:			
Invoice Entered Into Peachtree:			
Report Mailed:			
Scanned:			

Payment Information

Credit Hold: Yes No	**OK to start:** Yes No	**OK to release:** Yes No

Payment Procedure:
take file to: A/R PM call for payment run CC on file call for CC approval online payment

Date Paid:	Amount:	**Form of Payment:** cash check online CC	PO#:

Other Workorders paid with this transaction: Yes No	If so, total credit card transaction amount:

Notes

Paperless?: Yes No

Exhibit #3
Preliminary Bulk Asbestos Report

Company Name:

PLMID: SampleID:

1 Description:

Asbestos Fibers						Non-Fibrous					
CHRYS	AMOS	CROC	ANTH	TREM	ACTIN	VERM	BIOT	MICA	PERLITE	AGGR	STYR
Non-Asbestos Fibers						Others					
SYNTH	MWOOL	FIBGL	CELL	A HAIR	ANTIG	AL	BIT	RESIL	GLUE	BIND	

2 Description:

Asbestos Fibers						Non-Fibrous					
CHRYS	AMOS	CROC	ANTH	TREM	ACTIN	VERM	BIOT	MICA	PERLITE	AGGR	STYR
Non-Asbestos Fibers						Others					
SYNTH	MWOOL	FIBGL	CELL	A HAIR	ANTIG	AL	BIT	RESIL	GLUE	BIND	

3 Description:

Asbestos Fibers						Non-Fibrous					
CHRYS	AMOS	CROC	ANTH	TREM	ACTIN	VERM	BIOT	MICA	PERLITE	AGGR	STYR
Non-Asbestos Fibers						Others					
SYNTH	MWOOL	FIBGL	CELL	A HAIR	ANTIG	AL	BIT	RESIL	GLUE	BIND	

4 Description:

Asbestos Fibers						Non-Fibrous					
CHRYS	AMOS	CROC	ANTH	TREM	ACTIN	VERM	BIOT	MICA	PERLITE	AGGR	STYR
Non-Asbestos Fibers						Others					
SYNTH	MWOOL	FIBGL	CELL	A HAIR	ANTIG	AL	BIT	RESIL	GLUE	BIND	

	Type	Refractive Index		Biref	Color	Pleo	Elong	Extinction Angle	Morphology
1				L M H	W Bl G Br Y	Y N	+ -	Isotropic Parallel Oblique UNDULOSE	Wavy Kinky Flexible Straight Sickle Ribbon
2				L M H	W Bl G Br Y	Y N	+ -	Isotropic Parallel Oblique UNDULOSE	Wavy Kinky Flexible Straight Sickle Ribbon
3				L M H	W Bl G Br Y	Y N	+ -	Isotropic Parallel Oblique UNDULOSE	Wavy Kinky Flexible Straight Sickle Ribbon
4				L M H	W Bl G Br Y	Y N	+ -	Isotropic Parallel Oblique UNDULOSE	Wavy Kinky Flexible Straight Sickle Ribbon

$\lambda\gamma$		$\lambda\alpha$								**Analyst**
$\lambda\gamma$		$\lambda\alpha$								QC
$\lambda\gamma$		$\lambda\alpha$								

Comments:

AES

ANALYTICAL ENVIRONMENTAL SERVICES, INC.

Bulk Sample Summary Report

NVLAP

Lab Code 102082-0

14-Mar-18

Client Name:	[redacted]	AES Job Number:	[redacted]
Project Name:	[redacted]	Project Number:	[redacted]

Client ID	AES ID	Location	Asbestos Mineral Percentage						Comments
			CH	AM	CR	AN	TR	AC	
FCH1-PI-1 Layer: 1	1803E23-006A	Females Bathroom Chase 1/Black Foam Glass Pipe Insulation	10	ND	ND	ND	ND	ND	Black Mastic (on foam glass)
FCH1-PI-1 Layer: 2	1803E23-006A	Females Bathroom Chase 1/Black Foam Glass Pipe Insulation	ND	ND	ND	ND	ND	ND	Black foam glass
FCH1-PI-1 Layer: 3	1803E23-006A	Females Bathroom Chase 1/Black Foam Glass Pipe Insulation	ND	ND	ND	ND	ND	ND	Black Mastic (on tape)
FCH1-PI-1 Layer: 4	1803E23-006A	Females Bathroom Chase 1/Black Foam Glass Pipe Insulation	ND	ND	ND	ND	ND	ND	Brown tape
FCH1-PI-2 Layer: 1	1803E23-007A	Females Bathroom Chase 1/Black Foam Glass Pipe Insulation	N/A	N/A	N/A	N/A	N/A	N/A	Not analyzed
FCH2-PI-3 Layer: 1	1803E23-008A	Females Bathroom Chase 2/Black Foam Glass Pipe Insulation	N/A	N/A	N/A	N/A	N/A	N/A	Not analyzed

Note: CH=chrysotile, AM=amosite, CR=crocidolite, AC=actinolite, TR=tremolite, AN=anthophylite

For comments on the samples, see the individual analysis sheets.

ND = None Detected

AES,Inc. is accredited by NIST's National Voluntary Laboratory Accreditation Program (NVLAP) for Polarized Light Microscopy (PLM) analysis, Lab Code 102082-0. All analyses performed in accordance with EPA "Interim Method for the Determination of Asbestos in Bulk Insulation Samples" (EPA 600/M4-82-020), 1982 as found in 40 CFR, Part 763, Appendix E to Subpart E and "Method for the Determination of Asbestos in Bulk Building Materials" (EPA/600/R-93/116), 1993.

These test results apply only to those samples actually tested, as submitted by the client. All percentages are reported by visually estimated volume.

PLM is not consistently reliable in detecting small concentrations of asbestos in floor tiles and similar nonfriable materials, quantitative TEM is currently the only method that can be used to determine conclusive asbestos content.

This report must not be reproduced except in full without written approval of Analytical Environmental Services, Inc.

Microanalyst: [redacted]

[redacted]

QC Analyst: [redacted]

[redacted]

ANALYTICAL ENVIRONMENTAL SERVICES, INC.

3080 Presidential Drive
Atlanta,GA 30340
Tel :(770) 457-8177
Fax:(770) 457-8188

AES Job Number: [redacted]

NVLAP
Lab Code 102082-0

14-Mar-18

Bulk Sample Analysis

Client Name:	[redacted]	AES Job Number:	[redacted]
Project Name:	[redacted]	AES Lab ID:	[redacted]
Client Sample ID:	[redacted]	Project Number:	[redacted]
Location:	Mens Bathroom Chase 1/Black Foam Glass Pipe Insulation	Layer:	1
Sample Description:	Black semi-hard bitumenous with fibers		

All percentages given below are visually estimated by volume

ASBESTOS FIBERS	
Chrysotile:	10
Amosite:	ND
Crocidolite:	ND
Anthophyllite:	ND
Tremolite:	ND
Actinolite:	ND

NON-FIBROUS MATERIALS	
Vermiculite:	ND
Biotite:	ND
Mica:	ND
Perlite:	ND
Aggregates:	ND
Styrofoam:	ND

NON-ASBESTOS FIBERS	
Synthetics:	ND
Mineral Wool:	ND
Fiberglass:	ND
Cellulose:	ND
Animal Hair:	ND
Antigonite:	ND

OTHERS	
Aluminum:	ND
Bitumen:	90
Resilient Material:	ND
Glue:	ND
Binders:	ND

Comments: Black Mastic

ND = None Detected

AES,Inc. is accredited by NIST's National Voluntary Laboratory Accreditation Program (NVLAP) for Polarized Light Microscopy (PLM) analysis, Lab Code 102082-0. All analyses performed in accordance with EPA "Interim Method for the Determination of Asbestos in Bulk Insulation Samples" (EPA 600/M4-82-020), 1982 as found in 40 CFR, Part 763, Appendix E to Subpart E and "Method for the Determination of Asbestos in Bulk Building Materials" (EPA/600/R-93/116), 1993.
These test results apply only to those samples actually tested, as submitted by the client. All percentages are reported by visually estimated volume. PLM is not consistently reliable in detecting small concentrations of asbestos in floor tiles and similar nonfriable materials, quantitative TEM is currently the only method that can be used to determine conclusive asbestos content.
This report must not be reproduced except in full without written approval of Analytical Environmental Services, Inc.

Microanalyst: [redacted]
[redacted]

QC Analyst: [redacted]
[redacted]

Exhibit #4
Contamination Control Testing Sheet

			Blanks on Laboratory Equipment			
Analyst	**Date**	**Glass Slides**	**Cover Slips**	**Refractive Index Oils**	**Sampling Instrument**	**Comments**

Exhibit #5

Microscope Alignment Sheet

Operator	Date	Orientation of Polarizer & Analyzer	Orientation of Center Ocular Crosshairs	Centerized Objectives	Centerized Condenser & Diaphram	Refractive Index			Comments
						1.55	1.62	1.68	

Exhibit #6

Blind Recounts Form

Analyst	Date	AES ID #	First Counting	Second Counting	Relative Error %

Exhibit #7
Monthly Precision Summary

PLM QUALITY ASSURANCE PROGRAM
SUMMARY OF MONTHLY PRECISION

Date: ________________________20 _______________

Microanalyst: ______________________________

PLM SAMPLES	AMOUNTS	
1. Total Analysis		
2. Duplicate / Replicate		
3. % QA= 2 / 1		
4. Accepted Analysis		
5. Rejected Analysis	Minor Errors	
	# Misclassify	
	# of false –positives	
	# of false – negatives	
6. Precision 100%-(5/1 * 100)		
7. Overall Precision		

Corrective action for Deficiencies____
____
____

Lab Manager: __________________________ Date:_________________________

QA Officer: __________________________ Date:_________________________

Exhibit #8
NIST HB 150 / Bulk Laboratory (PLM) Audit Checklists

NVLAP LAB CODE: ☐

NIST HANDBOOK 150 CHECKLIST

Instructions to the Assessor: This checklist addresses the general accreditation criteria prescribed in NIST Handbook 150, *NVLAP Procedures and General Requirements* (2006 edition). The checklist items are numbered to correspond to the requirements found in Clauses 4 and 5, and Annexes A and B of the handbook. Items marked with ♦ indicate a change in requirements from the 2001 edition of NIST Handbook 150.

Place an "X" beside each checklist item that represents a nonconformity. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and written nonconformity explanation and/or comment on the comment sheet(s) at the end of the checklist. Write "OK" beside all other items you observed or verified as compliant at the laboratory.

4 Management requirements for accreditation

4.1 Organization

____ **4.1.1** The laboratory or the organization of which it is part shall be an entity that can be held legally responsible.

Legal name of laboratory ownership: ________________________________

____ **4.1.2** It is the responsibility of the laboratory to carry out its testing and calibration activities in such a way as to meet the requirements of this handbook and to satisfy the needs of the customer, the regulatory authorities or organizations providing recognition.

____ **4.1.3** The management system shall cover work carried out in the laboratory's permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

____ **4.1.4** If the laboratory is part of an organization performing activities other than testing and/or calibration, the responsibilities of key personnel in the organization that have an involvement or influence on the testing and/or calibration activities of the laboratory shall be defined in order to identify potential conflicts of interest.

NOTE 1 Where a laboratory is part of a larger organization, the organizational arrangements should be such that departments having conflicting interests, such as production, commercial marketing or financing do not adversely influence the laboratory's compliance with the requirements of this handbook.

NOTE 2 If the laboratory wishes to be recognized as a third-party laboratory, it should be able to demonstrate that it is impartial and that it and its personnel are free from any undue commercial, financial and other pressures which might influence their technical judgment. The third-party testing or calibration laboratory should not engage in any activities that may endanger the trust in its independence of judgment and integrity in relation to its testing or calibration activities.

4.1.5 The laboratory shall:

____ a) have managerial and technical personnel who, irrespective of other responsibilities, have the authority and resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system, and to identify the occurrence of departures from the management system or from the procedures for performing tests and/or calibrations, and to initiate actions to prevent or minimize such departures (see also 5.2);

____ b) have arrangements to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;

____ c) have policies and procedures to ensure the protection of its customers' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results;

____ d) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgement or operational integrity;

____ e) define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services;

____ f) specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the tests and/or calibrations;

____ g) provide adequate supervision of testing and calibration staff, including trainees, by persons familiar with methods and procedures, purpose of each test and/or calibration, and with the assessment of the test or calibration results;

____ h) have technical management which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of laboratory operations;

Name of person: ____

Area of responsibility: ____

Repeat as necessary: ____

____ i) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the management system related to quality is implemented and followed at all times; the quality manager shall have direct access to the highest level of management at which decisions are made on laboratory policy or resources;

Name of person: ____

____ j) appoint deputies for key managerial personnel (see Note).

Name(s): ______________________________

Name(s): ______________________________

Name(s): ______________________________

___♦ k) ensure that its personnel are aware of the relevance and importance of their activities and how they contribute to the achievement of the objectives of the management system.

NOTE Individuals may have more than one function and it may be impractical to appoint deputies for every function.

___♦ **4.1.6** Top management shall ensure that the appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

4.2 Management system

4.2.1

____ a) The laboratory shall establish, implement and maintain a management system appropriate to the scope of its activities.

____ b) The laboratory shall document its policies, systems, programs, procedures and instructions to the extent necessary to assure the quality of the test and/or calibration results.

____ c) The system's documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

____ **4.2.2** The laboratory's management system policies related to quality, including a quality policy statement, shall be defined in a quality manual (however named). The overall objectives shall be established, and shall be reviewed during management review.

Date of most recent quality manual: ______________________________

The quality policy statement shall be issued under the authority of top management. It shall include at least the following:

____ a) the laboratory management's commitment to good professional practice and to the quality of its testing and calibration in servicing its customers;

____ b) the management's statement of the laboratory's standard of service;

____ c) the purpose of the management system related to quality;

____ d) a requirement that all personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work; and

___♦ e) the laboratory management's commitment to comply with this handbook and to continually improve the effectiveness of the management system.

NOTE The quality policy statement should be concise and may include the requirement that tests and/or calibrations shall always be carried out in accordance with stated methods and customers' requirements. When the test and/or calibration laboratory is part of a larger organization, some quality policy elements may be in other documents.

___♦ **4.2.3** Top management shall provide evidence of commitment to the development and implementation of the management system and to continually improve its effectiveness.

___♦ **4.2.4** Top management shall communicate to the organization the importance of meeting customer requirements as well as statutory and regulatory requirements.

4.2.5

____ a) The quality manual shall include or make reference to the supporting procedures including technical procedures.

____ b) It shall outline the structure of the documentation used in the management system.

____ **4.2.6** The roles and responsibilities of technical management and the quality manager, including their responsibility for ensuring compliance with this handbook, shall be defined in the quality manual.

___♦ **4.2.7** Top management shall ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

4.3 Document control

4.3.1 General

____ The laboratory shall establish and maintain procedures to control all documents that form part of its management system (internally generated or from external sources), such as regulations, standards, other normative documents, test and/or calibration methods, as well as drawings, software, specifications, instructions and manuals.

NOTE 1 In this context "document" could be policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

NOTE 2 The control of data related to testing and calibration is covered in 5.4.7. The control of records is covered in 4.13.

4.3.2 Document approval and issue

4.3.2.1

____ a) All documents issued to personnel in the laboratory as part of the management system shall be reviewed and approved for use by authorized personnel prior to issue.

____ b) A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the management system shall be established and be readily available to preclude the use of invalid and/or obsolete documents.

4.3.2.2 The procedure(s) adopted shall ensure that:

____ a) authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;

____ b) documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;

____ c) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use;

____ d) obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

4.3.2.3 Management system documents generated by the laboratory shall be uniquely identified. Such identification shall include:

____ a) the date of issue and/or revision identification,

____ b) page numbering,

____ c) the total number of pages or a mark to signify the end of the document, and

____ d) the issuing authority(ies).

4.3.3 Document changes

____ **4.3.3.1** Changes to documents shall be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval.

____ **4.3.3.2** Where practicable, the altered or new text shall be identified in the document or the appropriate attachments.

4.3.3.3

____ a) If the laboratory's document control system allows for the amendment of documents by hand pending the reissue of the documents, the procedures and authorities for such amendments shall be defined.

____ b) Amendments shall be clearly marked, initialed and dated. A revised document shall be formally reissued as soon as practicable.

____ **4.3.3.4** Procedures shall be established to describe how changes in documents maintained in computerized systems are made and controlled.

4.4 Review of requests, tenders and contracts

____ **4.4.1** The laboratory shall establish and maintain procedures for the review of requests, tenders and contracts. The policies and procedures for these reviews leading to a contract for testing and/or calibration shall ensure that:

____ a) the requirements, including the methods to be used, are adequately defined, documented and understood (see 5.4.2);

____ b) the laboratory has the capability and resources to meet the requirements;

____ c) the appropriate test and/or calibration method is selected and is capable of meeting the customers' requirements (see 5.4.2).

____ d) Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the customer.

NOTE 1 The request, tender and contract review should be conducted in a practical and efficient manner, and the effect of financial, legal and time schedule aspects should be taken into account. For internal customers, reviews of requests, tenders and contracts can be performed in a simplified way.

NOTE 2 The review of capability should establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests and/or calibrations in question. The review may also encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial test or calibration programs using samples or items of known value in order to determine uncertainties of measurement, limits of detection, confidence limits, etc.

NOTE 3 A contract may be any written or oral agreement to provide a customer with testing and/or calibration services.

____ **4.4.2** Records of reviews, including any significant changes, shall be maintained. Records shall also be maintained of pertinent discussions with a customer relating to the customer's requirements or the results of the work during the period of execution of the contract.

NOTE For review of routine and other simple tasks, the date and the identification (e.g., the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate. For repetitive routine tasks, the review need be made only at the initial enquiry stage or on granting of the contract for ongoing routine work performed under a general agreement with the customer, provided that the customer's requirements remain unchanged. For new, complex or advanced testing and/or calibration tasks, a more comprehensive record should be maintained.

____ **4.4.3** The review shall also cover any work that is subcontracted by the laboratory.

____ **4.4.4** The customer shall be informed of any deviation from the contract.

____ **4.4.5** If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel.

4.5 Subcontracting of tests and calibrations

____ **4.5.1** When a laboratory subcontracts work whether because of unforeseen reasons (e.g., workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g., through permanent subcontracting, agency or franchising arrangements), this work shall be placed with a competent subcontractor. A competent subcontractor is one that, for example, complies with this handbook for the work in question.

____ **4.5.2** The laboratory shall advise the customer of the arrangement in writing and, when appropriate, gain the approval of the customer, preferably in writing.

____ **4.5.3** The laboratory is responsible to the customer for the subcontractor's work, except in the case where the customer or a regulatory authority specifies which subcontractor is to be used.

____ **4.5.4** The laboratory shall maintain a register of all subcontractors that it uses for tests and/or calibrations and a record of the evidence of compliance with this handbook for the work in question.

4.6 Purchasing services and supplies

____ **4.6.1** The laboratory shall have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the tests and/or calibrations. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the tests and calibrations.

4.6.2

____ a) The laboratory shall ensure that purchased supplies and reagents and consumable materials that affect the quality of tests and/or calibrations are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the tests and/or calibrations concerned. These services and supplies used shall comply with specified requirements.

____ b) Records of actions taken to check compliance shall be maintained.

____ **4.6.3** Purchasing documents for items affecting the quality of laboratory output shall contain data describing the services and supplies ordered. These purchasing documents shall be reviewed and approved for technical content prior to release.

NOTE The description may include type, class, grade, precise identification, specifications, drawings, inspection instructions, other technical data including approval of test results, the quality required and the management system standard under which they were made.

4.6.4

____ a) The laboratory shall evaluate suppliers of critical consumables, supplies and services which affect the quality of testing and calibration, and

____ b) shall maintain records of these evaluations and list those approved.

4.7 Service to the customer

____ **4.7.1** The laboratory shall be willing to cooperate with customers or their representatives in clarifying the customer's request and in monitoring the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other customers.

NOTE 1 Such cooperation may include:

a) providing the customer or the customer's representative reasonable access to relevant areas of the laboratory for the witnessing of tests and/or calibrations performed for the customer;

b) preparation, packaging, and dispatch of test and/or calibration items needed by the customer for verification purposes.

NOTE 2 Customers value the maintenance of good communication, advice and guidance in technical matters, and opinions and interpretations based on results. Communication with the customer, especially in large assignments, should be maintained throughout the work. The laboratory should inform the customer of any delays or major deviations in the performance of the tests and/or calibrations.

___♦ **4.7.2** The laboratory shall seek feedback, both positive and negative, from its customers. The feedback shall be used and analyzed to improve the management system, testing and calibration activities and customer service.

NOTE Examples of the types of feedback include customer satisfaction surveys and review of test or calibration reports with customers.

4.8 Complaints

____ **4.8.1** The laboratory shall have a policy and procedure for the resolution of complaints received from customers or other parties.

____ **4.8.2** Records shall be maintained of all complaints and of the investigations and corrective actions taken by the laboratory (see also 4.11).

4.9 Control of nonconforming testing and/or calibration work

____ **4.9.1** The laboratory shall have a policy and procedures that shall be implemented when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer. The policy and procedures shall ensure that:

____ a) the responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports and calibration certificates, as necessary) are defined and taken when nonconforming work is identified;

____ b) an evaluation of the significance of the nonconforming work is made;

____ c) correction is taken immediately, together with any decision about the acceptability of the nonconforming work;

____ d) where necessary, the customer is notified and work is recalled;

____ e) the responsibility for authorizing the resumption of work is defined.

NOTE Identification of nonconforming work or problems with the management system or with testing and/or calibration activities can occur at various places within the management system and technical operations. Examples are customer complaints, quality control, instrument calibration, checking of consumable materials, staff observations or supervision, test report and calibration certificate checking, management reviews and internal or external audits.

____ **4.9.2** Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 4.11 shall be promptly followed.

4.10 Improvement

___♦ The laboratory shall continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

4.11 Corrective action

4.11.1 General

____ The laboratory shall establish a policy and a procedure and shall designate appropriate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the management system or technical operations have been identified.

NOTE A problem with the management system or with the technical operations of the laboratory may be identified through a variety of activities, such as control of nonconforming work, internal or external audits, management reviews, feedback from customers and from staff observations.

4.11.2 Cause analysis

____ The procedure for corrective action shall start with an investigation to determine the root cause(s) of the problem.

NOTE Cause analysis is the key and sometimes the most difficult part in the corrective action procedure. Often the root cause is not obvious and thus a careful analysis of all potential causes of the problem is required. Potential causes could include customer requirements, the samples, sample specifications, methods and procedures, staff skills and training, consumables, or equipment and its calibration.

4.11.3 Selection and implementation of corrective actions

____ a) Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.

____ b) Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem.

____ c) The laboratory shall document and implement any required changes resulting from corrective action investigations.

4.11.4 Monitoring of corrective actions

____ The laboratory shall monitor the results to ensure that the corrective actions taken have been effective.

4.11.5 Additional audits

____ Where the identification of nonconformities or departures casts doubts on the laboratory’s compliance with its own policies and procedures, or on its compliance with this handbook, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with 4.14 as soon as possible.

NOTE Such additional audits often follow the implementation of the corrective actions to confirm their effectiveness. An additional audit should be necessary only when a serious issue or risk to the business is identified.

4.12 Preventive action

4.12.1

____ a) Needed improvements and potential sources of nonconformities, either technical or concerning the management system, shall be identified.

___♦ b) When improvement opportunities are identified or if preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformities and to take advantage of the opportunities for improvement.

____ **4.12.2** Procedures for preventive actions shall include the initiation of such actions and application of controls to ensure that they are effective.

NOTE 1 Preventive action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

NOTE 2 Apart from the review of the operational procedures, the preventive action might involve analysis of data, including trend and risk analyses and proficiency-testing results.

4.13 Control of records

4.13.1 General

____ **4.13.1.1** The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventive actions.

4.13.1.2

____ a) All records shall be legible and shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss.

____ b) Retention times of records shall be established.

NOTE Records may be in any media, such as hard copy or electronic media.

____ **4.13.1.3** All records shall be held secure and in confidence.

____ **4.13.1.4** The laboratory shall have procedures to protect and back up records stored electronically and to prevent unauthorized access to or amendment of these records.

4.13.2 Technical records

4.13.2.1

____ a) The laboratory shall retain records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report or calibration certificate issued, for a defined period.

____ b) The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original.

____ c) The records shall include the identity of personnel responsible for the sampling, performance of each test and/or calibration and checking of results.

NOTE 1 In certain fields it may be impossible or impracticable to retain records of all original observations.

NOTE 2 Technical records are accumulations of data (see 5.4.7) and information which result from carrying out tests and/or calibrations and which indicate whether specified quality or process parameters are achieved. They may include forms, contracts, work sheets, work books, check sheets, work notes, control graphs, external and internal test reports and calibration certificates, customers' notes, papers and feedback.

____ **4.13.2.2** Observations, data and calculations shall be recorded at the time they are made and shall be identifiable to the specific task.

4.13.2.3

____ a) When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialed by the person making the correction.

____ b) In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

4.14 Internal audits

4.14.1

____ a) The laboratory shall periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the management system and this handbook. The internal audit program shall address all elements of the management system, including the testing and/or calibration activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management.

Dates of most recent internal audit: ______________________________

Note to assessor: Attach a copy of the full internal audit schedule.

____ b) Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

NOTE The cycle for internal auditing should normally be completed in one year.

____ **4.14.2** When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test or calibration results, the laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that the laboratory results may have been affected.

____ **4.14.3** The area of activity audited, the audit findings and corrective actions that arise from them shall be recorded.

____ **4.14.4** Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

4.15 Management reviews

____ **4.15.1** In accordance with a predetermined schedule and procedure, the laboratory's top management shall periodically conduct a review of the laboratory's management system and testing and/or calibration activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements.

Date(s) of most recent management review: ________________________

What is the review schedule? ____________________________________
____

The review shall take account of:

____ a) the suitability of policies and procedures;

____ b) reports from managerial and supervisory personnel;

____ c) the outcome of recent internal audits;

____ d) corrective and preventive actions;

____ e) assessments by external bodies;

____ f) the results of interlaboratory comparisons or proficiency tests;

____ g) changes in the volume and type of the work;

____ h) customer feedback;

____ i) complaints;

___♦ j) recommendations for improvement;

____ k) other relevant factors, such as quality control activities, resources and staff training.

NOTE 1 A typical period for conducting a management review is once every 12 months.

NOTE 2 Results should feed into the laboratory planning system and should include the goals, objectives and action plans for the coming year.

NOTE 3 A management review includes consideration of related subjects at regular management meetings.

4.15.2

____ a) Findings from management reviews and the actions that arise from them shall be recorded.

____ b) The management shall ensure that those actions are carried out within an appropriate and agreed timescale.

5 Technical requirements for accreditation

5.1 General

5.1.1 Many factors determine the correctness and reliability of the tests and/or calibrations performed by a laboratory. These factors include contributions from:

i) human factors (5.2);

ii) accommodation and environmental conditions (5.3);

iii) test and calibration methods and method validation (5.4);

iv) equipment (5.5);

v) measurement traceability (5.6 and Annex B);

vi) sampling (5.7);

vii) the handling of test and calibration items (5.8).

____ **5.1.2** The extent to which the factors contribute to the total uncertainty of measurement differs considerably between (types of) tests and between (types of) calibrations. The laboratory shall take account of these factors in developing test and calibration methods and procedures, in the training and qualification of personnel, and in the selection and calibration of the equipment it uses.

5.2 Personnel

5.2.1

____ a) The laboratory management shall ensure the competence of all who operate specific equipment, perform tests and/or calibrations, evaluate results, and sign test reports and calibration certificates.

____ b) When using staff who are undergoing training, appropriate supervision shall be provided. Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

NOTE 1 In some technical areas (e.g., nondestructive testing) it may be required that the personnel performing certain tasks hold personnel certification. The laboratory is responsible for fulfilling specified personnel certification requirements. The requirements for personnel certification might be regulatory, included in the standards for the specific technical field, or required by the customer.

NOTE 2 The personnel responsible for the opinions and interpretation included in test reports should, in addition to the appropriate qualifications, training, experience and satisfactory knowledge of the testing carried out, also have:

i) relevant knowledge of the technology used for the manufacturing of the items, materials, products, etc. tested, or the way they are used or intended to be used, and of the defects or degradations which may occur during or in service;

ii) knowledge of the general requirements expressed in the legislation and standards; and

iii) an understanding of the significance of deviations found with regard to the normal use of the items, materials, products, etc. concerned.

5.2.2

____ a) The management of the laboratory shall formulate the goals with respect to the education, training and skills of the laboratory personnel.

____ b) The laboratory shall have a policy and procedures for identifying training needs and providing training of personnel.

____ c) The training program shall be relevant to the present and anticipated tasks of the laboratory.

___♦ d) The effectiveness of the training actions taken shall be evaluated.

5.2.3

____ a) The laboratory shall use personnel who are employed by, or under contract to, the laboratory.

____ b) Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's management system.

____ **5.2.4** The laboratory shall maintain current job descriptions for managerial, technical and key support personnel involved in tests and/or calibrations.

NOTE Job descriptions can be defined in many ways. As a minimum, the following should be defined:

i) the responsibilities with respect to performing tests and/or calibrations;

ii) the responsibilities with respect to the planning of tests and/or calibrations and evaluation of results;

iii) the responsibilities for reporting opinions and interpretations;

iv) the responsibilities with respect to method modification and development and validation of new methods;

v) expertise and experience required;

vi) qualifications and training programs;

vii) managerial duties.

5.2.5

____ a) The management shall authorize specific personnel to perform particular types of sampling, test and/or calibration, to issue test reports and calibration certificates, to give opinions and interpretations and to operate particular types of equipment.

____ b) The laboratory shall maintain records of the relevant authorization(s), competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel.

____ c) This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

NVLAP Note: This requirement also applies to Approved Signatories (see 1.5.2).

5.3 Accommodation and environmental conditions

5.3.1

____ a) Laboratory facilities for testing and/or calibration, including but not limited to energy sources, lighting and environmental conditions, shall be such as to facilitate correct performance of the tests and/or calibrations.

The laboratory shall ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. Particular care shall be taken when sampling and tests and/or calibrations are undertaken at sites other than a permanent laboratory facility.

____ b) The technical requirements for accommodation and environmental conditions that can affect the results of tests and calibrations shall be documented.

5.3.2

____ a) The laboratory shall monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention shall be paid, for example, to biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, and sound and vibration levels, as appropriate to the technical activities concerned.

____ b) Tests and calibrations shall be stopped when the environmental conditions jeopardize the results of the tests and/or calibrations.

____ **5.3.3** There shall be effective separation between neighboring areas in which there are incompatible activities. Measures shall be taken to prevent cross-contamination.

____ **5.3.4** Access to and use of areas affecting the quality of the tests and/or calibrations shall be controlled. The laboratory shall determine the extent of control based on its particular circumstances.

____ **5.3.5** Measures shall be taken to ensure good housekeeping in the laboratory. Special procedures shall be prepared where necessary.

5.4 Test and calibration methods and method validation

5.4.1 General

____ a) The laboratory shall use appropriate methods and procedures for all tests and/or calibrations within its scope. These include sampling, handling, transport, storage and preparation of items to be tested and/or calibrated, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of test and/or calibration data.

____ b) The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of items for testing and/or calibration, or both, where the absence of such instructions could jeopardize the results of tests and/or calibrations.

____ c) All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be kept up to date and shall be made readily available to personnel (see 4.3).

____ d) Deviation from test and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the customer.

NOTE International, regional or national standards or other recognized specifications that contain sufficient and concise information on how to perform the tests and/or calibrations do not need to be supplemented or rewritten as internal procedures if these standards are written in a way that they can be used as published by the operating staff in a laboratory. It may be necessary to provide additional documentation for optional steps in the method or additional details.

5.4.2 Selection of methods

____ a) The laboratory shall use test and/or calibration methods, including methods for sampling, which meet the needs of the customer and which are appropriate for the tests and/or calibrations it undertakes. Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so.

____ b) When necessary, the standard shall be supplemented with additional details to ensure consistent application.

____ c) When the customer does not specify the method to be used, the laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated.

____ d) The customer shall be informed as to the method chosen.

____ e) The laboratory shall confirm that it can properly operate standard methods before introducing the tests or calibrations. If the standard method changes, the confirmation shall be repeated.

____ f) The laboratory shall inform the customer when the method proposed by the customer is considered to be inappropriate or out of date.

5.4.3 Laboratory-developed methods

____ a) The introduction of test and calibration methods developed by the laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources.

____ b) Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured.

5.4.4 Non-standard methods

____ a) When it is necessary to use methods not covered by standard methods, these shall be subject to agreement with the customer and shall include a clear specification of the customer's requirements and the purpose of the test and/or calibration.

____ b) The method developed shall have been validated appropriately before use.

NOTE For new test and/or calibration methods, procedures should be developed prior to the tests and/or calibrations being performed and should contain at least the following information:

a) appropriate identification;

b) scope;

c) description of the type of item to be tested or calibrated;

d) parameters or quantities and ranges to be determined;

e) apparatus and equipment, including technical performance requirements;

f) reference standards and reference materials required;

g) environmental conditions required and any stabilization period needed;

h) description of the procedure, including:

  i) affixing of identification marks, handling, transporting, storing and preparation of items,

  ii) checks to be made before the work is started,

  iii) checks that the equipment is working properly and, where required, calibration and adjustment of the equipment before each use,

  iv) the method of recording the observations and results,

  v) any safety measures to be observed;

i) criteria and/or requirements for approval/rejection;

j) data to be recorded and method of analysis and presentation;

k) the uncertainty or the procedure for estimating uncertainty.

5.4.5 Validation of methods

5.4.5.1 Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

5.4.5.2

____ a) The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application.

____ b) The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

NOTE 1 Validation may include procedures for sampling, handling and transportation.

NOTE 2 The techniques used for the determination of the performance of a method should be one of, or a combination of, the following:

i) calibration using reference standards or reference materials;

ii) comparison of results achieved with other methods;

iii) interlaboratory comparisons;

iv) systematic assessment of the factors influencing the result;

v) assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.

NOTE 3 When some changes are made in the validated non-standard methods, the influence of such changes should be documented and, if appropriate, a new validation should be carried out.

____ **5.4.5.3** The range and accuracy of the values obtainable from validated methods (e.g., the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the customers' needs.

NOTE 1 Validation includes specification of the requirements, determination of the characteristics of the methods, a check that the requirements can be fulfilled by using the method, and a statement on the validity.

NOTE 2 As method-development proceeds, regular review should be carried out to verify that the needs of the customer are still being fulfilled. Any change in requirements requiring modifications to the development plan should be approved and authorized.

NOTE 3 Validation is always a balance between costs, risks and technical possibilities. There are many cases in which the range and uncertainty of the values (e.g., accuracy, detection limit, selectivity, linearity, repeatability, reproducibility, robustness and cross-sensitivity) can only be given in a simplified way due to lack of information.

5.4.6 Estimation of uncertainty of measurement

____ **5.4.6.1** A calibration laboratory, or a testing laboratory performing its own calibrations, shall have and shall apply a procedure to estimate the uncertainty of measurement for all calibrations and types of calibrations.

____ **5.4.6.2** Testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

NOTE 1 The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

i) the requirements of the test method;

ii) the requirements of the customer;

iii) the existence of narrow limits on which decisions on conformity to a specification are based.

NOTE 2 In those cases where a well recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 5.10).

____ **5.4.6.3** When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

NOTE 1 Sources contributing to the uncertainty include, but are not necessarily limited to, the reference standards and reference materials used, methods and equipment used, environmental conditions, properties and condition of the item being tested or calibrated, and the operator.

NOTE 2 The predicted long-term behavior of the tested and/or calibrated item is not normally taken into account when estimating the measurement uncertainty.

NOTE 3 For further information, see ISO 5725 and the Guide to the Expression of Uncertainty in Measurement (see 1.4).

NVLAP Note: ANSI/NCSL Z540-2-1997 and NIST Technical Note 1297, 1994 edition, are considered to be equivalent to the Guide to the Expression of Uncertainty in Measurement (GUM).

5.4.7 Control of data

____ **5.4.7.1** Calculations and data transfers shall be subject to appropriate checks in a systematic manner.

5.4.7.2 When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of test or calibration data, the laboratory shall ensure that:

____ a) computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;

____ b) procedures are established and implemented for protecting the data; such procedures shall include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;

____ c) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test and calibration data.

NOTE Commercial off-the-shelf software (e.g., word processing, database and statistical programs) in general use within their designed application range may be considered to be sufficiently validated. However, laboratory software configuration/modifications should be validated as in 5.4.7.2 a).

5.5 Equipment

5.5.1

____ a) The laboratory shall be furnished with all items of sampling, measurement and test equipment required for the correct performance of the tests and/or calibrations (including sampling, preparation of test and/or calibration items, processing and analysis of test and/or calibration data).

____ b) In those cases where the laboratory needs to use equipment outside its permanent control, it shall ensure that the requirements of this handbook are met.

5.5.2

____ a) Equipment and its software used for testing, calibration and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the tests and/or calibrations concerned.

____ b) Calibration programs shall be established for key quantities or values of the instruments where these properties have a significant effect on the results.

____ c) Before being placed into service, equipment (including that used for sampling) shall be calibrated or checked to establish that it meets the laboratory’s specification requirements and complies with the relevant standard specifications. It shall be checked and/or calibrated before use (see 5.6).

____ **5.5.3** Equipment shall be operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) shall be readily available for use by the appropriate laboratory personnel.

____ **5.5.4** Each item of equipment and its software used for testing and calibration and significant to the result shall, when practicable, be uniquely identified.

____ **5.5.5** Records shall be maintained of each item of equipment and its software significant to the tests and/or calibrations performed. The records shall include at least the following:

____ a) the identity of the item of equipment and its software;

____ b) the manufacturer's name, type identification, and serial number or other unique identification;

____ c) checks that equipment complies with the specification (see 5.5.2);

____ d) the current location, where appropriate;

____ e) the manufacturer's instructions, if available, or reference to their location;

____ f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;

____ g) the maintenance plan, where appropriate, and maintenance carried out to date;

____ h) any damage, malfunction, modification or repair to the equipment.

____ **5.5.6** The laboratory shall have procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment to ensure proper functioning and in order to prevent contamination or deterioration.

NOTE Additional procedures may be necessary when measuring equipment is used outside the permanent laboratory for tests, calibrations or sampling.

5.5.7

____ a) Equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labeled or marked as being out of service until it has been repaired and shown by calibration or test to perform correctly.

____ b) The laboratory shall examine the effect of the defect or departure from specified limits on previous tests and/or calibrations and shall institute the "Control of nonconforming work" procedure (see 4.9).

____ **5.5.8** Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labeled, coded or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

____ **5.5.9** When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

____ **5.5.10** When intermediate checks are needed to maintain confidence in the calibration status of the equipment, these checks shall be carried out according to a defined procedure.

____ **5.5.11** Where calibrations give rise to a set of correction factors, the laboratory shall have procedures to ensure that copies (e.g., in computer software) are correctly updated.

____ **5.5.12** Test and calibration equipment, including both hardware and software, shall be safeguarded from adjustments which would invalidate the test and/or calibration results.

5.6 Measurement traceability

5.6.1 General

____ a) All equipment used for tests and/or calibrations, including equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of the test, calibration or sampling shall be calibrated before being put into service.

____ b) The laboratory shall have an established program and procedure for the calibration of its equipment.

NOTE Such a program should include a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform tests and calibrations.

NVLAP Note: See Annex B for requirements for the implementation of traceability policy in NVLAP-accredited laboratories.

5.6.2 Specific requirements

5.6.2.1 Calibration

5.6.2.1.1

____ a) For calibration laboratories, the program for calibration of equipment shall be designed and operated so as to ensure that calibrations and measurements made by the laboratory are traceable to the International System of Units (SI) (*Système international d'unités*).

A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute.

____ b) When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability.

____ c) The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification (see also 5.10.4.2).

NOTE 1 Calibration laboratories fulfilling the requirements of this handbook are considered to be competent. A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to this handbook, for the calibration concerned, is sufficient evidence of traceability of the calibration data reported.

NOTE 2 Traceability to SI units of measurement may be achieved by reference to an appropriate primary standard (see VIM:1993, 6.4) or by reference to a natural constant, the value of which in terms of the relevant SI unit is known and recommended by the General Conference of Weights and Measures (CGPM) and the International Committee for Weights and Measures (CIPM).

NOTE 3 Calibration laboratories that maintain their own primary standard or representation of SI units based on fundamental physical constants can claim traceability to the SI system only after these standards have been compared, directly or indirectly, with other similar standards of a national metrology institute.

NOTE 4 The term “identified metrological specification” means that it must be clear from the calibration certificate which specification the measurements have been compared with, by including the specification or by giving an unambiguous reference to the specification.

NOTE 5 When the terms “international standard” or “national standard” are used in connection with traceability, it is assumed that these standards fulfill the properties of primary standards for the realization of SI units.

NOTE 6 Traceability to national measurement standards does not necessarily require the use of the national metrology institute of the country in which the laboratory is located.

NOTE 7 If a calibration laboratory wishes or needs to obtain traceability from a national metrology institute other than in its own country, this laboratory should select a national metrology institute that actively participates in the activities of BIPM either directly or through regional groups.

NOTE 8 The unbroken chain of calibrations or comparisons may be achieved in several steps carried out by different laboratories that can demonstrate traceability.

____ **5.6.2.1.2** There are certain calibrations that currently cannot be strictly made in SI units. In these cases calibration shall provide confidence in measurements by establishing traceability to appropriate measurement standards such as:

____ a) the use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material;

____ b) the use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned.

____ c) Participation in a suitable program of interlaboratory comparisons is required where possible.

5.6.2.2 Testing

____ **5.6.2.2.1** For testing laboratories, the requirements given in 5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

NOTE The extent to which the requirements in 5.6.2.1 should be followed depends on the relative contribution of the calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements should be strictly followed.

____ **5.6.2.2.2** Where traceability of measurements to SI units is not possible and/or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required as for calibration laboratories (see 5.6.2.1.2).

5.6.3 Reference standards and reference materials

5.6.3.1 Reference standards

____ a) The laboratory shall have a program and procedure for the calibration of its reference standards.

____ b) Reference standards shall be calibrated by a body that can provide traceability as described in 5.6.2.1.

____ c) Such reference standards of measurement held by the laboratory shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards shall be calibrated before and after any adjustment.

5.6.3.2 Reference materials

____ Reference materials shall, where possible, be traceable to SI units of measurement, or to certified reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

5.6.3.3 Intermediate checks

____ Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials shall be carried out according to defined procedures and schedules.

5.6.3.4 Transport and storage

____ The laboratory shall have procedures for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

NOTE Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations or sampling.

5.7 Sampling

5.7.1

____ a) The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent testing or calibration.

____ b) The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the test and calibration results.

NOTE 1 Sampling is a defined procedure whereby a part of a substance, material or product is taken to provide for testing or calibration of a representative sample of the whole. Sampling may also be required by the appropriate specification for which the substance, material or product is to be tested or calibrated. In certain cases (e.g., forensic analysis), the sample may not be representative but is determined by availability.

NOTE 2 Sampling procedures should describe the selection, sampling plan, withdrawal and preparation of a sample or samples from a substance, material or product to yield the required information.

____ **5.7.2** Where the customer requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with the appropriate sampling data and shall be included in all documents containing test and/or calibration results, and shall be communicated to the appropriate personnel.

____ **5.7.3** The laboratory shall have procedures for recording relevant data and operations relating to sampling that forms part of the testing or calibration that is undertaken. These records shall include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

5.8 Handling of test and calibration items

____ **5.8.1** The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of test and/or calibration items, including all provisions necessary to protect the integrity of the test or calibration item, and to protect the interests of the laboratory and the customer.

5.8.2

____ a) The laboratory shall have a system for identifying test and/or calibration items.

____ b) The identification shall be retained throughout the life of the item in the laboratory.

____ c) The system shall be designed and operated so as to ensure that items cannot be confused physically or when referred to in records or other documents.

____ d) The system shall, if appropriate, accommodate a sub-division of groups of items and the transfer of items within and from the laboratory.

5.8.3

____ a) Upon receipt of the test or calibration item, abnormalities or departures from normal or specified conditions, as described in the test or calibration method, shall be recorded.

____ b) When there is doubt as to the suitability of an item for test or calibration, or when an item does not conform to the description provided, or the test or calibration required is not specified in sufficient detail, the laboratory shall consult the customer for further instructions before proceeding and shall record the discussion.

5.8.4

____ a) The laboratory shall have procedures and appropriate facilities for avoiding deterioration, loss or damage to the test or calibration item during storage, handling and preparation.

____ b) Handling instructions provided with the item shall be followed.

____ c) When items have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded.

____ d) Where a test or calibration item or a portion of an item is to be held secure, the laboratory shall have arrangements for storage and security that protect the condition and integrity of the secured items or portions concerned.

NOTE 1 Where test items are to be returned into service after testing, special care is required to ensure that they are not damaged or injured during the handling, testing or storing/waiting processes.

NOTE 2 A sampling procedure and information on storage and transport of samples, including information on sampling factors influencing the test or calibration result, should be provided to those responsible for taking and transporting the samples.

NOTE 3 Reasons for keeping a test or calibration item secure can be for reasons of record, safety or value, or to enable complementary tests and/or calibrations to be performed later.

5.9 Assuring the quality of test and calibration results

5.9.1

____ a) The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken.

____ b) The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results.

____ c) This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

  ____ 1) regular use of certified reference materials and/or internal quality control using secondary reference materials;

  ____ 2) participation in interlaboratory comparison or proficiency-testing programs;

  ____ 3) replicate tests or calibrations using the same or different methods;

  ____ 4) retesting or recalibration of retained items;

  ____ 5) correlation of results for different characteristics of an item.

NOTE The selected methods should be appropriate for the type and volume of the work undertaken.

___♦ **5.9.2** Quality control data shall be analyzed and, where they are found to be outside pre-defined criteria, planned action shall be taken to correct the problem and to prevent incorrect results from being reported.

5.10 Reporting the results

5.10.1 General

____ a) The results of each test, calibration, or series of tests or calibrations carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test or calibration methods.

____ b) The results shall be reported, usually in a test report or a calibration certificate (see Note 1), and shall include all the information requested by the customer and necessary for the interpretation of the test or calibration results and all information required by the method used. This information is normally that required by 5.10.2, and 5.10.3 or 5.10.4.

____ c) In the case of tests or calibrations performed for internal customers, or in the case of a written agreement with the customer, the results may be reported in a simplified way. Any information listed in 5.10.2 to 5.10.4 which is not reported to the customer shall be readily available in the laboratory which carried out the tests and/or calibrations.

NOTE 1 Test reports and calibration certificates are sometimes called test certificates and calibration reports, respectively.

NOTE 2 The test reports or calibration certificates may be issued as hard copy or by electronic data transfer provided that the requirements of this handbook are met.

5.10.2 Test reports and calibration certificates

Each test report or calibration certificate shall include at least the following information, unless the laboratory has valid reasons for not doing so:

____ a) a title (e.g., “Test Report” or “Calibration Certificate”);

____ b) the name and address of the laboratory, and the location where the tests and/or calibrations were carried out, if different from the address of the laboratory;

____ c) unique identification of the test report or calibration certificate (such as the serial number), and on each page an identification in order to ensure that the page is recognized as a part of the test report or calibration certificate, and a clear identification of the end of the test report or calibration certificate;

____ d) the name and address of the customer;

____ e) identification of the method used;

____ f) a description of, the condition of, and unambiguous identification of the item(s) tested or calibrated;

____ g) the date of receipt of the test or calibration item(s) where this is critical to the validity and application of the results, and the date(s) of performance of the test or calibration;

____ h) reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;

____ i) the test or calibration results with, where appropriate, the units of measurement;

____ j) the name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the test report or calibration certificate;

____ k) where relevant, a statement to the effect that the results relate only to the items tested or calibrated.

NVLAP Note: NVLAP defines the person(s) who authorizes the test report or calibration certificate as the Approved Signatory (see 1.5.2).

NOTE 1 Hard copies of test reports and calibration certificates should also include the page number and total number of pages.

NOTE 2 It is recommended that laboratories include a statement specifying that the test report or calibration certificate shall not be reproduced except in full, without written approval of the laboratory.

5.10.3 Test reports

5.10.3.1 In addition to the requirements listed in 5.10.2, test reports shall, where necessary for the interpretation of the test results, include the following:

____ a) deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;

____ b) where relevant, a statement of compliance/non-compliance with requirements and/or specifications;

____ c) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

____ d) where appropriate and needed, opinions and interpretations (see 5.10.5);

____ e) additional information which may be required by specific methods, customers or groups of customers.

5.10.3.2 In addition to the requirements listed in 5.10.2 and 5.10.3.1, test reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

____ a) the date of sampling;

____ b) unambiguous identification of the substance, material or product sampled (including the name of the manufacturer, the model or type of designation and serial numbers as appropriate);

____ c) the location of sampling, including any diagrams, sketches or photographs;

____ d) a reference to the sampling plan and procedures used;

____ e) details of any environmental conditions during sampling that may affect the interpretation of the test results;

____ f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

5.10.4 Calibration certificates

5.10.4.1 In addition to the requirements listed in 5.10.2, calibration certificates shall include the following, where necessary for the interpretation of calibration results:

____ a) the conditions (e.g., environmental) under which the calibrations were made that have an influence on the measurement results;

____ b) the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof;

____ c) evidence that the measurements are traceable (see Note 2 in 5.6.2.1.1).

5.10.4.2

____ a) The calibration certificate shall relate only to quantities and the results of functional tests.

____ b) If a statement of compliance with a specification is made, this shall identify which clauses of the specification are met or not met.

____ c) When a statement of compliance with a specification is made omitting the measurement results and associated uncertainties, the laboratory shall record those results and maintain them for possible future reference.

____ d) When statements of compliance are made, the uncertainty of measurement shall be taken into account.

____ **5.10.4.3** When an instrument for calibration has been adjusted or repaired, the calibration results before and after adjustment or repair, if available, shall be reported.

____ **5.10.4.4** A calibration certificate (or calibration label) shall not contain any recommendation on the calibration interval except where this has been agreed with the customer. This requirement may be superseded by legal regulations.

5.10.5 Opinions and interpretations

____ When opinions and interpretations are included, the laboratory shall document the basis upon which the opinions and interpretations have been made. Opinions and interpretations shall be clearly marked as such in a test report.

NOTE 1 Opinions and interpretations should not be confused with inspections and product certifications as intended in ISO/IEC 17020 and ISO/IEC Guide 65.

NOTE 2 Opinions and interpretations included in a test report may comprise, but not be limited to, the following:

i) an opinion on the statement of compliance/noncompliance of the results with requirements;

ii) fulfillment of contractual requirements;

iii) recommendations on how to use the results;

iv) guidance to be used for improvements.

NOTE 3 In many cases it might be appropriate to communicate the opinions and interpretations by direct dialogue with the customer. Such dialogue should be written down.

5.10.6 Testing and calibration results obtained from subcontractors

____ a) When the test report contains results of tests performed by subcontractors, these results shall be clearly identified.

____ b) The subcontractor shall report the results in writing or electronically.

____ c) When a calibration has been subcontracted, the laboratory performing the work shall issue the calibration certificate to the contracting laboratory.

5.10.7 Electronic transmission of results

____ In the case of transmission of test or calibration results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of this handbook shall be met (see also 5.4.7).

5.10.8 Format of reports and certificates

____ The format shall be designed to accommodate each type of test or calibration carried out and to minimize the possibility of misunderstanding or misuse.

NOTE 1 Attention should be given to the layout of the test report or calibration certificate, especially with regard to the presentation of the test or calibration data and ease of assimilation by the reader.

NOTE 2 The headings should be standardized as far as possible.

5.10.9 Amendments to test reports and calibration certificates

____ a) Material amendments to a test report or calibration certificate after issue shall be made only in the form of a further document, or data transfer, which includes the statement:

“Supplement to Test Report [or Calibration Certificate], serial number . . . [or as otherwise identified],” or an equivalent form of wording.

____ b) Such amendments shall meet all the requirements of this handbook.

____ c) When it is necessary to issue a complete new test report or calibration certificate, this shall be uniquely identified and shall contain a reference to the original that it replaces.

Annex A (normative)

Referencing NVLAP accreditation

A.1 Conditions for referencing the NVLAP term, logo, and symbol

The term *NVLAP* and the NVLAP logo are registered marks of the Federal Government, which retains exclusive rights to control the use thereof. Permission to use the term and symbol (NVLAP logo with approved caption) is granted to NVLAP-accredited laboratories for the limited purpose of announcing their accredited status, and for use on reports that describe only testing or calibration within the scope of accreditation. NVLAP reserves the right to control the quality of the use of the NVLAP term, logo, and symbol.

In order to become and remain accredited, laboratories shall comply with the following conditions pertaining to the use of the term *NVLAP*, the NVLAP logo, and NVLAP symbol. Failure to comply with these conditions may result in suspension or revocation of a laboratory's accreditation.

____ **a)** An applicant laboratory that has not yet achieved accreditation may make reference to its applicant status. If the NVLAP Lab Code is used, it shall be accompanied by a statement accurately reflecting the laboratory's status. An applicant laboratory shall not use the NVLAP term, logo or symbol in a manner that implies accreditation.

____ **b)** The laboratory shall have a policy and procedure for controlling the use of the term *NVLAP* and the NVLAP symbol.

____ **c)** The term and/or symbol shall not be used in a manner that brings NVLAP into disrepute or misrepresents a laboratory's scope of accreditation or accredited status.

____ **d)** When the term *NVLAP* is used to reference a laboratory's accredited status, it shall be accompanied by the NVLAP Lab Code.

____ **e)** When the NVLAP symbol used to reference a laboratory's accredited status, it shall be comprised of the NVLAP logo and the NVLAP Lab Code in an approved caption. The caption shall appear below and in close proximity to the logo. The following captions have been approved by NVLAP:

- "For the scope of accreditation under NVLAP Lab Code 000000-0"
- "NVLAP Lab Code 000000-0".

See Annex A of NIST Handbook 150 for examples of the logo with captions.

f) When the NVLAP symbol is used, the form of the NVLAP logo must conform to the following guidelines:

____ 1) The logo shall stand by itself and shall not be combined with any other logo, symbol, or graphic.

____ 2) The aspect ratio (width to height) shall be 2.25 to 1.

____ 3) The logo and caption shall be of a size that allows the caption to be easily read. The size of the caption shall not exceed the size of the logo itself.

____ 4) The logo shall appear in black, blue, or other color approved by NVLAP, and may be filled or unfilled. In the case of a filled logo, the same color shall be used for the outline and the fill.

____ **g)** The name of at least one Approved Signatory shall appear on a test or calibration report that displays the NVLAP symbol or references NVLAP accreditation. A computer-generated report may have the Approved Signatory's name printed along with the test or calibration results, as long as there is evidence that there is a system in place to ensure that the report cannot be generated without the review and consent of the Approved Signatory. There may be legal or contractual requirements for original signatures to appear on the report.

h)

____ 1) When the term and/or symbol are used on test or calibration reports, such use shall be limited to reports in which some or all of the data are from tests or calibrations performed by the laboratory under its scope of accreditation.

____ 2) A test or calibration report that contains both data covered by the accreditation and data not covered by the accreditation shall clearly identify the data that are not covered by the accreditation.

____ 3) The report must prominently display the following statement at the beginning of the report: "This report contains data that are not covered by the NVLAP accreditation."

i)

____ 1) When the term and/or symbol are used on test or calibration reports that also include work done by subcontracted laboratories, such use shall be limited to reports in which some or all of the data are from tests or calibrations performed by the laboratory under its scope of accreditation.

____ 2) A test or calibration report that contains both data covered by the accreditation and data provided by a subcontractor shall clearly identify the data that were provided by the subcontracted laboratory.

____ 3) The report must prominently display the following statement at the beginning of the report: “This report contains data that were produced under subcontract by Laboratory X.” If the subcontracted laboratory is accredited by NVLAP, then its Lab Code should also be stated.

____ 4) If the subcontracted laboratory is accredited by a body other than NVLAP, then the name of the accreditation body and the laboratory’s number or other unique identifier should also be stated. If the subcontracted laboratory is not accredited, then this must be stated.

____ **j)** Each test or calibration report bearing the term and/or symbol shall include a statement that the report must not be used by the client to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the Federal Government.

____ **k)** When used in a contract or proposal, the term and/or symbol shall be accompanied by a description of the laboratory’s scope of accreditation and current accreditation status.

____ **l)** Laboratories shall not use the terms *certified* or *registered* when referencing their NVLAP accreditation or conformance to ISO/IEC 17025 requirements. The correct term is *accredited*.

Annex B (normative)

Implementation of traceability policy in accredited laboratories

B.1 Policy overview

It is a fundamental requirement that the results of all accredited calibrations and the results of all calibrations required to support accredited tests shall be traceable to the SI (the International System of Units) through standards maintained by the National Institute of Standards and Technology (NIST) or other internationally recognized national metrology institutes (NMIs). NIST Handbook 150 (and ISO/IEC 17025) details the specific requirements for traceability to be met by testing and calibration laboratories. This annex provides guidance as to how these requirements may be met and how traceability of measurement can be assured by an accredited laboratory.

Internationally recognized NMIs are those that are signatory to the Comité International des Poids et Mesures (CIPM) Mutual Recognition Arrangement (MRA) titled “Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes” and that have the necessary calibration services listed in Appendix C of the MRA, Calibration and Measurement Capabilities (CMC). For more details on the CIPM MRA and the CMC database, please see <http://www.bipm.org/en/convention/mra/> or visit the NVLAP web site.

B.2 General

____ a) Laboratories shall be able to demonstrate proper use of traceable standards and test and measurement equipment by competent laboratory personnel in a suitable environment in performing the tests for which accreditation is desired or held. This demonstration will include the determination of the appropriate measurement uncertainty.

____ b) Calibration certificates received by NVLAP-accredited testing and calibration laboratories with new or recalibrated equipment shall meet the requirements of ISO/IEC 17025. The certificates must include the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof.

Note to assessor: The NVLAP assessor(s) must, for each measurement parameter, indicate which method the laboratory has employed to achieve traceability. Select from B.3.1, B.3.2, B.3.3, B.3.4, or B.3.5 below. If B.3.4 or B.3.5 is selected, supporting documentation is also required as indicated.

B.3 Demonstration of traceability

____ **B.3.1** NVLAP-accredited laboratories may submit appropriate physical standards and test and measurement equipment directly to NIST or, when appropriate, to another national metrology institute. Accredited laboratories may obtain certified reference materials from NIST (called Standard Reference Materials under copyright) or from another national metrology institute. Use of a national metrology institute other than NIST shall be documented and will be assessed by NVLAP.

____ **B.3.2** Testing laboratories that perform calibrations only for themselves do not need to be accredited as calibration laboratories. Calibration laboratories that perform specific calibrations only for themselves to support their accredited services do not need to be accredited for those calibrations. For the purpose of assuring traceability, an accredited laboratory may calibrate its own equipment if the appropriate requirements of NIST Handbook 150 have been met.

____ **B.3.3** NVLAP-accredited laboratories that do not demonstrate traceability as described in B.3.1 or B.3.2, shall use accredited calibration laboratory services wherever available. Accredited calibration laboratories are those accredited by NVLAP or by any accrediting body with which NVLAP has a mutual recognition arrangement. A listing of NVLAP-accredited calibration laboratories and of accreditation bodies with which NVLAP currently has agreements is available from NVLAP.

____ **B.3.4** If a NVLAP-accredited laboratory submits physical standards or test and measurement equipment to a calibration service provider that is not accredited by NVLAP or by an accrediting body with which NVLAP has a mutual recognition arrangement, the laboratory shall:

____ a) document that an appropriate accredited calibration service provider is not available;

____ b) audit the claim of traceability of the provider of the calibration service and document the following areas related to the calibration and claim of traceability of its standards and test and measurement equipment:

    ____ 1) information regarding assessment of the quality system used by the calibration service provider,

    ____ 2) the calibration procedure(s) used by the calibration service provider,

    ____ 3) the physical standards or other test and measurement equipment used by the calibration service provider (including evidence of traceability to standards maintained by NIST or an appropriate national metrology institute and copies of relevant calibration certificates),

    ____ 4) information regarding the calibration intervals of relevant standards or other test and measurement equipment,

    ____ 5) the environmental conditions of the laboratory,

____ 6) the method(s) by which uncertainties are determined (e.g., *Guide to the Expression of Uncertainty in Measurement* (GUM), and

____ 7) the relative uncertainties achieved at all steps of the process;

____ c) pursue the traceability chain until traceability to appropriate stated references is completely validated, when a calibration service provider submits physical standards and/or test and measurement equipment used in the calibration to another laboratory(s) not accredited by NVLAP;

____ d) enter the audit documentation, including all findings of nonconformance and resolutions of those findings, into the laboratory's quality management record-keeping system.

NOTE An on-site visit to the provider of the calibration service is encouraged, but is not required as long as the information listed above is obtained and otherwise verified. Self-declaration of compliance to ISO/IEC 17025 or other relevant standards by a calibration service provider is not acceptable evidence of verification of traceability. Citation of a NIST Test Number by the calibration service provider likewise is not acceptable evidence of verification of traceability.

____ **B.3.5** If traceable calibration services are not available or appropriate, laboratories may demonstrate comparison to a widely used standard that is clearly specified and mutually agreeable to all parties concerned, particularly in measurements where NIST does not maintain a U.S. national standard. For example, NIST does not maintain a standard for all hardness testing scales. There are several widely used commercial standards available for hardness. However, these standards may not all give equivalent measurement results; therefore, it is important to specify which standard is used and to obtain agreement among all parties involved that the choice made is acceptable.

NVLAP LAB CODE: ☐

NIST HANDBOOK 150 CHECKLIST
COMMENTS AND NONCONFORMITIES

Instructions to the Assessor: Use this sheet to document comments and nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a “C” and nonconformities with an “X.” If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	*C or X*	*Comments and/or Nonconformities*

NVLAP LAB CODE:

NIST HANDBOOK 150 CHECKLIST
COMMENTS AND NONCONFORMITIES

Instructions to the Assessor: Use this sheet to document comments and nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a “C” and nonconformities with an “X.” If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	***C or X***	***Comments and/or Nonconformities***

Enter Date: **Enter NVLAP Lab Code:**

NIST HANDBOOK 150-3 CHECKLIST

BULK ASBESTOS ANALYSIS

Instructions to the Assessor: This checklist addresses specific accreditation requirements prescribed in NIST Handbook 150-3, Bulk Asbestos Analysis (2006 edition).

- All items on this checklist shall be addressed.
- Place an "OK" beside each item that you observed or verified at the laboratory.
- Place an X" beside any of the following items that represent a nonconformity (formerly called "deficiency").
- Place a "C" beside each item on which you are commenting for other reasons.
- Place a "N/A" beside each item that does not apply.
- Record the item number and your nonconformity explanation and/or comments on the appropriate comment sheet(s).

Note: The numbering of the checklist items correlates to the numbering scheme in NIST Handbook 150-3, clauses 1, 3, 4, and 5.

1 General Information

1.4 References

The laboratory shall have the following documents available:

___ 1.4.1 NIST Handbook 150, *NVLAP Procedures and General Requirements*;

___ 1.4.2 NIST Handbook 150-3, *Bulk Asbestos Analysis*;

___ 1.4.3 U. S. Environmental Protection Agency (EPA) *Interim Method for the Determination of Asbestos in Bulk Insulation Samples* (EPA 600/M4-82-020) as found in 40 CFR, Part 763, Appendix E to Subpart E, or the current U. S. EPA method for the analysis of asbestos in building material;

___ 1.4.4 *Asbestos-containing Materials in Schools, Final Rule and Notice*, as found in the Federal Register, Volume 52, No. 210, pages 41826 – 41846;

___ 1.4.5 U. S. EPA *Method for the Determination of Asbestos in Bulk Building Materials* (EPA/600/R-93/116, July 1993), R. L. Perkins and B. W. Harvey;

___ 1.4.6 NIST Technical Note 1297, 1994 Edition, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, Barry N. Taylor and Chris E. Kuyatt;

___ 1.4.7 reference text(s) on optical mineralogy and crystallography;

___ 1.4.8 general reference text(s) on statistics and quality assurance.

3 Accreditation process

3.2 Management system review

___ 3.2.1 Prior to the assessment, the laboratory shall provide a copy of the laboratory's management system and relevant documented procedures to the assessor for review.

___ 3.2.2 The laboratory shall provide additional technical documentation and/or records as required.

___ 3.3 On-site assessment

___ 3.3.1 All laboratory equipment shall be available and in good working order.

___ 3.3.2 At least one staff member shall be available to answer questions during the assessment.

___ 3.3.3 Laboratory personnel shall be prepared to demonstrate selected procedures or tests, as requested.

___ 3.3.4 Results of past proficiency tests shall be made available for review during the assessment.

3.4 Proficiency testing

___ 3.4.1 The laboratory shall participate in the NVLAP Proficiency Testing (PT) Program (See NIST Handbook 150-3, Section 3.4, Proficiency Testing).

___ 3.4.2 PT analyses shall not be contracted out to another laboratory.

___ 3.4.3 The laboratory shall keep and utilize PT materials for use as in-house instructional materials.

___ 3.4.4 All analysts (*full, part-time, and those in sub-facilities)* shall participate in all PT rounds (*all analysts need not participate in PT prior to returning the results to NVLAP, but all analysts shall participate at a later date, without prior knowledge of the test results).*

___ 3.4.5 Each analyst shall separately analyze, record, and report test results.

___ 3.4.6 One result shall be reported back to NVLAP by the laboratory unless specified otherwise in the testing instructions.

___ 3.4.7 Procedures and calculations (if any) shall be documented as to how the result was determined.

___ 3.4.8 Problems indicated by PT shall be discussed with appropriate laboratory personnel and documented (see NIST Handbook 150, Section 4.11, *Corrective action).*

___ 3.4.9 Plans for resolving analytical problems shall be developed, implemented and documented.

___ 3.4.10 The PT results shall be used to verify accuracy and precision for each analyst and to judge the analyst's overall performance.

___ 3.4.11 PT results shall be used for inter-analyst comparisons and entered into the laboratory's management system records.

4 Management requirements for accreditation

4.1 Organization – sub-facilities

___ 4.1.1 A sub-facility is technically dependent on the main facility (i.e., technical management and supervision shall be provided by the main facility).

___ 4.1.2 Quality assurance activities of the sub-facility shall be directed by the main facility.

___ 4.1.3 The nature, scope, and frequency of on-site quality assurance reviews, by the main facility quality manager (or equivalent) shall be:

___ a) Clearly defined in the management system;

___ b) Appropriate for the nature and scope of the work performed by the sub-facility.

___ 4.1.4 All permanent quality assurance and personnel records shall be retained at the main facility.

___ 4.1.5 Quality assurance data from each sub-facility shall be regularly and routinely compared both to the main facility's data, and data from other sub-facilities.

___ 4.1.6 Records of such comparisons shall be retained in quality assurance records along with actions taken to evaluate and resolve differences.

___ 4.1.7 Analysts at sub-facilities shall participate in NVLAP proficiency testing and records shall be maintained of individual results.

4.2 Management system

(See NIST Handbook 150 checklist)

4.3 Document control

(See NIST Handbook 150 checklist)

4.4 Review of requests, tenders and contracts

(See NIST Handbook 150 checklist)

4.5 Subcontracting of tests and calibrations

___ 4.5.1 A laboratory that subcontracts AHERA work shall do so with another laboratory accredited by NVLAP for bulk asbestos analysis.

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

4.6 Purchasing services and supplies

(See NIST Handbook 150 checklist)

4.7 Service to the customer

(See NIST Handbook 150 checklist)

4.8 Complaints

(See NIST Handbook 150 checklist)

4.9 Control of nonconforming testing and/or calibration work

(See NIST Handbook 150 checklist)

4.10 Improvement

(See NIST Handbook 150 checklist)

4.11 Corrective action

(See NIST Handbook 150 checklist)

4.12 Preventive action

(See NIST Handbook 150 checklist)

4.13 Control of records

___ 4.13.1 The period of retention of records shall be three years, unless a longer period is required by the customer, regulation, or the laboratory's own procedures.

___ 4.13.2 The records maintained by the laboratory for three years shall include:

___ a) sample custody records;

___ b) original data collected (including all of the required optical data for each analysis it performs), signed (or initialed), and dated by the analyst;

___ c) contamination monitoring data;

___ d) calibration and verification data;

___ e) data and results of quality control;

___ f) equipment and maintenance records;

___ g) test reports;

___ h) records of proficiency testing results for each analyst.

4.14 Internal audits

___ 4.14.1 The laboratory shall conduct an annual internal audit, which is separate and distinct from management reviews, covering compliance with NVLAP requirements, the laboratory management system, regulatory, contractual, and testing requirements. The audit results shall be documented in the formal report addressing any corrective and/or preventive actions taken.

___ 4.14.2 An applicant laboratory shall conduct at least one complete internal audit prior to the first on-site assessment and a record of such shall be provided to the assessor before or during the on-site assessment.

___ 4.14.3 Reports for renewal laboratory internal audits conducted since the previous assessment shall be made available for review.

4.15 Management reviews

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

___ 4.15.2 Management reviews, which are separate and distinct from the internal audit, shall review all nonconformities and may reflect positive aspects of the management system.

___ 4.15.3 An applicant laboratory shall perform at least one complete management review prior to the first assessment and a record of such shall be provided to the assessor before or during the on-site assessment.

___ 4.15.4 Renewal laboratory management reviews conducted since the previous assessment shall be made available for review.

5 Technical requirements for accreditation

5.2 Personnel

___ 5.2.1 The laboratory shall maintain documentaiton for each staff member including:

___ a) staff member’s title and job position description;

___ b) job and quality assurance responsibilities, including assigned laboratory procedures and duties;

___ c) résumé;

___ d) training;

___ e) results of quality assurance activities, including precision and accuracy and the results of NVLAP proficiency testing;

___ f) accuracy, precision and error data;

___ g) correction of nonconformities.

___ 5.2.2 The laboratory shall have a detailed, documented description of its training program for new and current staff members.

___ 5.2.3 The laboratory training shall include:

___ a) criteria for successful completion;

___ b) training with blanks and blind testing to determine competency;

___ c) new analysts' results that have been checked by either an experienced analyst (with an acceptable error rate), or by an independent technique, until the analyst has an acceptable error rate.

___ 5.2.4 The laboratory shall document performance criteria to determine when a new analyst is qualified to work independently.

___ 5.2.5 Laboratory analysts shall be able to obtain enough information from the laboratory's quality documentation to perform analyses in the absence of the laboratory manager.

___ 5.2.6 All staff members shall be trained in their role in the quality assurance system.

___ 5.2.7 The laboratory shall ensure that staff members are aware of the extent of their area(s) of responsibility.

___ 5.2.8 The laboratory shall ensure that:

___ a) analysts and technical supervisors understand polarized light microscopy and its application to crystalline materials sufficiently to conduct analyses. That they understand what the various optical properties are, how they are measured or observed in the microscope, and how the data are used to form a conclusion about the identity of the component, (*e.g., an analyst using central and/or annular focal screening (dispersion staining) to measure refractive index must be able to explain what produces the observed color and how that color is used to determine refractive index*);

___ b) analysts are competent with the polarized light microscope, can properly align the microscope and identify all of the crucial parts;

___ c) technical supervisors have a fundamental knowledge of the method to assure the quality of the laboratory's results.

___ 5.2.9 The laboratory shall be organized so that staff members are not subjected to undue pressure or inducement that might influence their judgment or the results of their work.

___ 5.2.10 The laboratory shall be able to demonstrate that the sample workload required for each analyst is consistent with accurate and precise analytical measurements.

___ 5.2.11 The laboratory shall require and document that analysts and technical supervisors participate in some form of continuing education such as formal course work, in-house education, and scientific/technical meetings. Staff shall also have access to journals that describe advances in the field of microscopy and asbestos analysis.

5.3 Accommodation and environmental conditions

___ 5.3.1 The laboratory shall have the proper facilities, including space, lighting, environmental controls, etc., to perform analyses and store asbestos in accordance with federal, state and local laws, and to maintain sample integrity.

___ 5.3.2 The laboratory shall have the procedures for the use of blanks of asbsestos-free material to determine the presence, quantity, and consistency of asbestos contamination in their analytical process and have related procedures to control it.

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

5.4 Test and calibration methods and method validation

___ 5.4.1 The laboratory shall use the U. S. EPA *Interim Method for the Determination of Asbestos in Bulk Insulation Samples* as found in 40 CFR, Part 763, Subpart E, Appendix E (formerly Subpart F, Appendix A), or the current U. S. EPA method for the analysis of asbestos in building materials.

NOTE: On August 1, 1994, the EPA announced in the Federal Register that EPA/600/R-93/116 provides clarification and improvements to the 1982 protocol and recommended that it serve as a preferred substitute method. EPA has determined that it is more capable of producing accurate results than the 1982 method. However, NVLAP must still accredit to the 1982 method; laboratories that use the 1993 method must indicate such on their test reports.

___ 5.4.2 If departures from the method are made, the laboratory shall have written procedures detailing how the analyses are conducted.

___ 5.4.3 The laboratory shall have a written procedure for the analysis of samples and methods to ensure the accuracy and precision of analyses. The analytical procedure shall detail the PLM test method, including the measurement of each of the required optical/physical properties, as it is applied in the laboratory (a sample copy of the AHERA test method is not sufficient).

___ 5.4.4 The laboratory shall have a clear and documented definition of each asbestos type that includes the acceptable optical properties (e.g., such as the range in refractive indices) that the fibers can exhibit and still be identified as the particular asbestos type, and what constitutes asbestiform morphology.

___ 5.4.5 The laboratory shall determine the identification of fibrous materials by measuring the optical properties (see 5.10.2).

___ 5.4.6 The laboratory shall have a written procedure for dealing with samples in which the fibers are heavily coated with binder that hinders analysis.

___ 5.4.7 The laboratory shall maintain a list of non-asbestos fibers that can be confused with asbestos and the specific optical properties for each that can be used to distinguish between asbestos and non-asbestos.

___ 5.4.8 The laboratory shall measure and record at least one optical property for non-asbestos fibers that serves to distinguish them from asbestos.

___ 5.4.9 The laboratory shall have specific sample preparation techniques for dealing with samples that are semi- or non-friable.

___ 5.4.10 The laboratory shall use the point-count technique or a technique that it has demonstrated, and documented, to be equivalent for quantitative analysis (see 5.6.7)

___ 5.4.11 The laboratory shall homogenize the sample in some way or analyze a sufficient number of sub-samples to obtain a representative analysis (at least three sub-samples for negative samples, as per EPA/600/R-93/116).

___ 5.4.12 The laboratory shall have a working definition of trace and be able to distinguish between trace concentrations of asbetos and concentrations near 1 %.

___

NOTE: It is important to differentiate the following two cases for a very practical reason: whether additonal analysis by a different method is warranted to ascertain the asbestos concentration reported by the PLM analyst.

a) When reporting the asbetos content as “trace,” the analyst ascertains that although there are asbestos fibers in the sample, its concentration is so low that it is far from the level of 1 % by calibrated visual estimate (CVE) or point counting. In this case, the analyst is confident that it is not necessary to make a recommendation to the customer that the sample should be analyzed by a more accurate and precise method to verify results.

b) When reporting the asbestos content as near to, but less than 1 %, the analyst ascertains that the asbestos concentration is definitely not equal to or higher than 1 % by CVE or point counting. Due to the inherent uncertainty of the quantification technique(s) employed during analysis, the analyst should recommend to the customer that verification of the results is necessary by a more accurate and precise method.

5.5 Equipment

___ 5.5.1 The laboratory shall have documentation for equipment maintenance and calibration.

___ 5.5.2 The laboratory shall have the following equipment and materials:

___ a) HEPA-filtered, Class I biohazard hood, or a glove box with continuous airflow (negative pressure);

___ b) sampling utensils, (scalpels, forceps, probes, needles, tweezers, razors, etc.);

___ c) microscope slides and cover slips;

___ d) refractive index liquids, 1.490 – 1.720 in increments of less than or equal to 0.005 (high dispersion liquids are optional);

___ e) stereomicroscope or low power binocular microscope, approximately 10 X – 45 X, with light source;

___ f) mortar and pestle;

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

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

___ 5.5.3 The laboratory shall have a polarized light microscope (PLM) with:

___ a) binocular or monocular with cross hair reticule (or functional equivalent) that locks into position or is marked, and that does not rotate during normal operation of the microscope;

___ b) low (≥ 5 X and ≤ 15 X), medium (> 15 X and < 40 X), and high (≥ 40 X) objectives;

___ c) light source;

___ d) 360 ° rotating stage;

___ e) substage condenser with iris diaphragm;

___ f) polarizer and analyzer that can be positioned so that their privileged directions are at 90 ° to each other;

___ g) accessory slot at 45 ° to polarizers for compensators;

___ h) first-order red (or λ) compensator (530 – 550 nm retardation);

___ i) dispersion staining objective complete with accessories (optional);

___ j) test slide made of orthorhombic fibers, such as anthophyllite from SRM 1867, or other straight fibers of parallel extinction, such as polypropylene, polyethylene, etc., for aligning cross hairs with the privileged directions of the polarizer and analyzer.

___ 5.5.4 The laboratory shall ensure that each microscope is in proper working condition. The optical system, including objectives, condensers, polarizers, etc., shall not be damaged or modified in any way that would affect microscope resolution or depolarize the light, (i.e., the lens is relatively free of scratches, nicks, corrosion, signs of impact, etc., and there is no stop in the back focal plane other than for dispersion staining objectives). PCM objectives are generally not suitable for PLM work.

___ 5.5.5 The laboratory shall have written procedures for aligning the polarized light microscope daily (or prior to use) in such a way that:

___ a) the privileged directions of the sub-stage polarizer and the analyzer are oriented at 90 ° to one another. The orientation of the privileged directions of the polarizers shall be known. The accessory slot shall be at 45 ° to these privileged directions;

___ b) the ocular cross hairs coincide with the privileged directions of the polarizer and the analyzer. This condition shall be verified with a test slide (or similar standard);

___ c) the objectives and/or stage are centered to prevent any grains from leaving the fields of view during stage rotation;

___ d) the sub-stage condenser, which is visualized through the image of the (closed down) field diaphragm, is centered on the optic axis;

___ e) the daily alignment check is recorded in a logbook.

___ 5.5.6 The laboratory shall have calibrated refractive index solids, or a refractometer (or access to one), for calibrating refractive index liquids.

___ 5.5.7 The laboratory shall have written procedures for calibrating refractive index liquids, including the lot number for each of the measured oils, to determine whether their actual or calibrated RI value at 589 nm and 20 °C, are within ± 0.004 of their nominal values. The procedures shall include:

___ a) if the calibrated RI value at 589 nm and 25 °C, deviates more than ± 0.004 from the nominal value, the liquid shall not be used;

___ b) the temperature at the workstation at the time of calibration shall be recorded and, if not 25 °C, used to perform temperature correction of the calibrated RI value.

___ 5.5.8 The laboratory shall maintain the necessary equipment for any optional procedure(s) it performs.

5.6 Measurement traceability

___ 5.6.1 The laboratory shall have reference materials and if available, Standard Reference Materials (SRMs) with certificates for chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite and glass fiber traceable to NIST (SRM 1866 and SRM 1867);

***NOTE**: The SRMs, if available, may be purchased from the NIST Standard Reference Materials Program (SRMP), 100 Bureau Drive, Stop 2322, Gaithersburg, MD 20899-2322; phone (301) 975-8776.*

___ 5.6.2 The laboratory shall have a qualitative error rate of less than 1 %, which is calculated by dividing the number of qualitative errors (false negatives, false positives, and asbestos types) by the total number of QA samples.

___ 5.6.3 The laboratory shall identify problem samples, such as floor tiles, that are difficult to analyze qualitatively and shall have specific written procedures to deal with problem samples to reduce the errors to less than 1 %.

___ 5.6.4 The laboratory shall have written procedures describing how reference standards are used to verify the accuracy of an analyst's ability to correctly determine the optical properties of asbestos.

___ 5.6.5 The laboratory shall determine precision on the qualitative and quantitative analyses of samples by:

___ a) repeatability – repeat analyses by the same analyst;

___ b) comparison of results from multiple slide mounts of the same material;

___ c) reproducibility – analysis of samples by multiple analysts, if possible (single-analyst laboratories require more interlaboratory data);

___ d) interlaboratory analysis – analysis of samples by other laboratories.

___ 5.6.6 The laboratory shall determine the accuracy of the qualitative and quantitative analyses of samples by:

___ a) analysis of proficiency testing materials;

___ b) analysis of standards, either prepared in-house or purchased;

___ c) analysis of samples using independent methods (e.g., XRD, gravimetry, etc.).

___ 5.6.7 If an estimation technique that is equivalent to point counting is used, the laboratory shall use one or more of the following for calibration and have data to show equivalency:

___ a) bulk standards – percent asbestos in these standards must have been determined by non-visual estimation method(s). This could include repeat analysis by point counting, by quantitative XRD, or by gravimetric measurement of asbestos during sample formulation;

___ b) prepared (permanent) slides that have been point counted;

___ c) photomicrographs of grain mounts that have been calibrated for relative area;

___ d) other appropriate standards.

5.7 Sampling

___ Specific sampling procedures shall be documented where appropriate (especially for samples containing multiple layers).

5.8 Handling of test and calibration items

___ 5.8.1 The laboratory shall have written procedures and/or instructions describing the following:

___ a) sample custody and handling procedures, i.e., sample receipt, log-in, storage and disposal;

___ b) disposal/return of bulk samples (including retaining the documentation with all other data and information regarding the sample);

___ c) proper storage of materials to prevent damage or cross contamination;

___ d) sample retention (hold samples for a minimum of 30 days after analysis unless earlier return is requested by the customer or prevented by law or regulation);

___ e) safe disposal of bulk samples and in accordance with any and all federal, state and local regulations.

___ 5.8.2 The laboratory shall have a sample log system used to uniquely identify the test item and document the action. The log shall include:

___ a) the date of receipt of the test item;

___ b) the condition of the test item;

___ c) documentation of acceptance or rejection of test item, reasons for rejection (e.g., air samples mixed with bulk samples);

___ d) a unique laboratory identification number for each test sample;

___ e) the customer identification number, which is the number that the customer (or sample taker) assigns to the test item;

___ f) the initials of the person making the above entries in the sample log book.

5.9 Assuring the quality of test and calibration results

___ 5.9.1 The laboratory shall ensure that the quality assurance analyses represent at least 10 % of the total number of analyses performed.

___ 5.9.2 Quality assurance analyses shall be performed regularly covering all time periods, sample types, instruments, tasks, and personnel.

___ 5.9.3 The selection of samples shall be semi-random, focusing more on positive and difficult samples, and, when possible, the checks on personnel performance executed without their prior knoweldge.

___ 5.9.4 QC samples shall be analyzed routinely with actual workload, and in an on-going manner.

___ 5.9.5 The laboratory shall maintain and summarize all of the quality assurance activities at least monthly to include:

___ a) contamination checks using asbestos-free, non-fibrous material, such as corn starch or fine table salt;

___ b) internal and NIST proficiency testing for each analyst;

___ c) interlaboratory analyses;

___ d) overall accuracy and precision for each microscopist for qualitative and quantitative analyses as defined in its management system documentation;

___ e) control charts for accuracy and precision;

___ f) identification of any sample custody errors, such as mixing up samples, losing samples, etc.;

___ g) comparison of results of independent techniques with PLM results, if appropriate;

___ h) corrections to nonconformities;

___ i) the total qualitative error rate of the laboratory.

___ 5.9.6 The laboratory shall have a documented procedure for constructing control charts, including the algorithms for calculating warning and control limits.

5.10 Reporting the results

___ 5.10.1 The laboratory shall record the following stereomicroscopic data for bulk examination to include:

___ a) homogeneity;

___ b) texture;

___ c) color;

___ d) estimated concentration of asbestos.

___ 5.10.2 The laboratory shall record the following data for the asbestos type(s) identified by PLM examination:

___ a) morphology;

___ b) color and pleochroism;

___ c) indices of refraction (nD) parallel and perpendicular for each asbestiform;

___ d) birefringence;

___ e) extinction characteristics, including measurement of extinction angles, if observed;

___ f) sign of elongation;

___ g) estimated concentration of asbestos;

___ h) temperature at the workstation at the time of analysis;

___ i) result of the analysis.

___ 5.10.3 Each test report shall include the following information:

___ a) color;

___ b) presence or absence of asbestos;

___ c) type(s) of asbestos present;

___ d) estimate of the area percent for each type of asbestos present;

___ e) identity of other fibrous materials (if known);

___ f) estimate of the area percent for other fibrous materials present;

___ g) identity of matrix materials, if known;

___ h) a statement indicating if the sample is inhomogenous and if sub-samples of the components were analyzed separately;

___ i) a description of any problems encountered in the analysis;

___ j) departures from the test method;

___ k) an approved signatory's signature.

___ 5.10.4 The laboratory shall report the results of samples containing one or more layers consistent with the current EPA guidelines.

___ 5.10.5 The laboratory shall ensure that the customer receives a copy of the test report, either paper or electronic. Electronic copies must be in a tamper-resistant format.

DATE: **NVLAP LAB CODE:**

NIST HANDBOOK 150-3 CHECKLIST
COMMENTS AND NONCONFORMITIES

Instructions to the Assessor: Use this sheet to document comments and nonconformities. For each, identify the appropriate item number from the checklist. Identify each comment with a "C" and each nonconformity with an "X." If additional space is needed, make copies of this page or use additional blank sheets.

Item No.	*C or X*	*Comments and/or Nonconformities*

Exhibit #9
QUALITY ASSURANCE MANUAL TRAINING SUMMARY

Quality Assurance Manual Date and Revision Number: ___________________________

The current QA Manual is available on the AES_Portal Server Location: Document Library: Documents: Quality Assurance: QA Manual: Asbestos "(year) Asbestos QA Manual"

<u>Please restrict your use to viewing the read-only version on computer.</u>

My signature confirms that I have been trained on the ________ (year) QA Manual Rev _____

Supervisor: ____________________________________

Section/area: ____________________________________

Name	Date

Exhibit #10

PLM QUALITY ASSURANCE PROGRAM SUMMARY OF MONTHLY ACCURACY

Microanalyst: ____________________________________

Month: ____________________ Year: ________________

	Amounts
1. Internal Standards	
2. NVLAP Tests Samples	
3. Round Robin Samples	
4. Accepted Analysis	
5. Rejected Analysis	
6. Total Samples	
7. Overall Accuracy, %	

Corrective actions for deficiencies ______________________________________
____

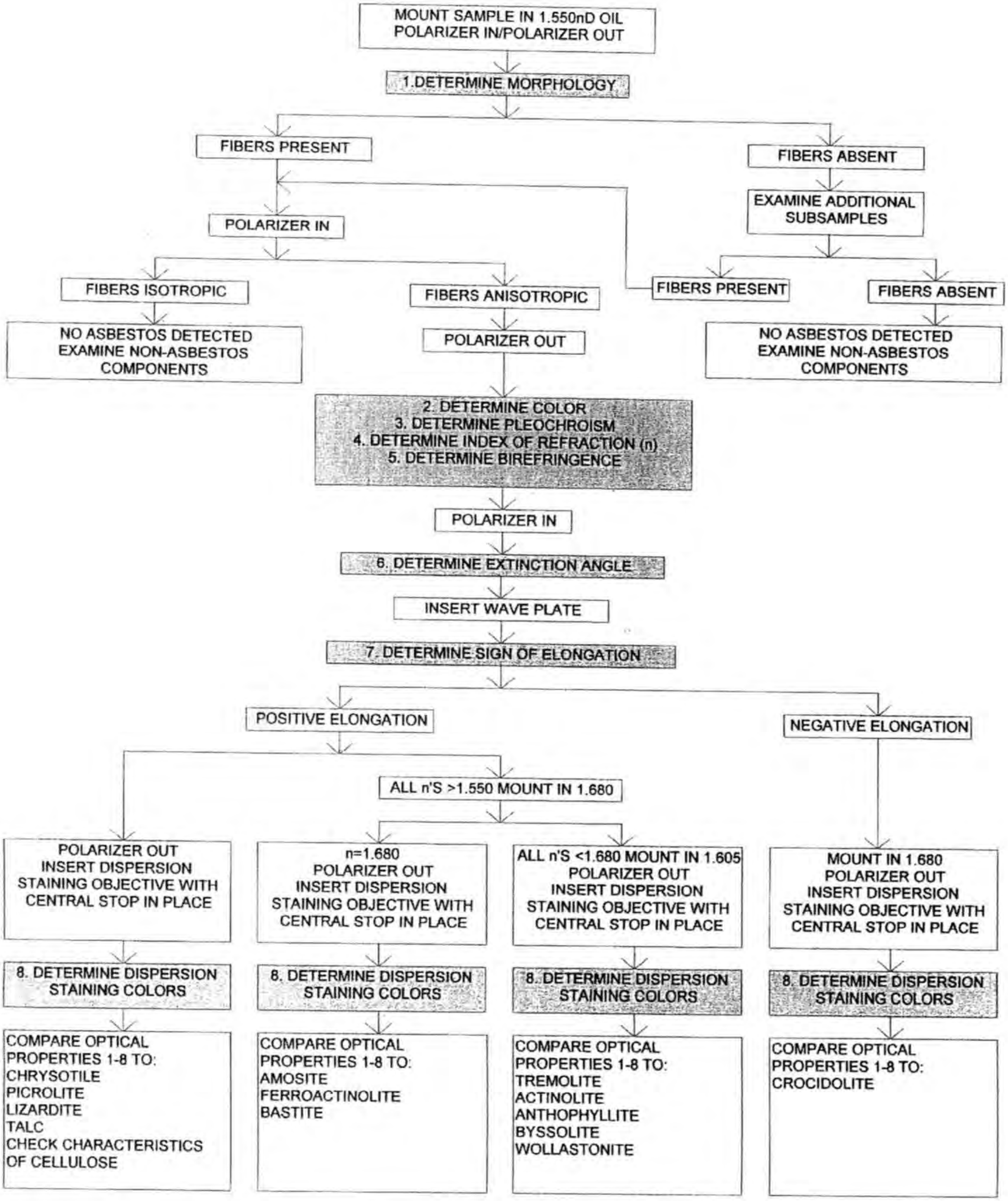
QA Officer __________________ Date ________________

Lab Manager __________________ Date ________________

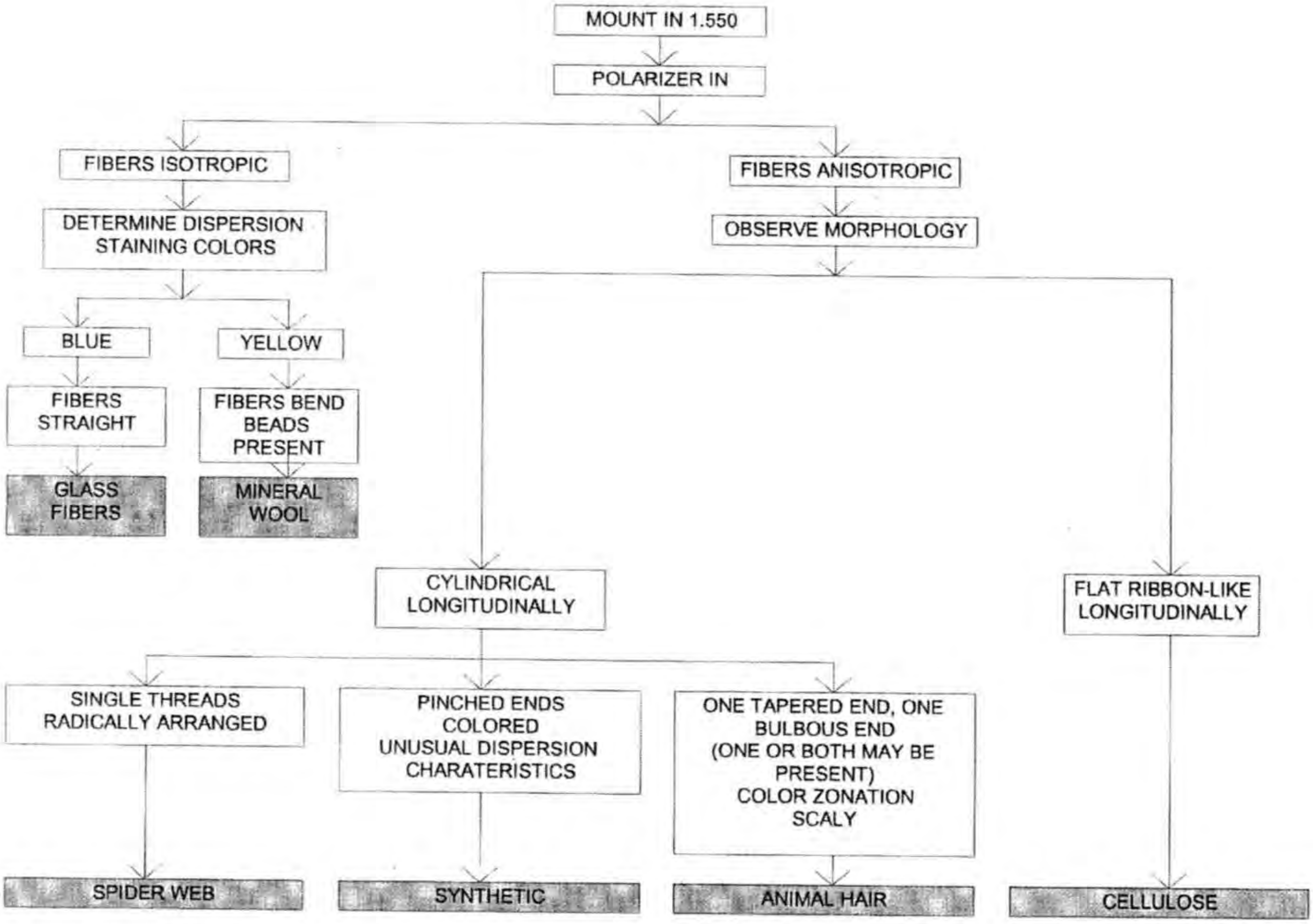
Exhibit #11

ANALYSIS SEQUENCE OF ASBESTOS FIBERS FLOW CHART

Analysis Sequence of Asbestos Fibers Flow Chart



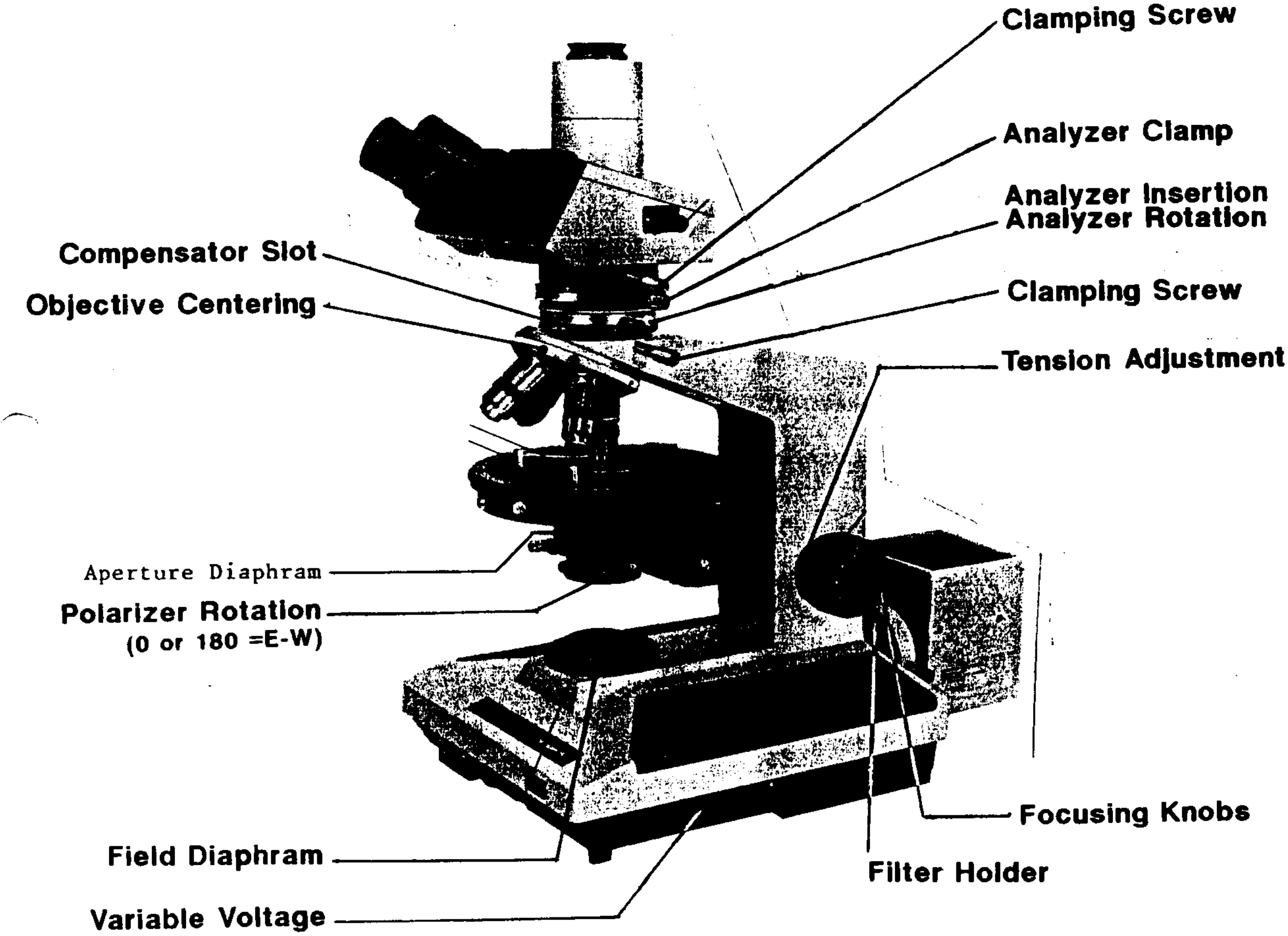
Analysis Sequence of Non-Asbestos Fibers Flow Chart



APPENDIX 1

Calibration of Microscope

OLYMPUS BH-2


Clamping Screw
Analyzer Clamp
Analyzer Insertion
Analyzer Rotation
Compensator Slot
Objective Centering
Clamping Screw
Tension Adjustment
Aperture Diaphram
Polarizer Rotation
(0 or 180 =E-W)
Focusing Knobs
Filter Holder
Field Diaphram
Variable Voltage

KOHLER ILLUMINATION WITH THE OLYMPUS BH-2

1. Turn on lamp; set at 6V.

2. Adjust the interocular distance for your eyes; and focus the 10X objective on an uncrowded field of particles (e.g., prepared slide 92).

3. Rotate 40X objective into position, trim focus with fine focus adjustment.

4. Focus ocular crosslines for your right eye by rotating the top lens of the right ocular. (You may then have to refocus the field of view with the fine focus.)

5. Focus microscope on a single very tiny pinpoint particle using the right eye and right ocular only. (Tilt your head slightly or otherwise to avoid looking through the left ocular.)

6. Next, look through only the left ocular with your left eye at the same particle but focus using the diopter adjustment on the left ocular tube. (Do not use the microscope fine focus adjustment).

7. Using both eyes on the focused preparation, rotate the stage. If all particles do not rotate about the center of the crosslines, bring the center of rotation to the center of the crosslines using the stage centering screws (just below the stage in the 10 & 2 o'clock position).

8. Close the field diaphram on the microscope base and focus its image in the field of view by raising or lowering the substage condenser using its rack and pinion focusing knob.

9. Center the field diaphram in the field of view by centering the substage condenser (8 & 4 o'clock knobs).

10. Open the field diaphram to the inside edge of the field of view; the focused iris leaves should be just visible inside the field.

11. Swing in the 20X objective and trim the focus on the particles.

12. Center the stage rotation with the 20X objective using the centering screws on the objective.

13. Repeat steps 12-13 for both 10X objectives.

14. Introduce the Bertrand lens and the 40X objective to check the focus and centration of the filaments. If significantly off, call the instructor.

15. Whenever you change objectives, adjust the focus with the fine focus adjustment, set the field diaphram to just inside the field of view and adjust the aperture diaphram for optimum contrast and resolution.

Olympus BH-2 Microscope
To Use The Dispersion Staining (D.S.) Objective

This BH-2 Pol 'scope is fitted with 40X, 20X, and two 10X objectives. One of the two 10X objectives is fitted with a central stop for dispersion staining. It is recognizable by the stop-centering openings 120° apart on the objective barrel. All lie on the yellow band which signifies a 10X objective. The opening at the back of the objective is fitted with a spring-loaded stop against which the centering screws in the front openings hold the internal central stop assembly (the details on this D.S. objective are covered by John Delly and Joe Sirovatka in *The Microscope* 36(3), pp. 205-211 under the title, "A Dedicated Central-Stop Dispersion Staining Objective").

To center this stop, swing in the Bertrand lens and close down the aperture diaphram to check centration of the stop with the aperture iris. If not centered, the stop can be moved laterally with two allen keys in the two centering openings. Do this with a single clean slide on the stage (see No. 2 of "Things To Watch" on p. 2.10).

These D.S. objectives have been fitted to a particular rotating turret opening on a particular microscope. They should never be moved from their special positions.

To use the D.S. objective, start with prepared slide No. 67 (olivine). Remove the Bertrand lens and, if in the light path, the analyzer. Open the aperture diaphram and focus on some of the mineral grains. Close the aperture diaphram until the field of view is black all the way to the edges of the field. If impossible, to achieve a fully black field, swing out the condenser top lens and reset the aperture diaphram to just eliminate all direct light, leaving a completely dark field of view. The olivine mineral grains should appear yellow (γ index), magenta (β index) or blue (α index). Rotate the (centered) stage to observe the colors corresponding to the different refractive indices.

An annular stop in the objective back focal plane transmits a central or axial beam with an NA of about 0.05. It yields edge colors on particles of the wavelength at which the particle and liquid have identical refractive indices, i.e., their dispersion curves cross at that wavelength. Because the central stop with its otherwise black field gives brighter, more easily read, dispersion staining colors, the central stop is used in preference to the annular stop and in spite of color complementary to the actual wavelength of match.

TO ALIGN POLARS ON MICROSCOPE

1. Insert analyzer and clamp setting on rotating analyzer scale at 0o.

2. Align 0o mark on substage polarizer with white fiducial mark on the condenser. Rotate the polarizer (loosen its set screw) observing a blank field of view until the field is the blackest possible.

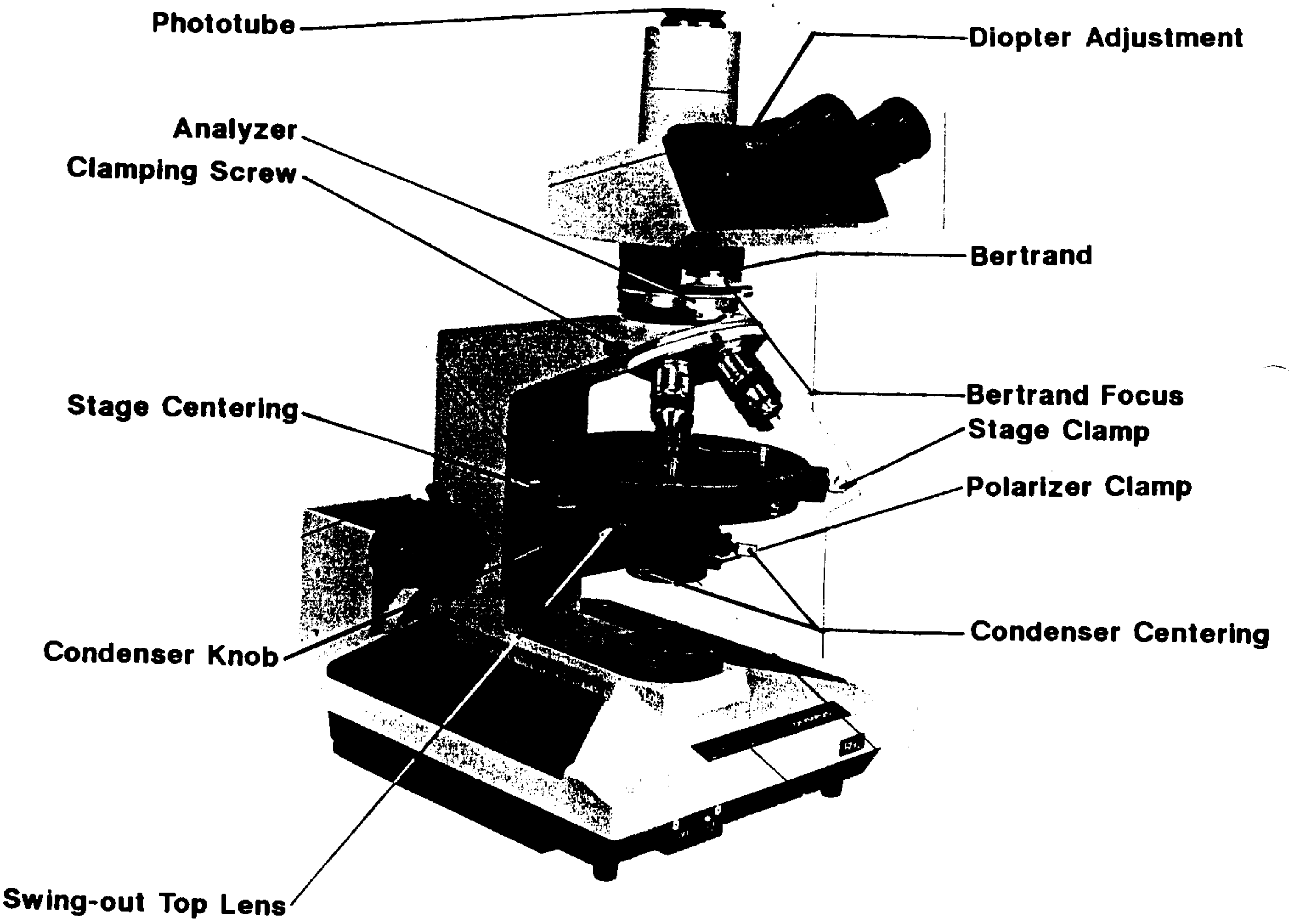
3. Make sure all crosslines are in the keyed position.

4. Focus on a polyester fiber (prepared slide 10) and rotate a centered filter about the crossline center to its blackest position at that crossline center.

5. Remove the analyzer and observe the sides of the fiber relative to the crosslines. The edges of the fiber at what was the darkest portion should be parallel to one of the crosslines. If not, loosen the trinocular head (above the analyzer) and rotate the head to achieve parallelism between the crosslines and fiber.

6. The microscope is now ready for use. If, however, the axes of oculars are unaesthetically aligned with the base of the microscope, one can restore alignment by loosening the analyzer section <u>and</u> ocular head <u>together</u> with their setscrew and rotate the two sections until parallel with the base. After tightening the bodytube in that position, observe the field of view, cross the polars and rotate the polarizer to the blackest field of view. The nylon fiber should still show good parallel extinction.

OLYMPUS BH-2


Phototube
Diopter Adjustment
Analyzer
Clamping Screw
Bertrand
Stage Centering
Bertrand Focus
Stage Clamp
Polarizer Clamp
Condenser Centering
Condenser Knob
Swing-out Top Lens

TO ALIGN POLARS ON OLYMPUS BH-2

1. Insert analyzer and clamp setting on rotating analyzer scale at 0° over 0°.

2. Align 0° mark on substage polarizer with white fiducial mark on the condenser. Rotate the polarizer (loosen its set screw) observing a blank field of view until the field is the blackest possible.

3. Clamp the polarizer in that position.

4. Focus on a nylon fiber (prepared slide 47) and rotate a centered fiber about the crossline center to its blackest position at that crossline center.

5. Remove the analyzer and observe the sides of the fiber relative to the crosslines. The edges of the fiber at what was the darkest portion should be parallel to one of the crosslines. If not, loosen the trinocular head (above the analyzer) and rotate the head to achieve parallelism between the crosslines and fiber.

6. The microscope is now ready for use. If, however, the axes of the oculars are unaesthetically aligned with the base of the 'scope—one can restore alignment by loosening the analyzer section and ocular head together with their setscrew and rotate the two sections until parallel with the base. After tightening the bodytube in that position, observe the field of view, cross the polars and rotate the polarizer to the blackest field of view. The nylon fiber should still show good parallel extinction.

2.10

Things To Watch

1. Centration of stops; centerable stops essential.
2. Watch for tilted coverslips; they form a wedge that then acts like a prism and offsets the aperture diaphram image thus uncentering the stops. Check by rotating the stage, if the aperture diaphram precesses, the preparation is a prism.
3. Light intensity should be high for central stop dispersion staining. The daylight filter should be used with 6V tungsten illumination.
4. Slides and coverslips must be very clean; good idea to use 3M Magic Tape™; clean objective front lens and substage condenser top lens also (same way).
5. Partly closed field iris often helps with small particles by increasing contrast.
6. High magnification dispersion staining objectives are not desirable; the colors are brighter with the 10X objective.
7. Trace components may be hidden by other particles.
8. Weak colors mean liquid and particle have similar dispersion; change liquid if possible; very thin particles also show weak colors.
9. Do not use crossed polars with dispersion staining.
10. If the centered aperture diaphram can't be closed behind the central stop in the objective back focal plane, the stop size should be increased or remove the top lens of the condenser.

Dispersion Staining Colors

Matching Wavelength λ_0 nm	Edge Colors† Annular Stop*	Edge Colors† Central Stop**	Becke Lines†† Central Stop Particle		Becke Lines†† Central Stop Liquid
<420	blue-black	pale yellow	pale yellow		
430	blue-violet	yellow	" "		
455	blue	golden-yellow	" "	+	violet
485	blue-green	golden-magenta	yellow	+	violet
520	green	red-magenta	orange	+	violet
560	yellow-green	magenta	red orange	+	blue-violet
595	yellow	blue-magenta	red	+	blue
625	orange	blue	faint red	+	blue
660	orange-red	blue-green	faint orange	+	blue-green
>680	brown-red	pale blue-green	yellow	+	pale blue-green

* observed on a brightfield
** observed on a darkfield

† in focus
†† on focusing up

Appendix 2

Training Program

TRAINING PROGRAM FOR PLM METHOD FOR ASBESTOS BULK SAMPLES

1. Familiarization with the PLM microscope (3 days).

   a. Microscope parts.
   b. Microscopical methods of identification.
   c. Calibration and maintenance.

2. Familiarization with the stereomicroscope (1 day).

3. Identification of non-asbestos components (1 day).

4. Identification of asbestos components (3 days).

5. Texture and description of non-asbestos components (1 day).

6. Texture and description of asbestos components (1 day).

7. Standard method for analysis of bulk samples (2 days).

   a. Interim EPA method.
   b. Point Counting method.

8. QA/QC program (2 days).

9. Participation in various accreditation programs (1 day).

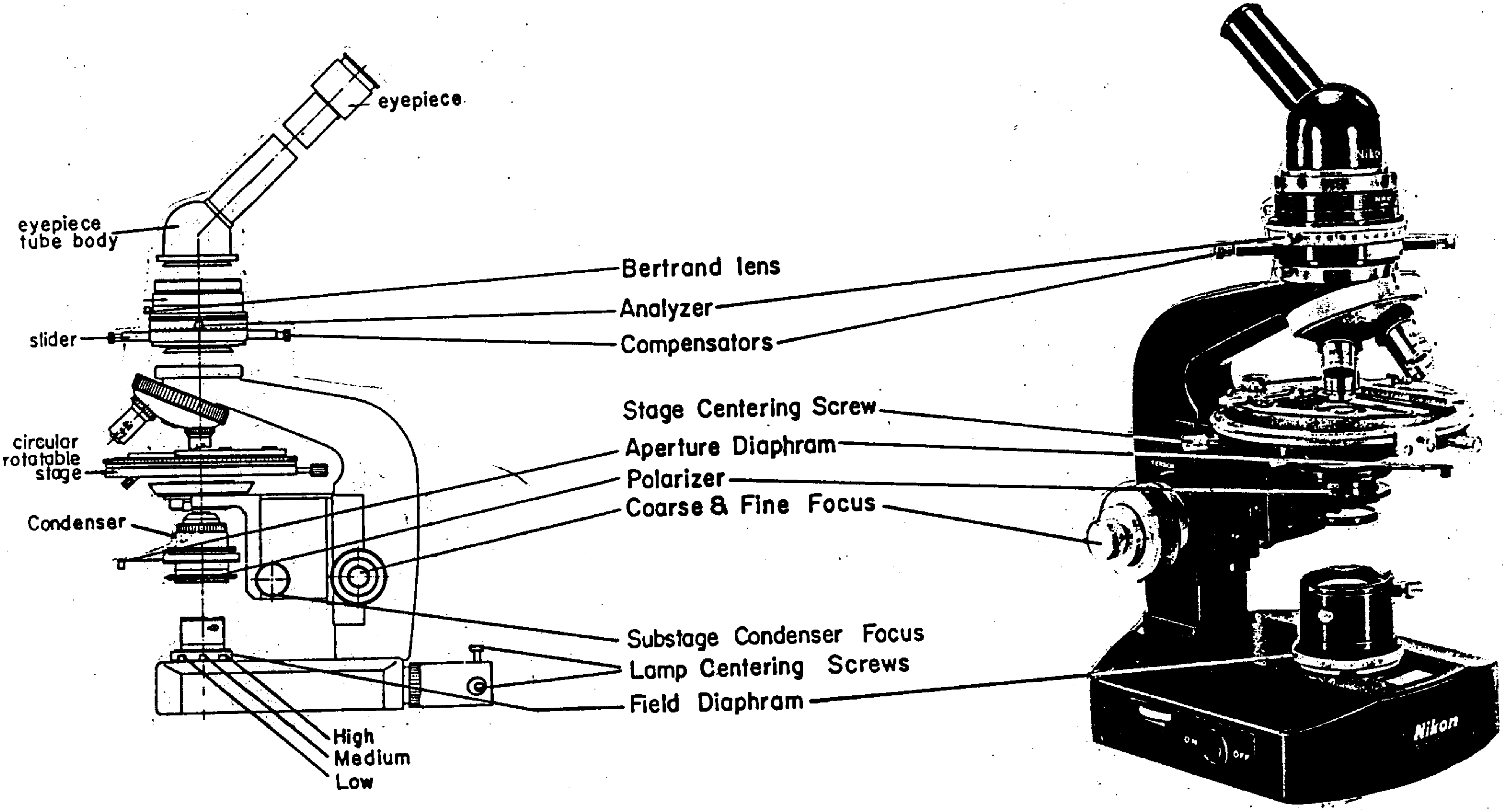
10. Regulation and laboratory Safety Procedures in the lab (1 day).

11. Review of all of the above (1/2 day).

12. Practice and training on previously analyzed samples (1 week).

13. Test for AES proficiency asbestos bulk analyst.

Polarized Light Microscopy (PLM)


eyepiece
eyepiece tube body
slider
circular rotatable stage
Condenser
Bertrand lens
Analyzer
Compensators
Stage Centering Screw
Aperture Diaphram
Polarizer
Coarse & Fine Focus
Substage Condenser Focus
Lamp Centering Screws
Field Diaphram
High
Medium
Low
Nikon

POLARIZED LIGHT MICROSCOPY

Bulk Sample Analysis for Asbestiform Minerals

LOGGING IN PROCEDURES

Bulk samples are received in various containers including plastic film cannisters to envelopes. GES protocol requires the logging in of all received bulk samples into a central log for bulk samples. All stored samples and containers must exhibit two I.D. numbers, a client and a GES I.D. number. Using a permanent marker these numbers are to be written on both the sides and the cap of the container for each respective bulk sample. If labels exist on the container(s) they must be covered with transparent tape because the containers will be washed after the examination of the contents, in order to eliminate any accidental contamination.

client

Large numbers of samples received from the same client at the same time are organized by numerical sequence prior to the entry of the entire group into the central bulk sample log. This is done to determine if all samples have been received (by checking any list supplied by client) and to determine the client I.D. numbers of any missing samples. After received samples are numbered and taped they are logged into the central bulk sample log. An example of a typical entry and the information entered into the log is included in this manual.

NOTE: All bulk samples which are received in containers other than pharmaceutical or photographic vials are transferred into the appropriate vials prior to the logging in process and subsequent storage.

BULK SAMPLE ANALYSIS PROCEDURES

The following section deals with the systematic analysis of bulk samples and provides technical information on asbestiform minerals and other possible components as well as proven techniques of analysis.

Pre-Analysis Sample Preparation

Prior to the microscopical examination of a bulk sample the sample must be removed from its container. A possible respiratory hazard may be created during this process and since the danger cannot be gauged until after the analysis of the sample, all samples are to be treated with equal caution. Removal of any bulk sample from its collection/transmittal container is done under the environment of negative vacuum provided by a HEPA

Nilfisk vacuum cleaner and beneath a protective hood system. The bulk sample is removed under the hood system into a shallow rigid pan for examination. The inside of the container should be checked to determine if the sample to be analyzed is complete (i.e. no sample remains in the transmittal container) and representative.

At this stage the sample should be checked to determine if it was collected using wet methods. If the sample appears wet it should be dried in an oven. This is done because hydrophyllic fibers, such as chrysotile, etc. will absorb surface water which may in turn alter their dispersion staining characteristics. Additionally, the volumetric percentage content of chrysotile fibers will be more difficult to access because binding materials will become attached to small fibrils effectively hiding them from the microscopist. Additionally, if it is apparent that the sample is hard or rigid and may require physical abrasion to determine its internal components, the abrading should be done safely while under the Nilfisk negative vacuum system. This process will speed the analysis, protect the analyst from possible entrainment of fibers and save fragile, expensive inspection tools.

Stereo-binocular Microscopic Examination

Once the sample is dried (if necessary) and otherwise prepared it can effectively be viewed by a stereo binocular microscope. Examination should proceed first by initial inspection using lowest magnification (10x) to observe general sample features; i.e. layering, size, shape, color, etc. If desirable higher magnifications may be used to closely examine specific components or features. However, visual volumetric percentage estimations must be done at lowest magnification. In order to guard against eye strain the microscopist should develop a couple of habits; 1) The focusing mechanism of the microscope should be racked up and down, effectively bracketing the focus plane and 2) the light source should be kept at the lowest comfortable intensity position.

Visual Observation of Gross Properties

During the initial gross examination of the bulk sample it is helpful to attempt to gauge the general quantity of each apparently different component. A description of the gross appearance of the sample must be developed for the bulk sample analysis report (a copy is included in the manual). This is done by observing the sample's general color, listing the textual descriptions of observed components in the order of their decreasing occurrence and identifying any surfacial coating or layer. For example a painted ceiling tile which is composed of pressed ~~mineral wool~~, perlite and some powdery binder materials may be described as "light brown, compact fibrous to granular and powdery with paint." Any outstanding properties of the sample should be noted; for instance, if the sample is layered aluminum/paper/bitumen covered mineral wool pipe insulation the description should be "light brown fibrous and layered aluminum, light brown compact fibrous with bitumen."

The following are common physical and textural descriptions used in gross sample appearance descriptions and examples of materials they describe:

- bitumenous (bitumen)
- cemented (concrete)
- compact (paper), etc.)
- corrugated (paper, aircell insulation)
- fibrous (mineral wool, amosite, etc.)
- granular (perlite)
- gummy (latex)
- hard (floor tile, concrete, etc.)
- platy (vermiculite)
- powdery (binder material, plaster, etc.)
- resilient (floor tile)
- resinous (resin)
- soft (styrofoam)
- vacuous (expanded glass)
- woven (cloth)

Common surfacial coatings or layers are: latex, paint, dust, aluminum, bitumen and plaster.

Volumetric Percentage Estimation

Due to the complexity of visually estimating the volumetric percentage of a particular component in a bulk sample GES utilizes the expertise of geologists/mineralogists in this work. Geologists are taught during their education how to estimate the component percentages in rock samples. In fact several classification schemes for sedimentary rocks are based on their percentage content of certain minerals. In order to reliably estimate percentages it is necessary for the beginning bulk sample analyst to view many samples with the supervision of an experienced microanalyst. During this training the most important aspect is determining accurately the smallest percentages of asbestos content (i.e. trace to 2 percent).

Note: Only careful supervised training and practice examining bulk samples of known content can develop the visual estimation abilities of a microanlayst. In order to guarantee our quality GES protocol requires the microscopic checking of every bulk sample analysis by at least two microanalysts.

BULK SAMPLE COMPONENTS

I. Asbestiform Components

CHRYSOTILE – $Mg_3Si_2O_5(OH)_4$ – White to yellow-green, to green, fibrous in bundles (asbestiform), fresh unabrated bundles display darker colors, pearly to silky luster – quite distinctive. Index of refraction not as high as other asbestiform minerals, thin abraded bundles display wavy, silky, long fibrils with extreme flexibility along c-axis. Fibrils occur in bundles of all sizes and shapes, scraping and abrasion along length of thick bundles creates matted bundles of smaller groups of fibrils fibers are hydrophyllic in nature.

Optical Properties – n_d(liq.) = 1.550. Transparent to nearly opaque. Strongly silky luster. Dispersion staining colors – magenta parallel to blue – light blue, perpendicular. Positive sign of elongation. Thin bundles of fibers often display kinks perpendicular to length when mounted and viewed with PLM.

Occurence in Bulk Samples – Chrysotile can occur in every type of bulk sample. Commonly found as the only asbestiform mineral in bitumen, floor tile, latex and plaster samples. Highly micaceous sprayed on ceiling materials typically contain only chrysotile as an asbestiform mineral component, although rarely amosite may be a component.

Distinguishing Characteristics – silky luster, wavy, extremely flexible fiber shape often displaying kinking in mounted slide and strong affinity for water which allows encapsulants (latex) and binders (calcite and gypsum) to adhere to fibers effectively coating them.

AMOSITE(var. Grunerite) – $(Fe^{+2}, Mg)_7Si_8O_{22}(OH)_2$ – white to golden brown to greenish brown, fibrous as bundles of fibrils (asbestiform), adamantine to vitreous luster, index of refraction is higher than chrysotile. Thin abraded bundles of fibrils display straight, long parallel groups of fibrils of various lengths. Fibrils are always straight, under medium stresses perpendicular to the c-axis the fibrils display flexibility and will regain their original shape as stress subsides. Fibrils cannot be bent as chrysotile (subsequently fibrils do not display kinking) but instead display a parting perpendicular to the c-axis under extreme stress in that direction. Caution should be shown when abrading bundles of amosite, due to its inability to withstand extreme stress along the c-axis parting and splintering will result and tend to entrain short thin shards of the original bundle. Fibers are hydrophobic in nature.

Optical Properties – n_D(liq.) – 1.680 – Transparent to nearly opaque. Adamantine to vitreous luster. Dispersion staining colors – gold parallel to blue perpendicular. Positive sign of elongation.

<u>Occurence in Bulk Samples</u> - The occurrence of amosite is not restricted to any specific type of bulk sample although it most commonly occurs in pipe, joint and tank insulations. It also occurs in ceiling tiles with similarly colored mineral wool which complicates identification. Amosite typically does not occur in floor tile, latex, or plaster, and uncommonly occurs in corrugated or woven insulation and highly micaceous sprayed-on ceiling materials.

<u>Distinguishing Characteristics</u> - straight, thin, long, and light colored bundles of fibers, high luster. Distinguishable from mineral wool and fiberglass by anisotropicity, positive sign of elongation and by morphology, as parallel groups of very fine fibers. Distinguished from chrysotile by luster, shape of fibers and index of refraction, and from crocidolite by color in plain light and sign of elongation.

CROCIDOLITE - $Na_2Fe_3^{+2}Fe_2^{+3}Si_8O_{22}(OH)_2$ - blue gray to dark blue, fibrous as bundles of fibrils (asbestiform), vitreous luster. Fibrils typically long and straight with physical properties similar to amosite. Fibers are flexible along C-axis and also exhibit a parting perpendicular to the c-axis. <u>Extreme caution</u> should be shown when handling crocidolite fibrils or bundles because the mineral is generally considered to be the most carcenogenic of the asbestiform minerals occurring in bulk samples.

<u>Optical properties</u> - n_D(liq.) - 1.680. Opaque to translucent vitreous luster. Dispersion staining colors yellow to pale yellow, parallel toperpendicular negative sign of elongation.

<u>Distinguishing Characteristics</u> - Blue color, vitreous luster, negative sign of elongation. Not typically confused with other asbestiform minerals or fibrous non-asbestos materials.

<u>Occurrence in Bulk Samples</u> - Crocidolite is the <u>least common</u> of the three most commonly occurring asbestiform minerals. It is most commonly found as a component in pipe, joint and tank insulations. The mineral almost never occurs in other materials (i.e. floor tile, plaster, latex ceiling tile, etc.)

<u>II. Other Asbestiform Minerals</u>

Several other minerals may occur in bulk samples. These include actinolite, tremolite, and anthophyllite. Important information related to the optical properties of these and other minerals is included in the following table of mineral data. Other common minerals are antigorite and its fibrous (asbestiform) variety <u>picrolite.</u> Picrolite is commonly found with chrysotile; the occurence is related to an original geological mineral assemblage.

III. Fibrous Non-Asbestos Components

MINERAL WOOL - Pneumatolytically formed glass fibers made from whole rock glass melt. Dark brown, green to light brown to white, colorless. Occurs as sickle shaped, straight, curved fibers of varying thicknesses. Fibers typically display attached glass globule(s) at one or both ends and sometimes large quantites of dark colored glass beads (glass globules which have broken off the ends of fibers). May be confused with fiberglass or amosite with stereo microscope. Mineral wool is isotropic and thus distinguishable from amosite. Mineral wool displays gold colors under dispersion staining examination mounted in $n_D$liq) 1.550 as opposed to fiberglass which displays vivid blue colors. Mineral wool can display many different refractive indexes.

FIBERGLASS - Extruded fibers made of SiO_2 rich glass. Occurs as white to colorless fibers which are always straight and exhibit cylindrical cross sections and uniform thicknesses, commonly occurring as columnar groups. Distinguished from amosite by uniform habit and its isotropic nature and from mineral wool by its vivid blue dispersion staining colors in n_d(liq) 1.550. Typically found as reinforcing fibers for aluminum coverings, tape, etc.

CELLULOSE - Plant fibers. Dark brown to white, flat, ribbon shaped fibers. Distinguishable from other fibers by shape and dispersion staining colors in h_D(liq.) 1.550 which are blue - parallel to yellow perpendicular. Occurs as single fibers, woven fibrous groups or as irregularly shaped wood shavings.

POLYESTER (and polyamine) - synthetic organic fibers. Occur as long cylindrical fibers of various colors and sizes. Fibers are flexible and rarely straight (some fabrics display straight fibers). Found in many types of bulk samples.

ANIMAL HAIR - Typically horse or cattle hair. Occurs as white to dark brown (color typically zoned light to dark from base to tip) cylindrical fibers with scaly surfaces and one pointed end. Distinguishable by shape and color zonation (pigmentation).

SPIDER WEB - Fine white, fibrous, silk, luster similiar to chrysotile. Distinguishable from chrysotile by fiber shape i.e. web silk are radial threads and do not occur in asbestiform groups nor do they display kinking of fiber groups. Dispersion staining colors in n_D (liq) = 1.550 <u>very similar</u> to chrysotile i.e. magenta to blue.

IV. Non Fibrous Components

MICAS (typically vermiculite and biotite) - Both biotite (brownish-black, and vermiculite (light brown to brownish gold) occur in bulk samples. Both are platy and display a pearly luster. Typically biotite occurs in samples with a high volumetric percentage of chrysotile. Vermiculite often occurs as a major constituent of some spray applied ceiling materials and in some paints.

PERLITE - Micro-vesiculed expanded glass (as opposed to expanded glass which is macro-vesiculed). Typically occurs as white to colorless, granular, spheroidal globules which are differientiated from styrofoam by rigidity. Perlite when crushed between tweezers breaks noisily whereas styrofoam yields to the pressure and rebounds to its original shape. Found as a component of spray applied ceiling materials and ceiling tiles.

AGGREGATE/SAND Multicolored sand -sized particles of quartz, feldspar, calcite, etc.

BITUMEN Black, sticky, and shiny tar substance. Commonly contains chrysotile. Occurs as a coating on many types of pipe run insulation roofing materials and as a mastic for floor tile.

STYROFOAM Expanded synthetic organic material (as a solidified foam). Occurs as white, spheriodal to irregularly shaped particles which yield to surface pressure plastically.

V. Binders

CALCITE Occurs as a white to light brown, fine powder. Identified by reaction with HCl and high birefringences displayed under crossed nichols. Found in many types of bulk samples.

GYPSUM Occurs as a white to light brown, fine powder. Identified by and its low birefringences under crossed nichols. Found in many types of bulk samples.

CEMENT Occurs as a light grey to white hard silicate binding material in concrete aggregates. Typically mixed with fine sand.

GLUE Occurs as a transparent to opaque material of various colors and thicknesses. Displays resinous luster and fracturing due to brittle nature and contraction while drying.

DYE Typically occurs as droplets of pink and orange pigment on fibers of mineral wool. Also occurs as pigment in several types of samples.

COAL DUSTS Most commonly occur as a fine black powder on the external surface of pipe; joint, tank and other insulations which were originally located in coal burning boiler rooms.

RESINS Occur as formed tiles (floor tiles) and table tops. Color varies according to use. Displays resinous luster and occasionally a concoidal fracture. Typically medium-hard and resilient.

PAINT Occurs in various colors and is typically layered, powdery and located on the external surface of a bulk sample.

LATEX Occurs most typically as a white, soft, gummy and pliable material. Usually found as a layer on the external surface of a bulk sample. An extremely difficult material from which to isolate fibrous components. Some latex may have been heated to extreme temperatures prior to installation; this would radically alter the optical properties of any included fibers.

VI. Problematic Bulk Sample

CEILING TILE Typically compact fibrous to granular or powdery with a painted surface (display). Common compounds are mineral wool, cellulose, perlite, calcite, paint and uncommonly chrysotile and amosite. Common types are 1) predominantly cellulose (dark brown) 2) predominantly mineral wool and 3) mixtures of cellulose mineral wool and perlite in varying proportions. Types 2 and 3 most commonly contain asbestiform minerals with 2 more likely to contain amosite than chrysotile and 3 more likely to contain chrysotile than amosite. Identifictaion of amosite in ceiling tile is difficult because it most commonly occurs as isolated bundles (inhomogeneous mixing), in small volumetric percentages and may closely resemble the color of the matrix mineral wool. Because of these problems ceiling tiles with large volumetric percentages of light colored mineral wool should be very carefully analyzed for amosite prior to reporting no asbestos content. Careful analysis includes cutting sample into small sections while searching for cut-resistant fibers (asbestiform minerals), mounting of several representative clumps of fibers and analysis of all the sample (because of heterogeneity).

PLASTER Typically white (or other light color) powdery with fibers and may display a painted surface. Common components are calcite, gypsum, cellulose, mineral wool and uncommonly chrysotile. Chrysotile fibers may be difficult to isolate due to low volumetric percentages therefore samples must be checked thoroughly before a final decision is made.

BITUMEN Typically black, sticky, tar with fibers. Common components are chrysotile, mineral wool and cellulose. Difficulty may arise attempting to isolate chrysotile fibers due to color of bitumen or occurrence of fibers in low volumetric percentages. Occasionally chrysotile fibers may be boiled with bitumen prior to application to insulations, the resulting surfacial chemical weathering on chrysotile fibers will affect their optical properties. These fibers can still be identified by their characteristic shape. Mounting of suspect fibers in n_D = 1.550 fluid presents no unusual problems because the bitumen is soluble in the R.I. fluid and does not mask dispersion staining colors.

DUST Typically is a mixture of both fibrous and non-fibrous particulates. Dust is likely to contain any component. Common components are cellulose, polyester and fine particulate grains. Because the composition of dust is so highly variable analysis of a sample is difficult. The difficulty is compounded by the typically small sample size. Isolation of suspect fibers is a long and tedious process which is necessary because the sample displays no compositional homogeneity.

LATEX Typically is white gummy with fibers. Common components are mineral wool, cellulose and chrysotile. An extremely difficult material from which to isolate fibrous components due to its exceptional binding properties. To isolate fibers sections of latex must be cut and fibers must be scraped meticulously to obtain desirable mounting specimens. Samples must be carefully analyzed to determine both fiber content and composition of component fibers. Component chrysotile fibers may be chemically and physically altered and display uncharacteristic optical properties. These fibers must be identified by morphology and habit.

FLOOR TILE Typically hard resilient and resinous with aggregate, fibers and surface mastic. Tiles often display marbled appearance. Common components are fine aggregate, cellulose and chrysotile. Due to the tough resilient nature of these tiles they must be examined using heavy duty tweezers and knives. Isolation and location of suspect fibers require cutting, pulling apart of cut sections and careful examination of the cutting interface for protruding fibers. Fine tweezers may then be used to pluck suspect fibers from cut tile sections for mounting and polarized light microscopical examination.

RECORDING OF BULK SAMPLE DATA

Copies of all bulk sample analysis reports are kept on file by GES job number. In addition, the central bulk sample log contains not only log in data but all analysis results. All original bulk samples are stored in photographic or pharmaceutical vials for future reference. Bulk samples are only returned to original clients upon written request and completion by the client of an indemnification form. A copy of this form is included in this manual.

REPORT PREPARATION

GES protocol requires that a short cover letter accompany the reporting of bulk sample analys(es). A bulk sample report will include all important data derived from the sample using both stereoscopic and polarizing light microscopic methods. Additionally, as an internal quality control check, all sample analyses are checked for a second time by an additional microanalyst. An example of a cover letter is included in this manual.

MINERALOGY AND CRYSTALLOGRAPHY

The purpose of this section is to acquaint you with the basic concepts of mineralogy and crystallography. Asbestos minerals, and most of the materials used in asbestos containing building materials, are minerals. It is therefore useful to understand the basics of mineralogy. The optical properties of minerals are related to the crystalline structure of the mineral. Therefore a thorough knowledge of crystallography is important to understanding the optical properties of minerals which enable us to identify them.

What is a mineral? It is a naturally occurring homogeneous solid with a definite (but generally not fixed) chemical composition and a highly ordered atomic arrangement of its chemical components. Minerals are generally formed by inorganic processes. Minerals are classified and identified based on their physical properties, chemical properties, and crystalline structure. Optical properties of minerals are the physical characteristics of most importance to the PLM analyst. Mineral optics will be discussed in the section covering PLM and asbestos mineralogy. Many excellent text covering the properties of minerals have been written. It would be useful for you to read the descriptions of asbestos minerals in these text as a supplement to the information presented in this course.

PHYSICAL PROPERTIES OF MINERALS

Physical properties are those properties which can be observed or measured without chemically changing or altering the mineral. A field geologist acquainted with physical properties may be capable of recognizing hundreds of mineral species on sight. The following list and descriptions gives some of the most useful physical properties of minerals:

Crystal form - all minerals possess an orderly internal arrangement of constituent atoms. This often leads to a regular external geometric form which is characteristic of the mineral species.

Crystal habit - Typical appearance of single crystals or crystal aggregates. Typical crystal habits are:

- acicular - needle-like crystals
- bladed - elongated, flattened crystals, like a knife blade
- fibrous - aggregates of slender fibers, they maybe parallel or radiating.
- micaceous - occurring as stacks of sheets of mineral
- massive - occurring as a compact mass with no distinct crystals

Cleavage - The bond strengths of some minerals may be weak in one or more planes of the crystal structure allowing the mineral to break or cleave in a flat plane. The cleavage may be well or poorly developed. Combinations of several different cleavage directions may yield a regular geometric form on broken fragments which may resemble a crystal.

Fracture - A mineral without cleavage exhibits fracture when it breaks. The fracture maybe uneven or irregular or may result in smooth curved surfaces called conchoidal fracture.

Hardness - the resistance of a mineral to abrasion by a substance of known hardness. Hardness is measured on a scale of 1 to 10 with 1(talc) being the softest and 10 (diamond) being the hardest.

Optical Properties - Those properties dependant on the interaction of the mineral with light. The microscopic optical properties will be discussed in the PLM section. Some of the gross optical characteristics include the following:

- Color - This refers to the color of the mineral when observing a mineral with the unaided eye. A thin mineral fragment may transmit a very different color when observed through the microscope.

- Luster - Refers to the general appearance of the mineral. A mineral may have a metallic or a non-metallic luster. The non-metallic minerals may be further subdivided into dull (or earthy), vitreous (or glassy), iridescent, or resinous.

<u>Magnetism</u> - The characteristic of a mineral which causes a mineral to be attracted to a magnet or act as a natural magnet. This can be determined with the microscope by passing a small magnet close to the objective while observing the specimen being tested. A magnetic mineral will move as the mineral is moved.

There are many other physical characteristics, but these will be of most use to the asbestos analyst.

CHEMICAL MINERALOGY

Minerals may be classified based on their chemical constituents as determined through chemical analysis. Chemical analysis is a destructive process, that is the sample tested is chemically altered during the test. The physical properties of minerals are due to the chemical elements present in the mineral as well as the arrangement of those constituents and the strength of bonding between each element.

The chemical composition of a mineral may be fixed (e.g. quartz SiO_2) or may vary within certain bounds. The variation in chemical composition is possible because of a phenomena known as solid solution. Solid solution occurs when chemical elements of similar size an electrical properties exchange for one another in the same space in the crystal lattice of the mineral. This is an important concept to the asbestos analyst because solid solutioning is common in the amphibole family to which 5 of the 6 asbestos minerals belong. This may explain the difference in chemical formulas for identical minerals. it also explains slight variations in optical properties for the same mineral, as slight changes in chemical composition yield noticeable changes in the optical characteristics.

Minerals are grouped according to the major chemical characteristics. The following is a list of the major chemical mineral groupings. Examples of minerals important to the asbestos analyst are listed where appropriate.

Native elements - Minerals found in nature in their pure elemental form.

Sulfides - Minerals formed by a combination of a metal and sulfur. Pyrite (FeS_2) is the most common.

Oxides - Minerals formed by the combination of oxygen and one or more metals. Magnetite (Fe_3O_4) is ubiquitous to chrysotile deposits. Hematite (Fe_2O_3) is used as a red pigment (coloring material) and synthetic Rutile (TiO_2) is used as a white pigment.

Halides - minerals formed by combining a metal with a halogen element, that is Chlorine, Fluorine, Bromine, or Iodine. Rock Salt or halite (NaCl) is the most common example.

Carbonates - Minerals formed from the combination of a metal(s) and a carbonate complex (CO_3) Calcite ($CaCO_3$) from limestone and marble deposits is widely used as a filler material or a bonding material (as with portland cement) in many asbestos containing building materials.

Sulfates - Minerals formed by combining a metal with the sulfate complex (SO_4). Gypsum ($CaSO_4 \cdot 2H_2O$) is widely used as filler (as with gypsum wall board) or as a cement.

Silicates - Silicates are the most abundant minerals. They are formed from combinations of various metals with silicon and oxygen. The basic structural building block of the silicate minerals is the silica tetrahedra. It is formed by combining 4 oxygen atoms with 1 silicon atom. Silicate minerals are further subdivided based on the number and arrangement of these silica tetrahedra. Figure 10-1 shows diagrams of each subclass. The following are descriptions of each subclass:

- Nesosilicates - Also called the "island silicates," are composed of isolated silica tetrahedra.

- Sorosilicates - Also called the "bow-tie silicates" are composed of pairs of silica tetrahedra.

- Cyclosilicates - These are composed of silica tetrahedra linked in a circle.

- Inosilicates - these are composed of silica tetrahedra linked end-to-end forming chains. They are therefore called the chain silicates. The chains may be single chains or double chains, two parallel chains of tetrahedra linked together. The amphiboles belong to the double chain silicate group.

- Phyllosilicates - these minerals are composed of a two-dimensional array of silica tetrahedra which forms sheets. These sheet silicates are also of importance to the asbestos analysis as this is the group to which the serpentine (chrysotile) minerals belong. When serpentine minerals form, the sheets may roll into fibers and become asbestiform. Micas, vermiculite, clays,and talc (fibrous and platy) are other minerals in this group.

- Tectosilicates - these are structurally complex silicate minerals formed of three dimensional networks of silica tetrahedra. Quartz and feldspars are tectosilicates commonly found in asbestos containing building materials. They are used as filler material.

CRYSTALLOGRAPHY

Crystallography is one of the two most important interrelated disciplines used in asbestos identification. Crystallography describes the internal order of a mineral. Mineral optics are related to this internal order of the mineral. Therefore an understanding of crystallography is critical to fully comprehending mineral optics.

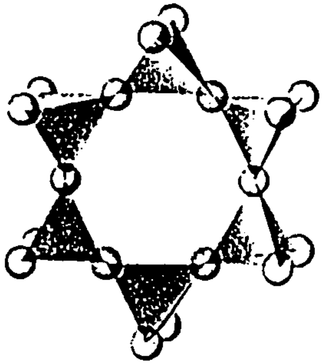
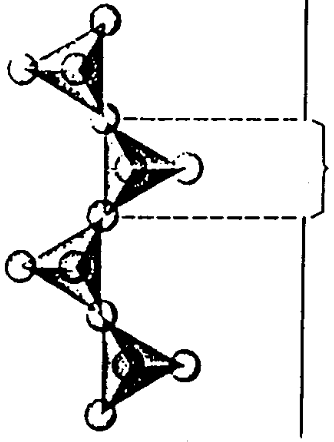
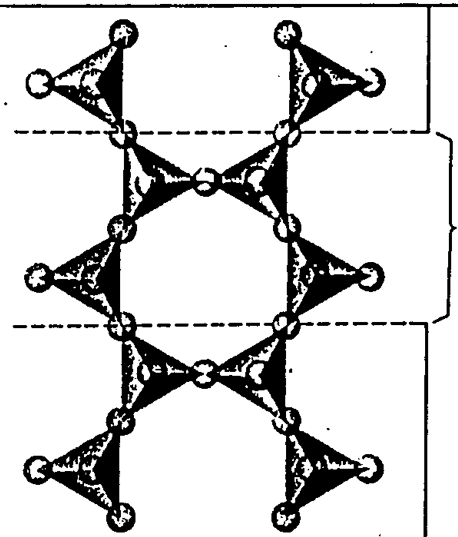
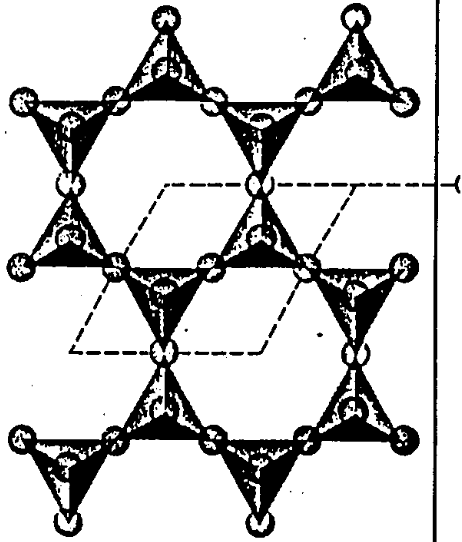
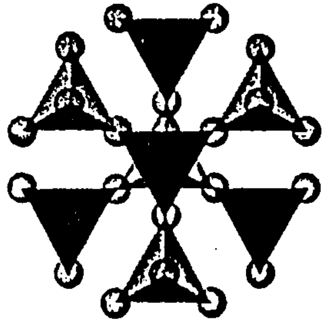
The internal arrangement of constituent elements/molecules is expressed in an external, regular geometric form. This form, the crystal form, is the same for each crystal of the same mineral. The external form is an expression of the repeated linking of countless microscopic building blocks known as unit cells. The unit dell is the smallest combination of constituent atoms which still contains the basic structure of the mineral. The unit cells are responsible for the crystal shape and habit, cleavage, and optical properties. The unit cells connect in preferred directions yielding a regular crystal form. The bond strength between unit cells may be different in different directions yielding cleavage. The optical properties may vary in certain orientations. The optical density or refractive index may be higher or lower in one direction than in another. some wavelengths of light may be absorbed in some directions and not others yielding color.

Minerals, by definition must possess an orderly internal structure, that is they are crystalline. A solid which has a chaotic internal structure in called a glass.

Crystals may be divided into six crystal systems. These systems can be defined based on geometric relationships of three crystallographic axis (four for the hexagonal system). A crystallographic axis is an imaginary line from the center of the crystal to the outside. The axes are parallel to the major or principal faces of the unit cell. The relative lengths of the axes are also important. The lengths of the axes are ratios of the dimensions of the unit cell of the mineral.

Figure 10-1

Silicate Mineral Subdivisions

Class	Arrangement of SiO_4 tetrahedra (central Si^{4+} not shown)	Unit composition	Mineral example
Nesosilicates		$(SiO_4)^{-4}$	Olivine, $(Mg, Fe)SiO_4$
Sorosilicates		$(Si_2O_7)^{-6}$	Hemimorphite, $Zn_4Si_2O_7(OH)\cdot H_2O$
Cyclosilicates		$(Si_6O_{18})^{-12}$	Beryl, $Be_3Al_2Si_6O_{18}$
Inosilicates (single chain)		$(SiO_3)^{-2}$	Pyroxene e.g. Enstatite, $MgSiO_3$
Inosilicates (double chain)		$(Si_4O_{11})^{-6}$	Amphibole e.g. Anthophyllite, $Mg_7Si_8O_{22}(OH)_2$
Phyllosilicates		$(Si_2O_5)^{-2}$	Mica e.g. Phologopite, $KMg_3(AlSi_3O_{10})(OH)_2$ Serpentine $Mg_3Si_2O_5(OH)_4$
Tectosilicates		$(SiO_2)^0$	High cristobalite, SiO_2

The Six Crystal Systems

Atomic arrangements in the different crystal systems are diagramed and described in Figures 10-2 thru 10-7.

The atomic arrangement in solid materials is obviously very important since it explains, first, why different crystalline materials have different shapes and, second, why and how the optical properties vary in different crystals.

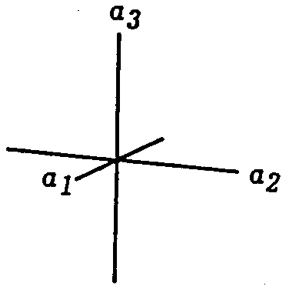


Figure 10-2Cubic Crystal System. Identical spacing of atoms along each of three mutually perpendicular directions a_1, a_2, a_3; isotropic, i.e., all refractive indices identical.

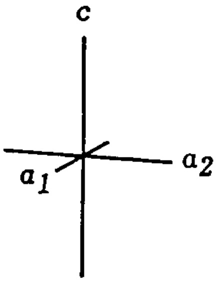


Figure 10-3 Tetragonal System. Three mutually perpendicular axes, two with identical spacings of atoms but greater or less than the spacing along the c axis; two principal refractive indices: ϵ parallel to c, ω for all directions in the a_1-a_2 plane perpendicular to c.

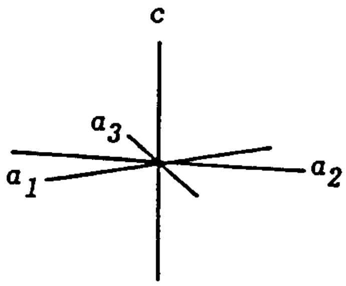


Figure 10-4 Hexagonal System. Three axes (a_1, a_2, a_3,) 120° apart in the same plane with identical spacings greater or less than the spacing along the c axis perpendicular to that plane; two principal refractive indices: ϵ parallel to c, ω for all directions in the a_1-a_2-a_3 plane.

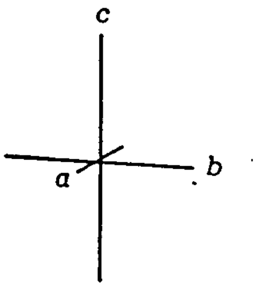


Figure 10-5 Orthorhombic System. Three different spacings along three mutually perpendicular directions (a, b, c); three principal refractive indices: α, β and γ each parallel to any one of the three axes: a, b and c.

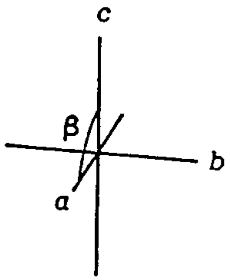


Figure 10-6 Monoclinic System. Three different spacings along three different axes (a, b, c); a is inclined to c but b is perpendicular to the a-c plane; three principal refractive indices: α, β and γ; any one of these indices is parallel to b, the other two are in the a-c plane.

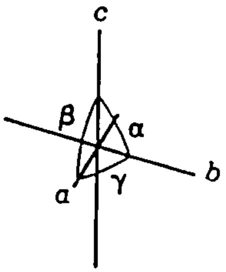


Figure 10-7 Triclinic System. Different spacings along three mutually oblique axes (a, b, c); three principal refractive indices: α, β and γ, all oblique (close to parallel occasionally, but always oblique) to all crystallographic axes.

POLARIZED LIGHT: A BASIC THEORETICAL UNDERSTANDING

Light has the nature of both a particle and a wave. In ordinary light, the wave nature is random, vibrating or oscillating in a cylinder whose radius is the amplitude of vibration and whose long axis is the direction of propagation of the light. The period of one full vibration or oscillation is called the wavelength and is measured along the direction of propagation. If one restricts the vibration direction of light so that the sides of the cylinder area pared down and a flat ribbon is all that remains, then the light is said to be plane-polarized (having definite poles of movement) and the plane of the ribbon perpendicular to the line of propagation is said to be the 'privileged' direction.

Any material that produces this effect is called a polarizer. Minerals polarize light, as do reflective surfaces (mirrors, sheets of glass, media boundary layers, calm water) and carefully grown organic crystals in sheet form called Polaroid® polarizing filters. Before Polaroid® sheets were common, carefully ground prisms, called Nicol Prisms, produced polarized light for commercial polarizing microscopes. In many articles and texts still in use today, crossed (opposed) polarizers (or crossed polars for short) are referred to as crossed Nicols.

Crossing polars 90° theoretically blocks out all transmitted light. This is nearly true, and a pair of polarizers at 90° will produce a very nearly black image. When observed through a polarizing microscope, this is even called 'blackfield' viewing. If the polars are not placed in opposition, some light is transmitted. Out of convention, the lower polar (nearer the light source) is called the polarizer and the upper (nearer the analyst) is called the analyzer. The polarizer transmits all light and all components of light vibrating in its privileged direction. Because of this effect, inserting a polarizer between two opposing polarizers will produce transmission, provided the privileged direction of the polarized inserted is not parallel to either the polarizer or the analyzer.

MINERAL OPTICS: A BASIC THEORETICAL UNDERSTANDING

The crystallography of a mineral will determine how light will interact with a transparent mineral. Light traveling unimpeded through a vacuum has a velocity (c) of 3×10^{11} centimeters per second. The velocity of light encountering any substance will be reduced. The ratio of c in a vacuum to c in a particular medium is called the refractive index, or n. All optically transparent media have at least one refractive index, some have two and still others, three principal indices. The orientation of the refractive indices are intimately, indeed inseparably associated with crystal structure and the density of the medium. All of the principal refractive indices of a multi-indexed material are mutually perpendicular. The refractive indices and there location in the material can be represented by a three-dimensional figure called the indicatrix.

When a crystalline substance has more than one refractive index, it acts as a polarizer, splitting light into two or more components. This property becomes important in the field of polarized light microscopy where the degrees of such phenomena are unique to individual minerals, and once measured can be positive tools in the identification of minerals and crystalline substances.

A material which has only one refractive index is said to be isotropic. If the substance is isotropic then the indicatrix is a sphere. Such a substance is either a liquid, a glass (having no regularly repeating atomic arrangement) or a solid crystallographically arranged in such a fashion that no direction of travel for light is any "faster" than any other direction (known as the isometric or cubic system in crystallography). Common table salt is a crystalline substance in the cubic system, and is isotropic.

In a two-index, or uniaxial material, the indicatrix is called an ellipsoid, and a cross-section through its axis of elongation is a circle. In the plane of its circle are the radii representing one of the two principal refractive indices, one that is commonly called omega (ω). The direction or axis of elongation perpendicular to the plane is called the optic axis, and is a line representing the second major refractive index, or epsilon (ϵ). Looking along epsilon in a line $\perp$ to the plane of the circle, one sees only omega (ω), radiating out equally in all directions. Looking along a line parallel to the plane of the circle, one sees an ellipse with the long axis being ϵ and the short being ω. In between ϵ and ω are intermediate indices known commonly as ϵ' (epsilon primes). If epsilon is greater than omega, the substance is said to be optically positive. The indacatrix then is a prolate spheroid. Conversely, if epsilon is less than omega, the flattened ellipsoid is called an oblate spheroid and the substance is optically negative.

If a crystal has three principal refractive indices, it is called biaxial and has an indicatrix also ellipsoidal in shape, but has two circular cross sections offset at a given angle. With the indicatrix arranged in an x-y-z or cartesian coordinate system, the vertical axis, or z is gamma ( ), the largest principal index. A cross-section $\perp$ to gamma reveals a plane containing another ellipse with beta (β) the intermediate principal R.I. along the y axis and alpha (α) on the x-axis, being the least R.I.. There are two circular cross-sections, both having a radius equal to β which are angled to and intersect the y-axis. The two axes $\perp$ to these circular sections are called optic axis (hence bi-axial). The angle between these two axes (2v) is also a property that varies according to the material exhibiting it. If 2v is 90° or less, the mineral is considered biaxial positive. If it is greater than 90°, then it is biaxial negative.

The difference between the greatest refractive index and the least refractive index is called birefringence. The product of the birefringence and the material's thickness (usually measured in millimeters) is called the retardation. These two properties are valuable in identifying minerals also, and will be further investigated shortly, in the section involving polarizing microscopes.

Another property of crystals dependent on their refractive indices with respect to the axis of elongation of fibrous materials is called the sign of elongation. If the larger refractive index is parallel (or within 45°) to the long axis of the crystal, then the crystal has positive elongation (also called length-slow because light is slowed more in that direction). If the smaller refractive index is parallel (or with 45°) to the long axis of a crystal, the crystal has negative elongation (length-fast because light is slowed less in that direction).

ANALYSIS WITH THE POLARIZED LIGHT MICROSCOPE

The method used for analysis of materials with the polarized light microscope (PLM) is the oil immersion technique. With this technique, the sample is placed on a microscope slide, immersed in a liquid with a refractive index (n) close to that of the particle to be analyzed. The particle is observed and the optical characteristics of the material are determined by the system outlined below. Once the optical characteristics are known, they are compared to reference values to identify the particle.

If the particle has only one refractive index (isotropic), identification is trivial. The particle is a glass or a mineral which forms cubic crystals (isometric), and identification can be made based on color and refractive index.

If the particle has more than one refractive index (anisotropic) then it will exhibit optical properties unique to certain orientations of the material. These characteristics, and their orientations are directly related to the chemical composition and the crystalline structure of the material. In isometric minerals, the crystal structure is equally developed in all directions. Consequently, there are no unique orientations for cubic crystals. In other crystalline structures, the packing of constituent chemical ions varies in different crystal orientations. The optical properties have maximum variation parallel to the crystal structure of the material. In ordinary non-polarized light, which has no preferred orientation, these different optical properties occur simultaneously and are therefore cancelled out or averaged. By inserting a polarizing filter, light can be polarized. Polarized light is unidirectional. It travels in a plane, with all light rays vibrating in the same direction. This is extremely useful because polarized light allows observation of one optical property at a time.

To do this the material is oriented so that the optical property to be measured is oriented parallel to the vibration direction of the polarizing filter. For instance, in order to measure the refractive index perpendicular to the length of a fiber, the fiber is oriented so that the long axis of the fiber is perpendicular to the privaleged direction of the polarizer.

The absorption color of anisotropic minerals may vary in different orientations. This means that the color of a mineral will change as its position is changed with respect to the polarizer. This property is called pleochroism and is diagnostic for certain minerals (see figure 11-1).

An anisotropic mineral splits plane polarized light into doubly polarized light with vibration directions paralleling maximum refractive index differences and at 90° to each other. If a second polarizer is placed in the microscope above the specimen, with its vibration direction perpendicular to the vibration direction of the polarizer, the background appears black. If the optic axes of the mineral parallels the vibration directions of the two opposing polarizing filters,it too will appear black. This phenomena is called extinction. This occurs at four possible orientations of the specimen, all at 90° to each other and parallel to a principle refractive index. If, however, the specimen is oriented at some other position, it is visible. Why? The doubly polarized light from the specimen is resolved or recombined into visible light by the second polarizing filter. This visible light is colored, and the color is a function of the birefringence (difference between the two refractive indices) and the thickness. The colors are referred to as interference colors, which indicate the retardation (or slowing) of light measured in nanometers. The greater the retardation, the paler the interference color.

If the principle optic axis of the mineral are parallel to the axis of elongation, the mineral will be extinct in crossed polarized light when the fiber is parallel to the vibration directions of the polarizing filters. This is called parallel extinction. If the principle optic axes of the mineral are not parallel to a crystal axis, then the mineral will be extinct at an angle to vibration directions of the polarizer or analyzer. The angle between the extinction position and vibration direction of the polarizers is called the extinction angle. Minerals exhibiting parallel extinction have a 0° extinction angle.

In a fibrous or elongated mineral it is useful to know whether the refractive index parallel to (or within 45° of) the length of the fiber is larger or smaller than the refractive index parallel to (or within 45° of) the width of the fiber. This property is referred to as the sign of elongation. If the n along the length of the fiber is greater, the sign of elongation is positive (+). If the n along the length of the fiber is less than the n across the width, the sign of elongation is negative (-). One way of determining this is by actually measuring the refractive indices and determine the relationship.

There is another quicker method of determining sign of elongation. The birefringence of the mineral can be increased or decreased by superimposing over the unknown mineral another crystalline substance whose optic orientations are known. The birefringence will be increased if the larger refractive index of the known material coincides with the larger refractive index of the specimen. The known material is typically a slice of the mineral gypsum ground to a thickness which will retard crossed polarized light 550 nanometers to produce a background color which is red. This accessory is known by several names: the 550nm compensator plate, the gypsum plate, or the red plate.

The long axis of the compensator is oriented in the northeast to southwest direction (also known as the 45° position) in the field of view in the polarized light microscope. The unknown fiber is oriented in the 45° position. If the fiber has a positive sign of elongation, the birefringence will be increased by the compensator plate; that is, the interference colors will become paler. If the fiber has a negative sign of elongation, the interference colors will become more intense.

MEASURING REFRACTIVE INDEX

The previous sections have detailed techniques for locating principle refractive indices (n) by finding extinction positions and even for determining which is the larger n (i.e. sign of elongation). By using only the polarizer (plane polarized lights), the analyst can isolate the refractive index to be measured. This is accomplished by orienting the mineral so that the refractive index to be measured is parallel to the privileged direction (or vibration direction) of the polarizer. Any one or combination of the following techniques can then be used to measure the n for that orientation.

There are three major techniques for measuring n: the contrast/relief method, the Becke (pronounced 'Becky') line test, and dispersion "staining". In each technique the principles involved are the same, only the observation method is changed.

The higher the refractive index, the greater the "optical density". That is, the more light will be slowed upon entering the media. If a mineral of one n is placed in a liquid with a different n, light at the mineral/liquid interface will bend toward the substance with the higher n. This effect is called refraction.

The greater this difference, the more the light will be bent or refracted. The more the light is bent, the greater the contrast of the mineral with its background. At a match of n's, light is not refracted at the interface, no contrast is produced and the edge of the grain is therefore nearly invisible (See figure 11-2). Through experimentation (by placing the unknown in liquids of known n) the n of the unknown can be determined by finding the liquid which produces the least contrast. This is the contrast/relief method.

Another technique is the Becke line method. As we have stated already, light is bent towards the substance with the greater n. This phenomena produces a thin bright line at the edge of the mineral when the focus is changed. As the distance between the unknown and the microscope objective is raised, the line (the Becke line) moves towards the substance with the higher n. To do this, first determine which direction you have to rotate the focus knob to increase the distance between object and objective. Make the change and note which way the Becke line moves. Adjust the n of the mounting medium until a match is found. If the n's match no Becke line is produced and no change in the grain boundary will occur when the plane of focus is moved.

The most common method for determining the refractive index of a material is called dispersion "staining". White light is composed of many wavelengths (colors) of light. The refractive index of a material is unique to one wavelength of light. Refractive indices for material are reported for the standard wavelength of 589 nm (sodium 'D'/orange) which falls near the center of visible wavelengths. This means that different wavelengths of light are refracted at different angles at the interface of two media of different n. White light is therefore separated into a band of colors, or spectrum. This is called dispersion. At a match of n's for two materials, the light with a wavelength of 589nm passes the interface undeviated. All other wavelengths are refracted (bent). The lenses of the microscope recombine all refracted and non-refracted wavelengths to form an image of the specimen which has the same color as the specimen. If, however, an opaque dot is placed in the microscope so that the undeviated wavelengths are blocked, then only the deviated (refracted) wavelengths will be used to form the image of the specimen. Since the deviated wavelengths are from the blue and red ends of the spectrum, that image will be purple. This phenomena is called dispersion "staining" -dispersion because it exploits the dispersion phenomena and staining because it produces color in an otherwise colorless specimen even though no 'stains' are used. The opaque dot (central stop) is at the back focal plane of the objective. To produce this phenomena, it is also necessary to use a narrow beam of white light such that the diameter of the beam coincides with the diameter of the central stop. This is accomplished by closing down the condenser diaphragm until the field of view is dark and dispersion "staining" colors are observed.

The refractive index of the liquid must be close to the refractive index of the mineral before dispersion "staining" colors will be seen. If they are a perfect match, the mineral will be bluish-purple. If the n of the mineral is close to, but less than the n of the oil the mineral will be blue or pale blue green. If the n of the mineral is close to, but greater than the n of the liquid the mineral will be reddish purple, golden purple, gold or straw yellow.

Dispersion "staining" is a quick and accurate method for determining the refractive index of small particles. The equipment (a dispersion "staining" objective) is readily available. This last technique is therefore widely used in asbestos identification.

A SUMMARY OF POLARIZED LIGHT MICROSCOPE ANALYSIS TECHNIQUES

Analysis with no polars:

- Morphology (shape))
- Aspect Ratio (if fibrous)
- Refractive index of isotropic substances
- Cleavage angles
- Color of non-pleochroic minerals

Analysis with one polar:

- Pleochroism
- Refractive Indices of anisotropic materials

Analysis with crossed polars:

- Anisotropy
- Birefringence of anisotropic materials
- Extinction angle of anisotropic materials
- Extinction characteristics (mottled, anomalous, parallel, inclined)

Analysis with crossed polars and a 550 nm compensator plate:

- Sign of elongation of fibrous materials

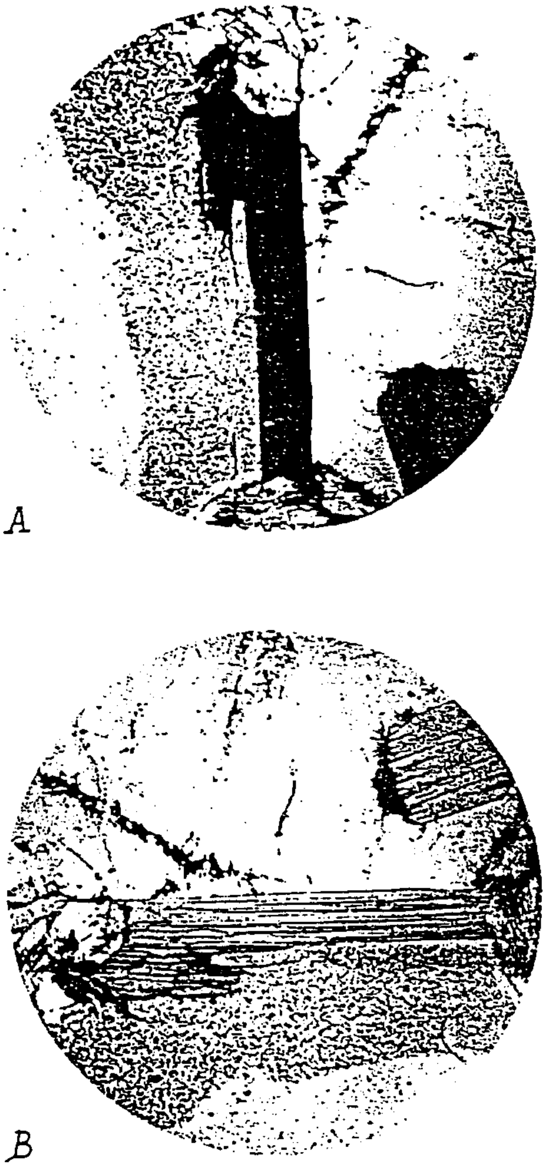


Figure 11-1. Absorption of light by biotite. (A) Biotite strongly absorbs light waves vibrating parallel to its cleavage. The analyzer is passing N-S waves. (B) Biotite weakly absorbs light waves vibrating perpendicular to its cleavage. The analyzer is passing N-S waves.

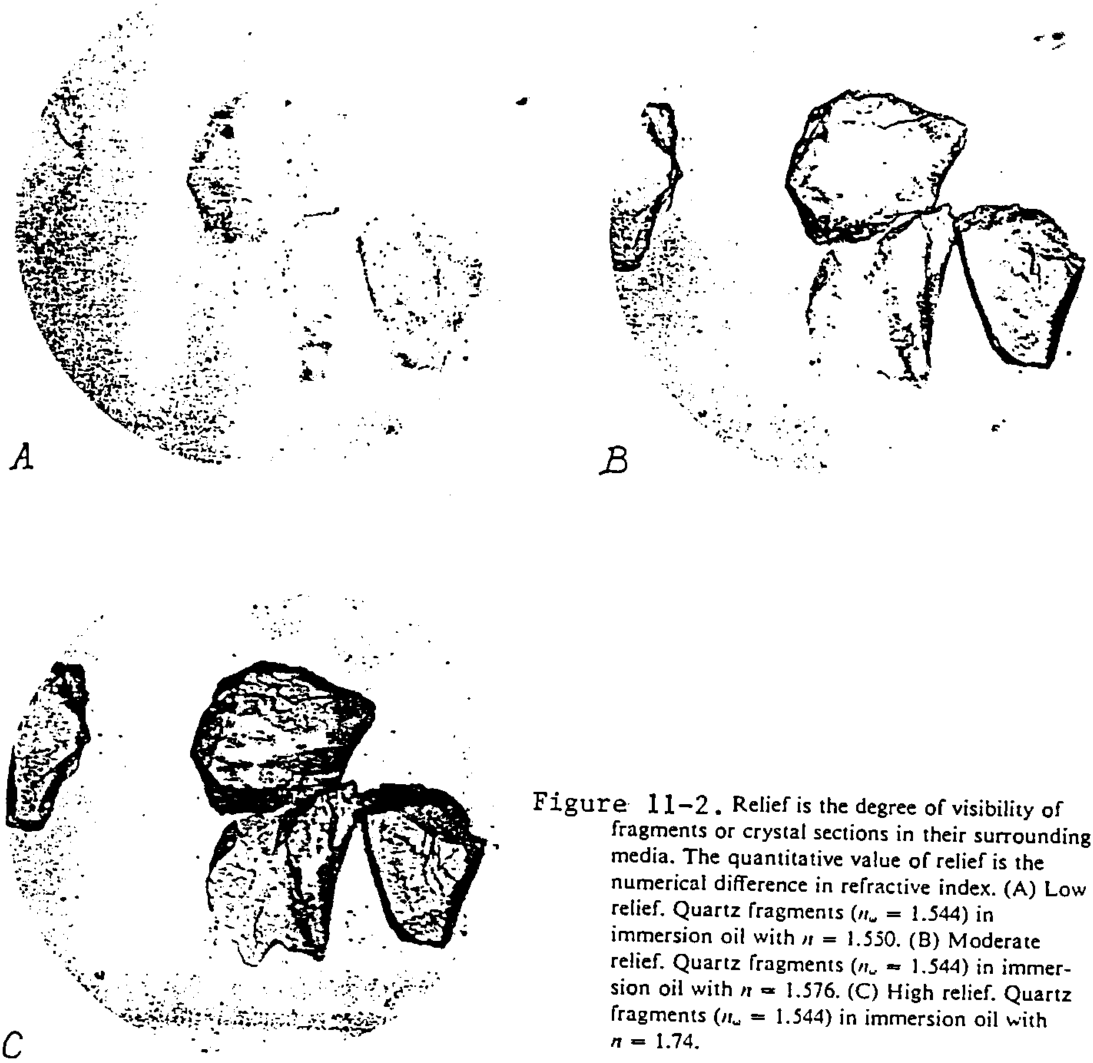
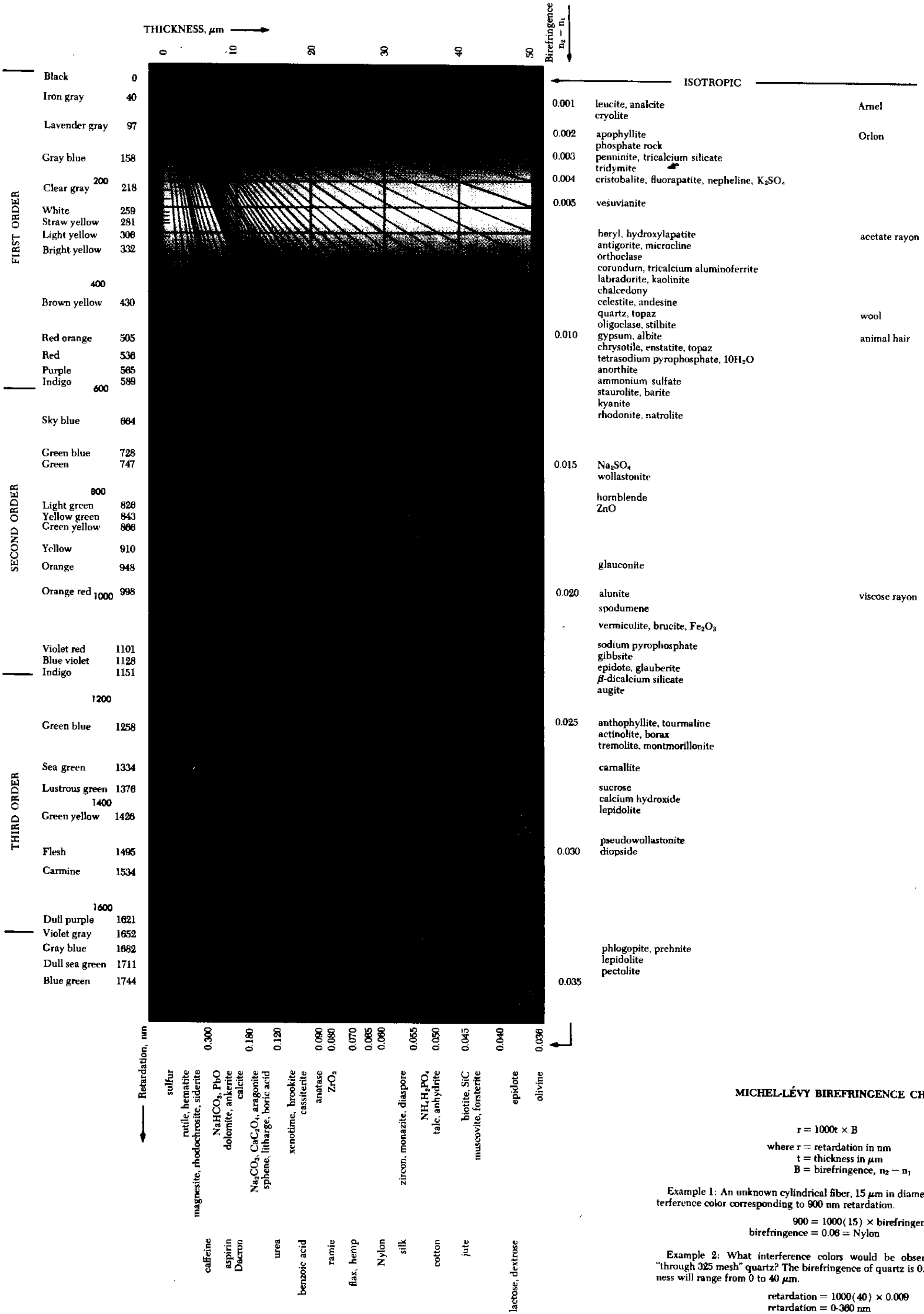


Figure 11-2. Relief is the degree of visibility of fragments or crystal sections in their surrounding media. The quantitative value of relief is the numerical difference in refractive index. (A) Low relief. Quartz fragments ($n_\omega = 1.544$) in immersion oil with $n = 1.550$. (B) Moderate relief. Quartz fragments ($n_\omega = 1.544$) in immersion oil with $n = 1.576$. (C) High relief. Quartz fragments ($n_\omega = 1.544$) in immersion oil with $n = 1.74$.



MICHEL-LÉVY BIREFRINGENCE CHART

$$r = 1000t \times B$$

where r = retardation in nm
t = thickness in µm
B = birefringence, $n_2 - n_1$

Example 1: An unknown cylindrical fiber, 15 µm in diameter, shows a yellow interference color corresponding to 900 nm retardation.

$$900 = 1000(15) \times \text{birefringence}$$
$$\text{birefringence} = 0.06 = \text{Nylon}$$

Example 2: What interference colors would be observed on a sample of "through 325 mesh" quartz? The birefringence of quartz is 0.009; assume the thickness will range from 0 to 40 µm.

$$\text{retardation} = 1000(40) \times 0.009$$
$$\text{retardation} = 0\text{-}360 \text{ nm}$$
interference colors = black, gray, white, yellow

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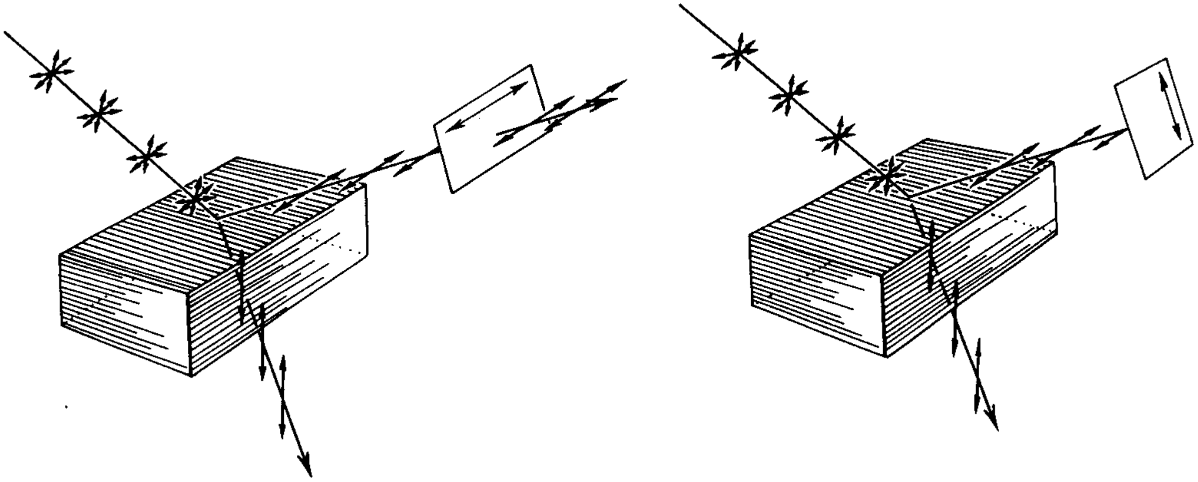


(a)

(b)

FIG. 238. Polarized light by reflection and refraction.

vibrating perpendicularly to it, is almost completely absorbed. Polarizing sheets, such as *Polaroid*, are made by aligning crystals on an acetate base. These crystals absorb very little light in one vibration direction, but are highly absorptive in the other. The light transmitted by the sheet is thus plane polarized. Because they are thin and can be made in large sheets, manufactured polarizing plates are extensively used in optical equipment, including many polarizing microscopes.

Polarized Light by Reflection. Light reflected from a smooth nonmetallic surface is partially polarized with the vibration directions parallel to the reflecting surface. The extent of polarization depends on the angle of incidence (Fig. 238) and the index of refraction of the reflecting surface. It is most nearly polarized when the angle between the reflected and refracted ray is 90° (Brewster's law). The fact that reflected light is polarized can be easily demonstrated by viewing it through a polarizing filter. When the vibration direction of the filter is parallel to the reflecting surface the light passes through the filter with only slight reduction in intensity; when the filter is turned 90°, only a small percentage of the light reaches the eye.

[redacted]

The polarizing microscope is the most important instrument for determining the optical properties of crystals; with it more information can be obtained easily and quickly than with more specialized devices. Several manufacturers each make a number of models of polarizing microscopes that vary in complexity of design and sophistication and hence in price. A student model made by E. Leitz (SM-Pol) is illustrated in Fig. 239 with the essential parts named.

Although a polarizing microscope differs in detail from an ordinary compound microscope, its primary function is the same: to yield an enlarged image of an

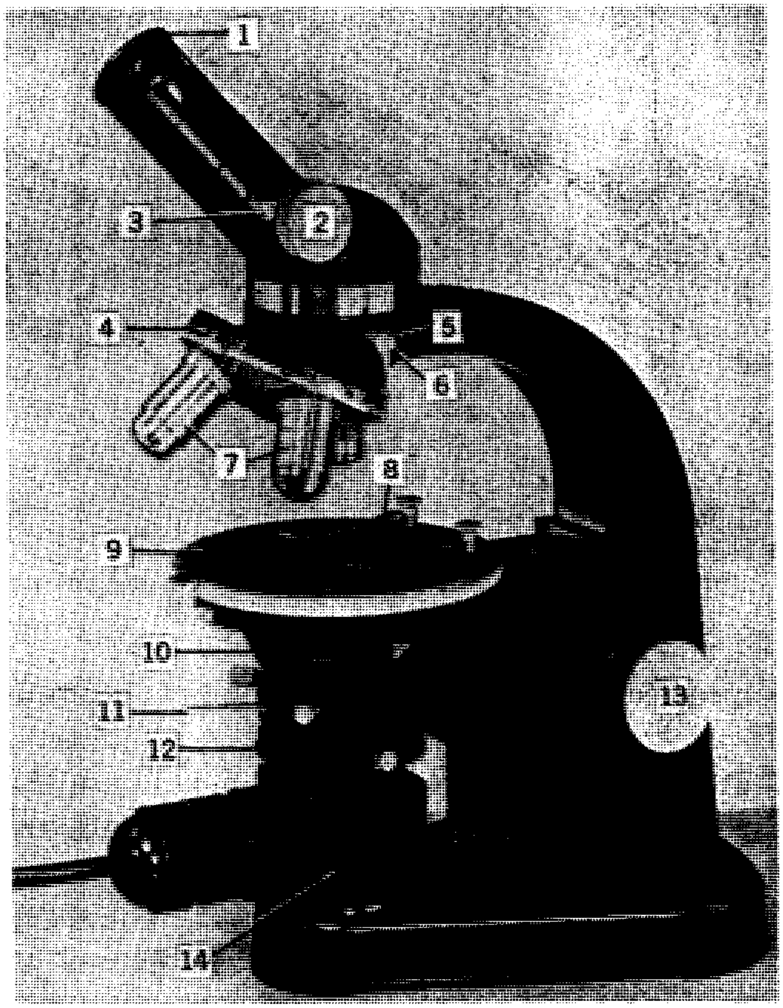


FIG. 239. **Polarizing microscope (SM-Pol E. Leitz, Inc.). 1. Ocular. 2. Amici-Bertrand lens. 3. Pinhole stop for auxiliary lens, swings out and in. 4. Revolving nosepiece for oculars. 5. Analyzer. 6. Tube slot for compensators (out of sight). 7. Objectives. 8. Knurled head for swinging out of top part of condenser (out of sight). 9. Rotating stage. 10. Condenser (behind shield). 11. Iris diaphragm. 12. Rotatable polarizer. 13. Focusing controls on both sides. 14. Substage illuminator.**

object placed on the stage. The magnification is produced by a combination of two sets of lenses: the objective and the ocular. The function of the objective lens, at the lower end of the microscope tube, is to produce an image that is sharp and clear. The ocular merely enlarges this image including any imperfection resulting from a poor quality objective. For mineralogical work it is desirable to have three objectives: low-, medium-, and high-power. In Fig. 239 these are shown mounted on a revolving nose piece and can be successively rotated into position. The magnification produced by an objective is usually indicated on its housing, such as 2x (low), 10x (medium), and 50x (high). Oculars also have different magnifications such as 5x, 7x, 10x. The total magnification of the image can be determined by multiplying the magnification of the objective by that of the ocular as: $50x \cdot 10x = 500x$. Although in routine work the three objectives are frequently interchanged, a single ocular usually suffices. The ocular assembly, which slips into the upper end of the microscope tube, carries cross hairs—one N–S (front–back) the other E–W. These enable us to locate under high power a particular mineral grain that has been brought to the center of the field under low power. They are also essential in aligning cleavage fragments for making angular measurements. A condenser is located below the stage. The upper lens of the condenser, used with high power objectives, makes the light strongly converging and can be rotated easily into or out of the optical system. The iris diaphragm, also located below the stage, can be opened or closed to control the depth of focus and to regulate the intensity of light striking the object.

In addition to the lenses, condenser and diaphragm mentioned above which are common to all compound microscopes, the polarizing microscope has several other features. The *polarizer* below the stage is a polarizing plate, or Nicol prism that transmits plane polarized light vibrating in a N–S (front–back) direction. The *analyzer*, fitted in the tube above the stage, is a similar plate or prism that transmits light vibrating only in an E–W direction. The polarizer and analyzer are collectively called *polars*. When both polars are in position they are said to be crossed and, if no anisotropic crystal is between them, no light reaches the eye. The polarizer remains fixed but the analyzer can be removed from the optical path at will. The Bertrand-Amici lens is an accessory that is used to observe interference figures. In working with crystals it is frequently necessary to change their orientation. This is accomplished by means of a rotating stage, whose axis of rotation is the same as the microscope axis.

Microscopic Examination of Minerals and Rocks

The polarizing microscope is also called the petrographic microscope because it is used in the study of rocks. In examining thin sections of rocks, the textural relationships are brought out and certain optical properties can be determined. It is equally effective in working with powdered mineral fragments. On such loose grains all the optical properties can be determined and in most cases they characterize a mineral sufficiently to permit its identification.

The optimum size of mineral grains for examination with a polarizing microscope is minus 50 mesh – plus 100 mesh, but larger or smaller sizes may be used. To prepare a mount for examination (1) put a few mineral grains on an object glass, a slide 46 mm × 27 mm, (2) immerse the grains in a drop of liquid, and (3) place a cover glass on top of the liquid. Using this type of mount the refractive indices of mineral grains are determined by the *immersion method.*

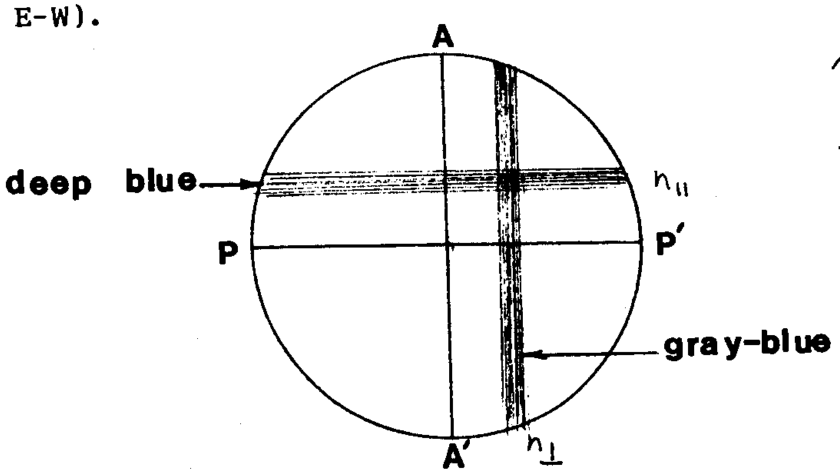
In using this method there should be available a series of calibrated liquids ranging in refractive index from 1.41 to 1.77, with a difference of 0.01 or less between adjacent liquids. These liquids cover the refractive index range of most

PLM UNIT OPERATIONS

CONTENTS:

1. To determine if a particle refractive index is > or <n of liquid it's mounted in (Becke line).
2. To observe pleochroism.
3. To set up dispersion staining.
4. To determine λ_0.
5. To do 1. (above) by dispersion staining.
6. To determine vibration direction of polarizer.
7. To cross polars properly.
8. To observe anisotropy.
9. To determine extinction angle.
10. To measure retardation.
11. To determine sign of elongation.

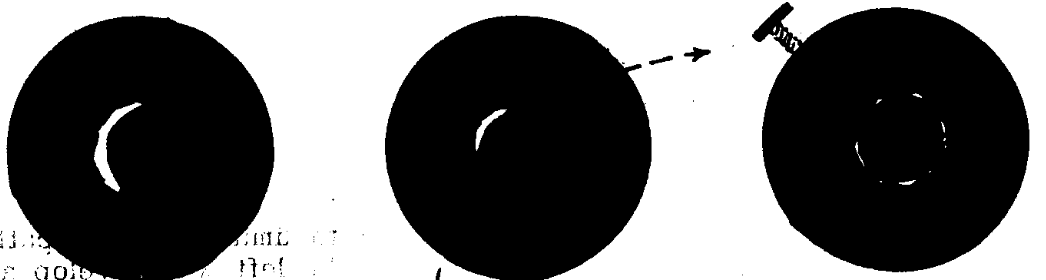
gray and when E-W, a deeper darker blue (the polarizer vibration direction must be E-W).



Also:
fibrous
— Hornblende
blue to blue green

3. **TO SET UP DISPERSION STAINING.**

The dispersion staining objective is used — hopefully with centered stage. After focusing on the particles of interest the Bertrand is introduced and the largest central stop ⊗ , or the largest annular stop ● is rotated into position. The aperture diaphram is then closed around the stop and the degree of centering of the two (stop and aperture diaphram image) is observed.



off-center **turret-centering** **centering-screw**

After centering is accomplished — first using the turret movement, then the centering screw on the objective turret the aperture diaphram is closed just behind the stop to give a fully dark objective back focal plane.

To observe central stop colors, remove the Bertrand lens.

To observe annular stop colors reintroduce the Bertrand lens, rotate the annular stop into the center of the aperture diaphram image, then remove the Bertrand to observe the particle colors.

Ⓓ λ_o is the recognition of colors
use central stop see white lt − λ_o

4. **TO DETERMINE λ_o.**

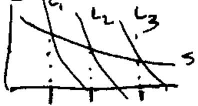
When any transparent particle is mounted in a refractive index medium and observed with an axial (closed aperture diaphram) beam of white light, that beam will be refracted at the "prism" edges of the particle. The angle of refraction will depend on the relative refractive indices of particle and liquid. The particle edges will be colored if these indices are close together. If the indices are identical it will usually be only at a single wavelength (λ_o) and the color observed by annular stop will have that wavelength; the central stop will then show the complementary color, i.e., white light minus the λ_o wavelength.

- Complimentary colors w/cent-stop
- Actual colors w/annular stop

Ex:
As (grn) blue or yellow
As (mag)
to determine complimentary color
Bl yell (green)
yell blue (mag)

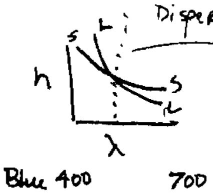
λ_o travels then if
Ⓒ

Ⓑ



3 different λ_o depending on n_o of mounting medium

Ⓐ



Dispersion curve
matching λ or λ_o

Blue 400 700 Red

— put annular stop to allow λ_o to reach viewing nately light and dark. When dark (extinction) it should be exactly parallel to one (either) of the crosslines.

If it is not, the crosslines are rotated so that they are parallel to the fiber edges when the fiber is at extinction. In some cases it may be simpler to change the position of the polars to achieve the same result. The polars should then be rotated (each by the same amount in order to keep the field of view black) leaving the fiber parallel to one of the crosslines at extinction. The polars are rotated until the fiber and field are black with the fiber edges exactly parallel to either of the crosslines (N-S or E-W).

A less satisfactory solution is to leave the polars misaligned but measure the angle of misalignment by rotating the stage and measuring the angle between fiber extinction and fiber aligned to crossline. This correction is then recorded and used subsequently as needed.

7. TO CROSS POLARS PROPERLY.

By this I mean having the polarizer E-W and the analyzer N-S. After going through Step 6, observe chrysotile fibers mounted in 1.50 HD Cargille liquid using only the polarizer in the light path. If the fibers have a lower λ_0 when aligned E-W and a higher λ_0 when aligned N-S the polarizer is properly aligned. If introduction of the analyzer (and opening the aperture diaphram or removing the central stop) yields a black field the analyzer is also properly aligned.

If the lengthwise chrysotile magenta color is shown when the fibers are aligned N-S the polarizer is also aligned N-S and should be rotated 90°. If the analyzer is then misaligned it should be rotated to yield a black field of view.

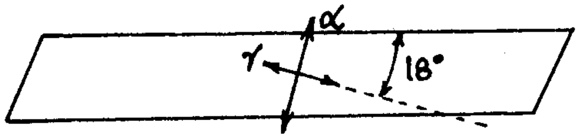
8. TO OBSERVE ANISOTROPY.

When the polars are crossed and the field of view is black (with the light on!) isotropic substances will also be black. These particles include cubic crystals and glasses (mineral or glass wool, pumice, perlite and diatoms), i.e., all substances having a single refractive index.

(non-isotropic) → Non-cubic crystals and oriented polymers (like cotton, silk, nylon, rayon etc.) will show brightness between crossed polars*. This is due to interference colors generated by particles having more than one refractive index.

9. TO DETERMINE EXTINCTION ANGLES.

Most elongated substances such as fibers will be aligned parallel to (properly aligned) crosslines when they show extinction and the extinction angle is then zero, i.e., parallel extinction. Some elongated substances will not be parallel to (properly aligned) crosslines at extinction and they must be rotated through a certain definite angle, the extinction angle, to align them with the nearest crossline. This angle will always be less than 45°.



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*Unless they lie in an extinction position.

Ⓑ

Ocular Adjustment for ~~Any~~ Ske Binocular Microscope

1. If a scale or crossline reticle is present in one of the oculars place that ocular on the right and focus the reticle by rotating the top lens of the (focusing) ocular. Ignore the preparation during this step.
2. Looking through both oculars set the interocular distance so that you see an identical image with each eye but fused to show a single field of view. Focus the preparation using a single very small line or dot detail using the right eye only with the bodytube coarse and fine adjustment. Ignore the left eye image.
3. Finally, focus the preparation for the left eye using only the independent focusing adjustment at the base of that ocular.

10-11-81

(C)

To Align Polars on Nikon SK-e

1. Insert analyzer and rotate to align 90° mark on analyzer with 0° mark on bodytube.

2. Remove substage condenser (loosen pin at 9 O'clock) and check that 90° mark on substage is aligned with marked red line on polarizer. Reinsert with 90° mark and red line at 3 O'clock position. Be sure condenser is all the way in (top lens <1 mm from stage level).

3. With light switch on, open aperture diaphram and look through the ocular at the field of view. Loosen set screw holding substage in but hold it in place so that you can rotate it in position to the blackest possible field of view, i.e., crossed polars; tighten condenser set screw.

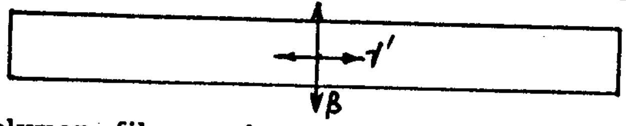
4. Place prepared slide of "12 Nylon, bright" on the stage and rotate the stage with a centered fiber to observe its extinction. It will disappear ("extinction") every 90° and hopefully when the fiber is parallel to either the E-W or N-S crosslines. If not, remove the analyzer and rotate the binocular head (after loosening its set screw, 3 O'clock position at top of bodytube) until the fiber edge is parallel to one of the crosslines.

5. If the scope is now unaesthetically askew, i.e., bodytube out of line with the base, loosen the bodytube set screw on the lower left side (near the analyzer) and rotate the bodytube to good alignment with the base.

6. The polars are now uncrossed but this can be remedied by rotating the substage to good extinction as in #3.

10-13-81

The extinction angle for a non-fibrous amphibole might be indicated: Rotated 90° about its length this crystal would show parallel extinction:



All organic polymer fibers show parallel extinction although cotton, sisal and some paper fibers show little or no extinction, i.e., remain bright in all orientations relative to the polar vibration directions.

10. TO MEASURE THE RETARDATION.

Retardation is the actual distance in nm (1 nanometer, nm, = 10^{-9} meters) the slow ray (high refractive index) is slowed behind the fast ray (low index) as both travel upward through any anisotropic substance. Retardation, r, depends on these two velocities (indices*) and on the distance traveled through the particle (its thickness — parallel to the propagation direction of the light).

r in nm = difference in n's x thickness (μm)
r = birefringence x thickness x 1000

It is observed as a color between crossed polars. This color, when compared with the Michel-Levy chart, yields the retardation directly. A compensator** may sometimes be necessary to identify some of the higher order colors.

11. TO DETERMINE SIGN OF ELONGATION.

By convention, a positive sign of elongation signifies $n_{\parallel} > n_{\perp}$; a negative sign, $n_{\perp} > n_{\parallel}$. Put another way, a positive sign means the slow component (high n) is parallel to the length. To determine the direction of the slow component requires locating the high index. This sometimes can be done by Becke line and contrast, by dispersion staining (the lower λ_0 direction is the higher n direction) or by compensator (a crystal plate with known slow component direction).

A first-order red compensator has a retardation of about 500 nm and a slow component direction usually lower left-upper right in the field of view. Superimposing this Red I plate over an elongated anisotropic particle will change the retardation by 500 nm, i.e., either increase (add the 2 retardations) or decrease it (subtract one from the other). Addition means parallel slow components or parallel high index directions. Usually we choose low order gray particles (150 nm) and a change to blue (650 nm) means addition; yellow, subtraction.

---

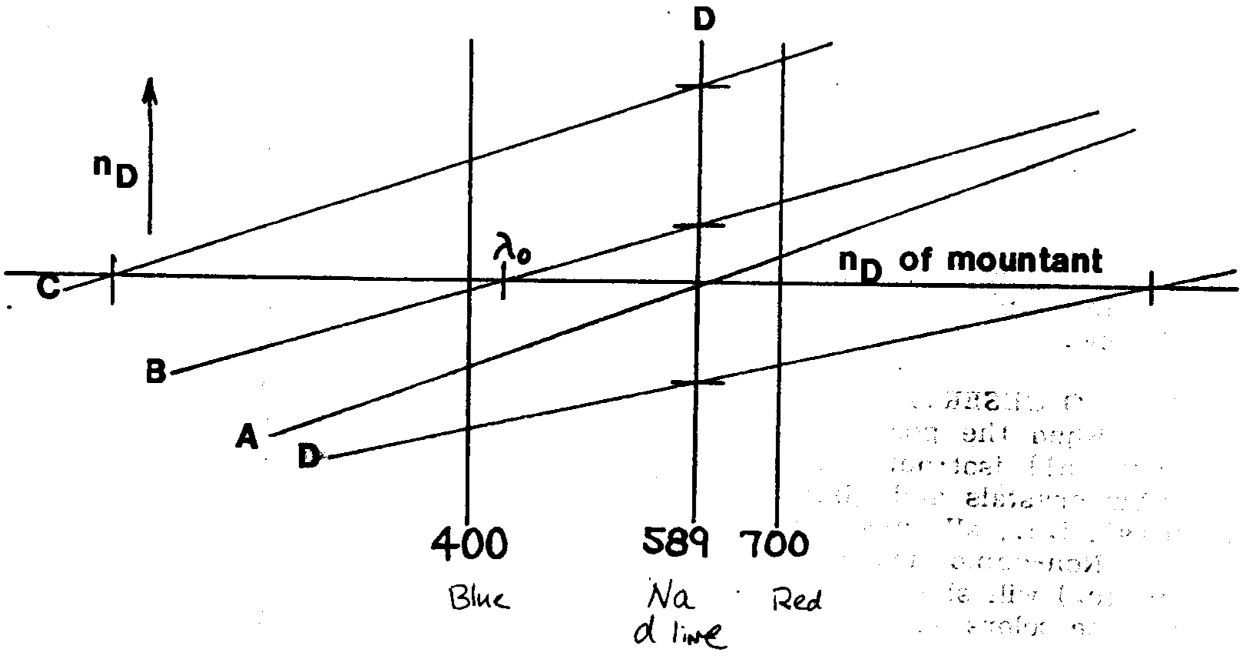
*Refractive index = $n = \dfrac{\text{Velocity in a vacuum}}{\text{Velocity in the substance}} = \dfrac{3 \times 10^{10}\text{cm/sec}}{<3 \times 10^{10}\text{cm/sec}}$

**Quartz wedge or other variable retardation compensator.

The matching wavelength, λ_0, is determined by observing the colors with annular and central stops as well as the colored Becke lines with the central stop and interpolating a λ_0 value from the table given both in the Polarized Light manual (p. 173) and the Asbestos Manual (p. 25).

5. TO DETERMINE WHETHER A PARTICLE HAS A LOWER OR HIGHER REFRACTIVE INDEX THAN THE MOUNTING LIQUID BY DISPERSION STAINING.

λ_0 values below 589 nm (D-line) indicate a particle index greater than the mounting liquid; above 589 nm a lower refractive index. λ_0 values well below 400 nm will show pale yellow with the central stop and dark blue with the annular stop. This signifies particle indices greater than the liquid. Corresponding colors with lower particle indices will be pale blue (C.S.) and dark brown (A.S.).

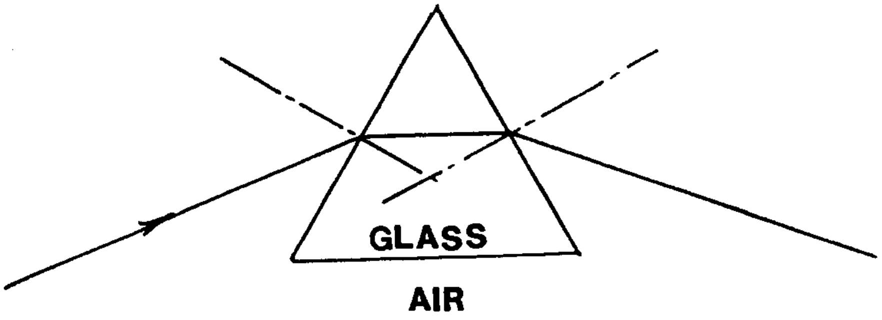


The intersection of each particle dispersion staining curve on the D-line (589 nm) is its refractive index.
A has the same n as the liquid at 589 nm.
B has a slightly higher n than the liquid (at 589).
C has a much higher n than the liquid (at 589).
D has a much lower n than the liquid (at 589).

6. TO DETERMINE VIBRATION DIRECTIONS OF THE POLARS (POLARIZER AND ANALYZER).

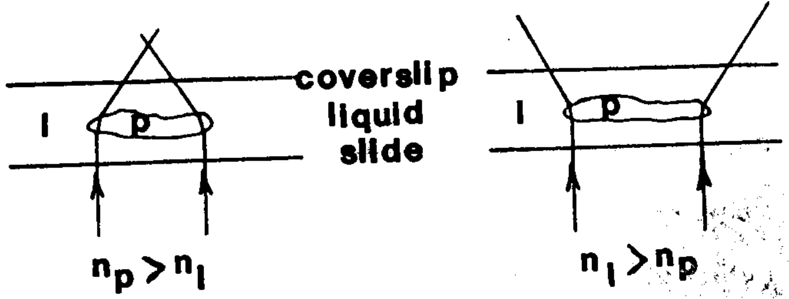
The polarizer may be left in the light path for all observations normally conducted with the PLM. On inserting the analyzer the field of view should be dark <u>AND</u> any particles that show parallel extinction should be aligned parallel to the crosslines when at extinction. A fiber of nylon, Dacron, rayon or any other synthetic fiber can be used for this purpose. Simply rotate the stage watching the fiber between crossed polars. It will be alter-

1. TO DETERMINE PARTICLE n VS LIQUID n BY BECKE LINE.



Refraction of light by a glass prism in air.

Light traveling upward through the microscope is refracted, i.e, changes direction, at each optical interface. It is bent toward the $\perp$ to that interface if it enters a medium of higher index (and vice versa).



The aperture diaphram must be closed in order to limit the light path to an axial beam. On focusing upward the particle on the left will develop a bright center. The particle on the right, on focusing higher, will show a dark center and the liquid immediately adjacent to the particle boundaries will show a bright halo of light, In either case, the brightness will appear in the medium of highest index hence:

— on focusing higher the halo (Becke line) will move to the higher index medium. On focusing lower the halo will move to the lower medium.

2. TO OBSERVE PLEOCHROISM.

Pleochroism is the variation in absorption color with direction of light vibration in an anisotropic colored particle — an isotropic particle or a colorless anisotropic particle will show no such color variation.

The particle, say, a crocidolite fiber bundle (must be thin) is observed in any mounting liquid (1.55, 1.605 or 1.68) with a single East-West polar (see Step 6). The stage must be centered (using the objective centering screws) so that the crocidolite fibers rotate about the center of the field as the stage rotates.

When this is done the thinner crocidolite fibers will show a difference in their blue color. When oriented N-S the fibers will be pale blue or blue-

D

Nikon SK-e Microscope

To Use Dispersion Staining (D.S.) Objective

Remove any 3 of the 4 objectives and insert D.S. objective (retaining ring on D.S. objective should be screwed part way up the threads).

Conoscopically, rotate D.S. turret to "no-stop" position and focus orthoscopically on prepared slide #20 (Calcite in $N_D = 1.662$). Center objective with its 2 centering screws (on nosepiece) relative to field diaphram and stage centers.

Conoscopically, rotate D.S. turret to largest diameter central stop and close the aperture diaphram to check its centration relative to D.S. stop. Center, if necessary, with D.S. objective centering screw on turret. (This is not a clamping screw.) Set lamp voltage to 6 volts and observe a well-cleaved calcite rhomb orthoscopically (one polar). The ω direction will show a blue central stop color (yellow annular stop color) when the bisector of the acute rhomb angle is parallel to the polarizer vibration direction. The ε' direction will be white (central stop: black with annular stop) because that index, 1.53, is so much lower than 1.662, the liquid index; λo is very much greater than 700 nm.

On removal of the D.S. objective the 3 regular objectives should be replaced in their original positions: 4X, 10X, 40X, 100X clockwise (from above). The objective now occupying the position of the D.S. objective should be recentered with its own centering screws relative to stage rotation and field diaphram. All objectives should now be fully centered and the microscope ready for use.

Min Fibers	Refractive	Indices	Biref.	Colc	Extcn.	Sign	Dispersion Staining		
	α	γ	γ-α			Elongn.	n_D(liq.)	∥ lg.	⊥ lg.
Chrysotile	1.532 - 1.549	1.545 - 1.556	0.015	white	∥	+	1.550	magenta	blue
Antigorite [1]	1.558 - 1.567	1.562 - 1.574	0.01	white	∥	+	1.550	gol. yel.	magenta
Lizardite	1.545	1.558	0.013	white	undulose	+ (edge)	1.550	gol. mag.	p. blue
Amosite C.	1.633 - 1.664	1.654 - 1.687	0.02	white	∥	+	1.680	blue	p. blue
G.	1.664 - 1.686	1.687 - 1.729	0.03	p. green	∥	+	1.680	gold	blue
Crocidolite	1.654 - 1.698	1.666 - 1.712	0.01	blue	∥ & obl.	–	1.680	p. yellow	yellow
Tremolite	1.603 - 1.620	1.627 - 1.642	0.02	white	∥ & obl.	+	1.605	yellow	blue [2]
Actinolite	1.620 - 1.667	1.642 - 1.686	0.02	white	∥ & obl.	+	1.605	v. p. yel.	p. yellow
Ferroactinolite	1.667 - 1.683	1.686 - 1.702	0.02	p. green	∥ & obl.	+	1.680	gol. yel.	p. blue
Anthophyllite	1.606 - 1.648	1.626 - 1.670	0.02	white	∥	+	1.605	yellow	gol. mag.
Byssolite	= fibrous tremolite/actinolite			white	∥ & obl.	+	1.605		
Richterite	1.590	1.611	0.02	white	∥ & obl.	+	1.605	magenta	p. blue
Brucite [3]	1.560 - 1.590(ω)	1.580 - 1.600(ε)	0.025	white	∥	+ –	1.550	yellow	p. yellow
Talc	1.539 - 1.550	1.589 - 1.600	0.05	white	∥	+	1.550	p. yellow	blue-gr.
Palygorskite [4]	ca. 1.52	ca. 1.53	ca. 0.01	white	∥	–	1.550	v. p. blue	v. p. blue
Epsomite [5]	1.433	1.461	0.03	white	∥	–	1.550	white	white
Prieskaite [6]	1.665	1.679	0.014	p. green	∥ & obl.	–	1.680	blue	p. blue
Bastite [7]	1.676	1.689	0.013	brown	∥	+	1.680	p. yellow	blue
Amosite [8]	ca. 1.74	ca. 1.94	0.2	red	∥	+	1.680	white	white
Wollastonite [9]	1.612	1.632	0.02	white	tricl.	+,–	1.605	p. yellow	p. yel. to mag.
Szaibelyite [10]	1.581	1.666	0.085	white	∥	–	1.605	v. p. blue	p. yellow

1. fibrous antigorite = picrolite

2. ⊥ lc. blue (obl. extcn.) magenta (∥ extcn.)

3. $Mg(OH)_2$

4. a clay, $Mg_2Si_3O_8 \cdot 4H_2O$

5. $MgSO_4 \cdot 7H_2O$

6. a ferro actinolite

7. a ferro magnesium silicate

8. heated

9. $CaSiO_3$

10. $(Mg, Mn)(BO_2)(OH)$

Abbreviations

∥ = parallel, ⊥ = perpendicular
gol. = golden
yel. = yellow
gr. = green
mag. = magenta
obl. = oblique
p. = pale
ca. = about
v. p. = very pale
bl. = blue
C. = cummingtonite
G. = grunerite

NONASBES IFORM FIBERS

MINER WOOL	FIBER GLASS	CELLULOSE	POLYESTER	ANIMAL HAIR	SPITER WEB
Morph: 8-20 mD varing thickness sickle shaped to straight glass globule isotropic RI>1.53 Confusion: 1)fiberglass DSC 1.55 LQ vivid blue MW-gold 2)amosite anisotropic Color: white dark brown green	Morph: 1-20 mD always straight cylindrical uniform thickness Color: white to colorless Isotropic RI<1.53 Confusion: 1)mineral wool DSC 1.55 LQ vivid blue 2)amosite anisotropic	Morph: flat, ribbon, irregular twisted, woven Color: drk brown to white anisotropic n<1.55 DSC 1.55 blue yellow	Morph: long cylindrical very flexible rarely straight Color: various	Morhp: scaly surface one pointed end Color: zoned colored DSC 1.55 blue blue	Morph: fine fibrous silky no kinking DSC similiar to chrysotile Confusion: chrysotile

IR and DTA can also be dismissed for all except routine samples containing high percentages of asbestos.

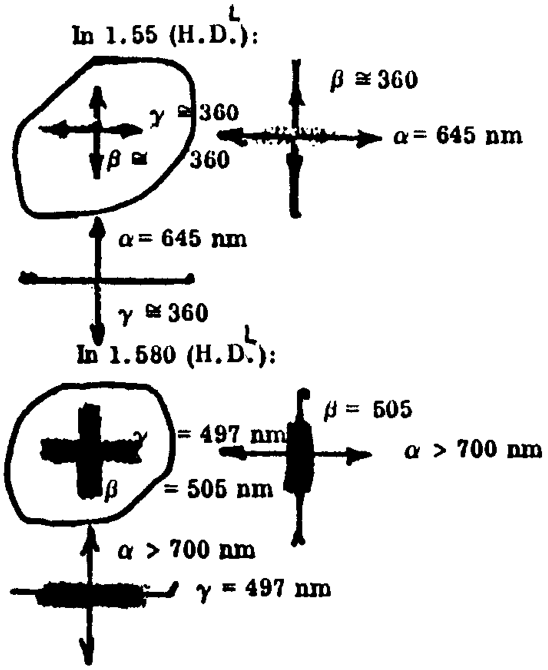
This brings us to PLM and DS on which we wish to spend more time because of our conviction that, of all the microanalytical techniques for asbestos, it is by far the most effective. It is the only method depending on the unique optical crystallographic properties of the various crystal phases in the sample. These properties – refractive indices, dispersion of refractive indices, birefringence, sign of elongation and extinction angle – are unique to the crystalline state and therefore unequivocally identify chrysotile, anthophyllite, tremolite, actinolite, grunerite, cummingtonite, etc.

The background for dispersion staining has been adequately covered elsewhere [1][1]. Very briefly, it imparts color to any transparent particle mounted in a liquid whose dispersion curve intersects the dispersion curve for the particle in the visible. The colors, related to this matching wavelength, characterize and identify any given substance. With polarized light, isotropic substances show a single characteristic color, but anisotropic substances show different colors corresponding to the different refractive indices in different orientations. Chrysotile, for example, shows blue and blue-magenta colors, crosswise and lengthwise respectively, for each needle crystal when mounted in Cargille high dispersion liquid $n_D^{25} = 1.550$.

The colors shown by the various types of asbestos and a few other associated minerals are indicated in Figures 1-20 by the wavelengths on each crystal view. These are the wavelengths at which the liquid indicated and that direction in the crystal have the same refractive index. This matching wavelength, λ_0, determines the dispersion staining colors.

TALC

$Mg_6(Si_8O_{20})(OH)_4$



n's (D, H, Z)*

α = 1.539-1.550
β = 1.589-1.594
γ = 1.589-1.600
δ ≅ 0.05 (-)

This sample (Vermont):

α = 1.546
β = 1.588
γ = 1.589
δ = 0.045 (-)

* Deer, Howie and Zussman.

Figure 1. Dispersion staining colors shown by talc crystals in Cargille high dispersion liquids $n_D = 1.550$ and $n_D = 1.580$.

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[1] Figures in brackets indicate the literature references at the end of this paper.

CHLORITE

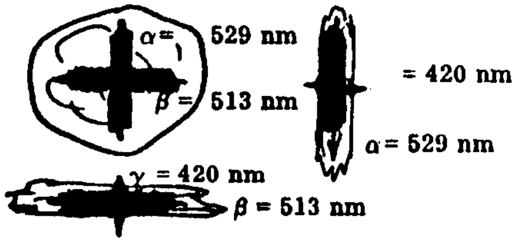
$(Mg,Al,Fe)_{12}[(Si,Al)_8O_{20}](OH)_{16}$

Figure 2. Chlorite.

In 1.55 (H.D.):

pale yellow to golden yellow

In 1.580 (H.D.):



n's (D, H, Z)

α = 1.57-1.66
β = 1.57-1.67
γ = 1.57-1.67
δ = 0-0.01

The sample (California):

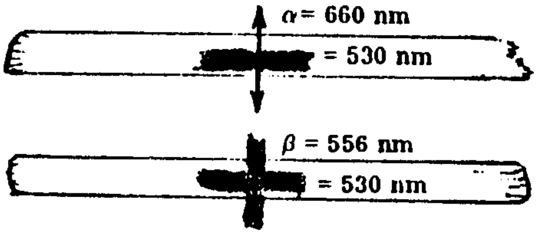
α = 1.586
β = 1.587
γ = 1.596
δ = 0.010 (+)

CHRYSOTILE

Figure 3. Chrysotile.

$Mg_3[Si_2O_5](OH)_4$

In 1.550 (H.D.):



n's (D, H, Z)

α = 1.532-1.549
β = 1.540-1.553
γ = 1.545-1.556
δ = 0.013-0.007 (-)

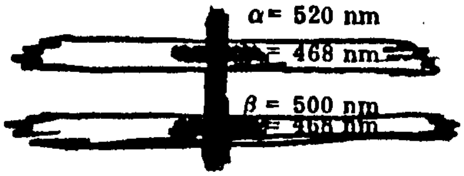
King's Mine, Quebec Sample → α = 1.5444
β = 1.5525
γ = 1.5555
δ = 0.0111

ANTIGORITE

$Mg_3(Si_2O_5)(OH)_4$

Figure 4. Antigorite.

In 1.550 (H.D.):



n's (D, H, Z)

α = 1.558-1.567
$\beta \cong$ 1.56 -1.57
γ = 1.562-1.574
δ = 0.004-0.007 (-)

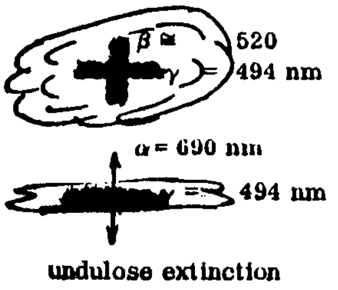
This sample:

α = 1.555
β = 1.559
γ = 1.561
δ = 0.006 (-)

LIZARDITE

$Mg_3(Si_2O_5)(OH)_4$

In 1.55 (H.D.):



n's (D, H, Z)

$\alpha = 1.538-1.544$
$\beta =$
$\gamma = 1.546-1.560$
$\delta = 0.016-0.008$

This sample:

$\alpha = 1.545$
$\beta \cong 1.555$
$\gamma = 1.557$
$\delta = 0.012$ (-)

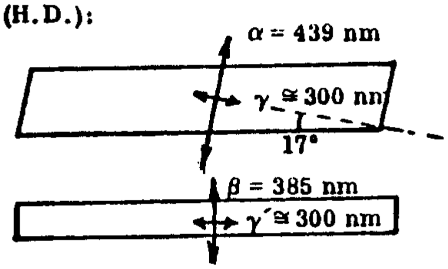
Figure 5. Lizardite.

TREMOLITE

$Ca_2(Mg, Fe^{+2})_5(Si_8O_{22})(OH, F)_2$

(low Fe)

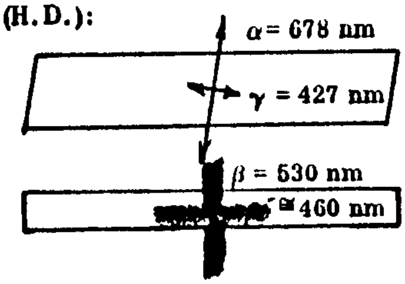
In 1.580 (H.D.):



n's (D, H, Z)

$\alpha = 1.604-1.619$
$\beta =$
$\gamma = 1.627-1.642$
$\delta = 0.021-0.023$ (-)

In 1.605 (H.D.):



This sample:

$\alpha = 1.599$
$\beta = 1.610^{+}$
$\gamma = 1.621$
$\delta = 0.022$ (-)

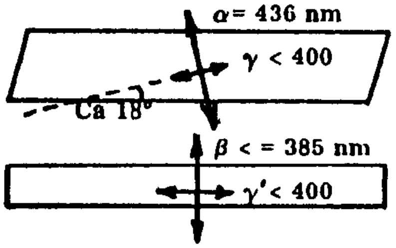
Figure 6. Tremolite.

ACTINOLITE

$Ca_2(Mg, Fe)_5(Si_8O_{22})(OH, F)_2$

(20-80% Fe & 80-20% Mg)

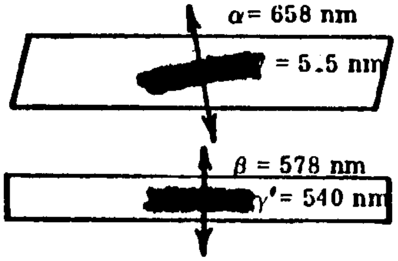
In 1.605 (H.D.):



n's (D, H, Z)

$\alpha = 1.619-1.668$
$\beta =$
$\gamma = 1.642-1.687$
$\delta = 0.023-0.019$ (-)

In 1.640 (H.D.):



This Sample (Virginia):

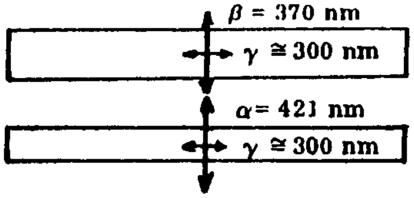
$\alpha = 1.633$
$\beta = 1.641$
$\gamma = 1.647$
$\delta = 0.014$ (-)

Figure 7. Actinolite.

ANTHOPHYLLITE

$(Mg, Fe^{2+})_7(Si_8O_{22})(OH, F)_2$

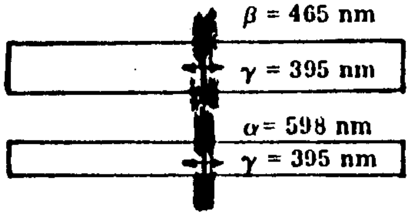
In 1.580 (H.D.):



n's (D, H, Z)

$\alpha = 1.596-1.694$
$\beta = 1.605-1.710$
$\gamma = 1.615-1.722$
$\delta = 0.013-0.028$
(+) (-)

In 1.605 (H.D.):



This sample (Pine Mt., Ga.)

$\alpha = 1.601$
$\beta = 1.618$
$\gamma = 1.628$
$\delta = 0.027$ (-)

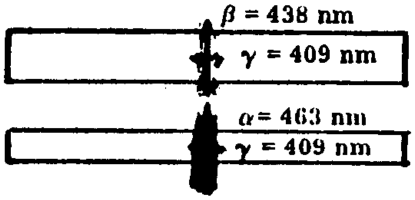
Figure 8. Anthophyllite.

ANTHOPHYLLITE

$(Mg, Fe^{2+})_7(Si_8O_{22})(OH, F)_2$

(Mg > Fe)

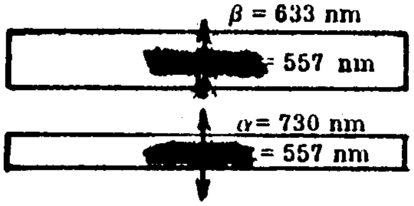
In 1.640 (H.D.):



n's (D, H, Z)

α = 1.596-1.694
β = 1.605-1.710
γ = 1.615-1.722
δ = 0.013-0.028
(+) (-)

In 1.67:



This sample (Connecticut):

α = 1.659
β = 1.666
γ = 1.674
δ = 0.015 (+)

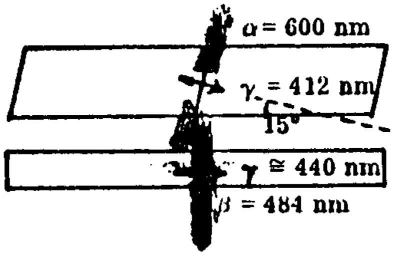
Figure 9. Anthophyllite.

GRUNERITE

$(Fe^{+2}, Mg)_7(Si_8O_{22})(OH)_2$

(high Fe)

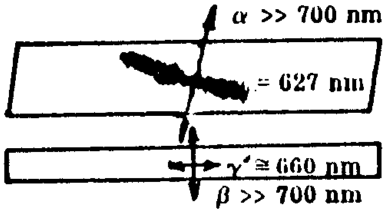
In 1.670:



n's (D, H, Z)

α = 1.663-1.686
β ≅ 1.681-1.707
γ = 1.697-1.729
δ = 0.034-0.043 (-)

In 1.700:



This sample:

α = 1.669
β = 1.684
γ = 1.697
δ = 0.028 (-)

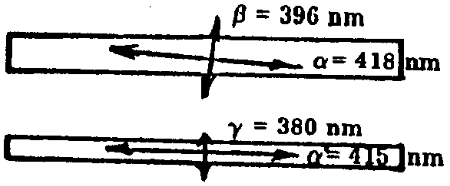
Figure 10. Grunerite.

CROCIDOLITE

$Na_2Fe_3^{+2}Fe_2^{+3}(Si_8O_{22})(OH)_2$

(no Mg, contains Fe^{+2})

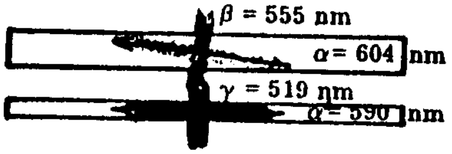
In 1.670:



n's (D, H, Z)

α = 1.654-1.701
β = 1.662-1.711
γ = 1.668-1.717
δ = 0.006-0.016 (-)

In 1.700:



This sample (Orange River, South Africa):

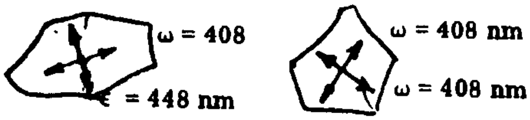
α = 1.698
β = 1.703
γ = 1.708
δ = 0.010 (-)

Figure 11. Crocidolite.

APATITE

$Ca_5(PO_4)_3(OH, Cl, F)$

In 1.605 (H.D.):



n's (D, H, Z)

ε = 1.624-1.666
ω = 1.629-1.667
δ = 0.001-0.007 (-)

In 1.64 (H.D.):

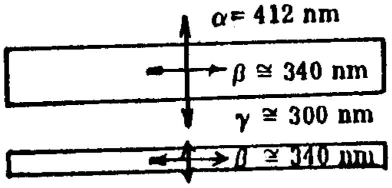
This sample:

ε = 1.6295
ω = 1.6357
δ = 0.0062 (-)

Figure 12. Apatite.

WOLLASTONITE
$Ca(SiO_3)$

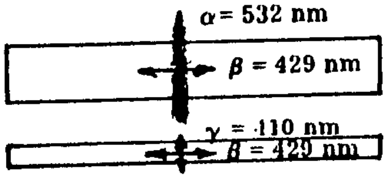
In 1.580 (H.D.):



n's (D, H, Z)

α = 1.616-1.640
β = 1.628-1.650
γ = 1.631-1.653
δ = 0.015-0.013 (-)

In 1.605:



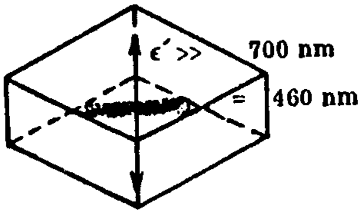
This sample:

α = 1.612
β = 1.628
γ = 1.632
δ = 0.020 (-)

Figure 15. Wollastonite.

CALCITE
$CaCO_3$

In 1.64 (H.D.):



n's (D, H, Z)

ϵ = 1.486-1.550
ω = 1.658-1.74
δ = 0.172-0.190 (-)

This sample:

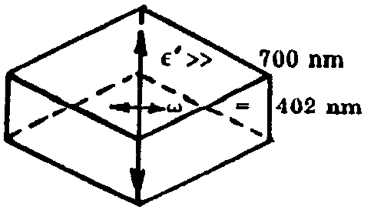
ϵ' = 1.525
ϵ = 1.486
ω = 1.653
δ = 0.167 (-)

Figure 16. Calcite.

DOLOMITE

$(Ca,Mg)CO_3$

In 1.64 (H.D.):

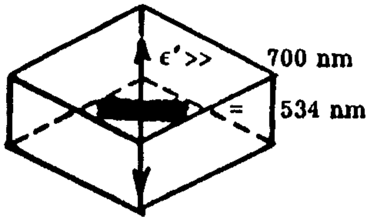


n's (D, H, Z)

$\epsilon = 1.500^{*}-1.520^{**}$
$\omega = 1.679^{*}-1.703^{**}$
$\delta = 0.179-0.185$ (-)

*pure dolomite
**plus Fe^{2+} for Mg

In 1.67:



This sample:

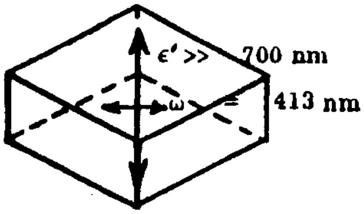
$\omega = 1.677$

Figure 17. Dolomite.

MAGNESITE

$MgCO_3$

In 1.670:

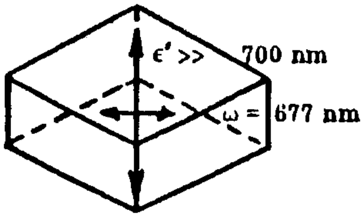


n's (D, H, Z)

$\epsilon = 1.509-1.563^{*}$
$\omega = 1.700-1.782^{*}$
$\delta = 0.190-0.218$ (-)

*with Fe replacing Mg

In 1.700:



This sample:

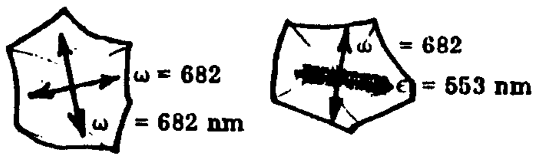
$\omega = 1.694$

Figure 18. Magnesite.

QUARTZ

SiO_2

In 1.550 (H.D.):



n's (D, H, Z)

$\omega = 1.544$
$\epsilon = 1.553$
$\delta = 0.009$ (+)

This sample:

$\omega = 1.544$
$\epsilon = 1.553$

Figure 19. Quartz.

ORGANIC FIBERS (1.550 liquid)

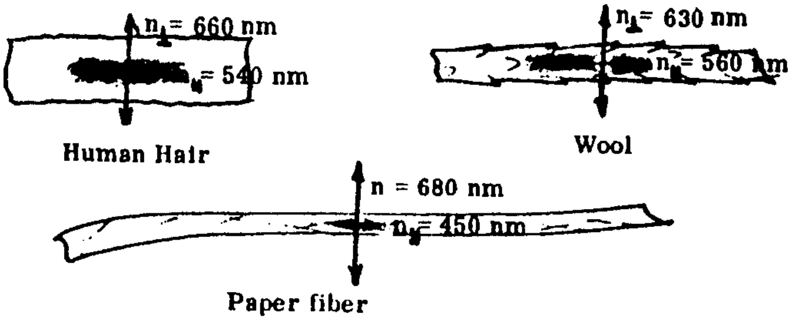


Figure 20. Organic fibers.

Although we speak of dispersion staining colors as specific for a given substance in a given liquid (at a given temperature) we sometimes observe closely similar colors for other substances. We must, especially when this possibility exists, make sure that we observe enough data to be able to state with certainty that the substance is, say, chrysotile. It is not sufficient to observe the proper color in one direction - both chrysotile and paper fibers can show the same blue color perpendicular to their lengths. Nor is it sufficient to observe the two colors on a single view of a crystal - both quartz and chrysotile can have two colors in common. If all colors shown by the crystal in all orientations correspond to the known data for a given substance, and if the crystal morphology shows the colors to be oriented properly, there is then very little chance of misidentification.

Fig. 1.
Fibre of Sheep's Wool. Magnified 600 diameters.

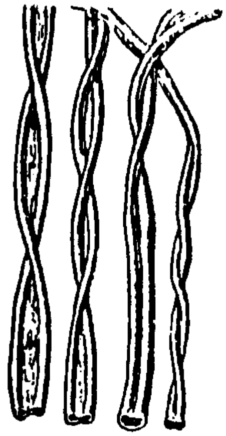


Fig. 2.
Filaments of Raw Cotton.

Fig. 3.
Fibre of Raw Silk

Fig. 4.
Radial Thread of Garden Spider.

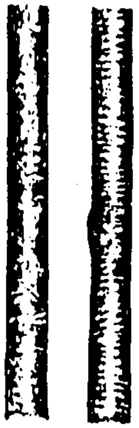
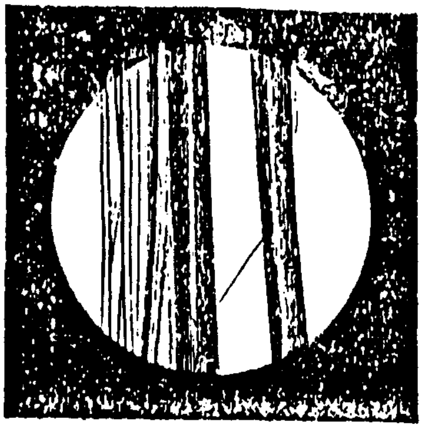
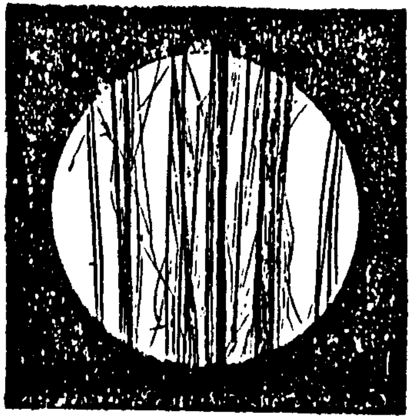


Fig. 6.
Spun Glass.



No. 1.—Thetford Ore.



No. 2.—Thetford Ore.

ASTM COMMITTEE D22.05 January 14, 1988

STANDARD METHOD OF TESTING FOR ASBESTOS CONTAINING MATERIALS BY POLARIZED LIGHT MICROSCOPY

1. SCOPE

1.1 The method describes the procedures for the determination of the presence or absence of six types of asbestos: chrysotile-asbestos, grunerite-asbestos (amosite), crocidolite (riebeckite-asbestos), anthophyllite-asbestos, tremolite-asbestos and actinolite-asbestos and for the determination of a quantitative estimate of the percent of asbestos. This method may be applied to bulk materials other than building materials but the accuracy of the method under these circumstances is not characterized. For non-building materials there may be more interference with a greater possibility for false positives or fibers dispersed below the resolution of the light microscope yielding a higher possibility of false negatives. When the content of asbestos in a sample is close to the 1% level, other more precise methods of quantification may be necessary if it is important to determine whether or not asbestos content is more or less than 1% by weight. This distinction may be important because the EPA defines asbestos-containing materials as those materials containing greater than 1% asbestos (Ref. 2 and 3).

2. APPLICABLE DOCUMENTS

2.1 U.S. Environmental Protection Agency, "Interim Method for the Determination of Asbestos in Bulk Insulation Samples", EPA 600/M4-82-020, Dec. 1982.

2.2 U.S. Environmental Protection Agency, "Guidance for Controlling Asbestos-Containing Materials in Buildings", EPA 560/5-85-024, 1985.

2.3 U.S. Environmental Protection Agency, "Asbestos-Containing Materials in School Buildings: Guidance for Asbestos-Analytical Programs", EPA 560/13-80-017A, 1980 (under revision).

2.4 ASTM STD 834, Definitions for asbestos and other health-related silicates, B. Levadie, ed., ASTM, 1916 Race Street, Philadelphia, PA 19103, 1984.

3. TERMINOLOGY

3.1 Asbestos: A commercial term applied to a group of highly fibrous silicate minerals that readily separate into long, thin, strong fibers of sufficient flexibility to be woven, are heat resistant and chemically inert, and possess a high electric insulation, and therefore are suitable for uses (as in yarn, cloth, paper, paint, brake linings, tiles, insulation, cement, fillers, and filters) where incombustible, nonconducting, or chemically resistant material is required. Federal regulation of asbestos is restricted to chrysotile-asbestos, grunerite-asbestos (amosite), crocidolite (riebeckite-asbestos), anthophyllite-asbestos, tremolite-asbestos and actinolite asbestos.

3.2 Asbestiform: said of a mineral that is like asbestos, i.e. crystallizes with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable such as long fiber length and high tensile strength. Under the light microscope, the asbestiform habit is generally recognized by the following characteristics:

1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5µm.
2) very thin fibrils, usually less than 0.5 micrometers in width, and
3) two or more of the following:
    a) parallel fibers occurring in bundles,
    b) fiber bundles displaying splayed ends,
    c) fibers in the form of thin needles,
    d) matted masses of individual fibers, and
    e) fibers showing curvature.

4. SUMMARY OF THE METHOD

4.1 Bulk samples of building materials taken for asbestos identification are first examined with a low-power binocular microscope for homogeneity, the presence or absence of fibrous constituents, preliminary fiber identification, and an estimate of fiber content. Possible identification of fibers or the confirmation of the absence of fibers is made by analysis of subsamples with the polarized light microscope.

5. SIGNIFICANCE AND USE

(5.1) This method of testing is applicable to building materials including insulation, ceiling tiles, surface coatings, asbestos board, pipe coverings, etc. It is not recommended for floor tiles. However, if fibers can be liberated from a non-friable matrix, they can be identified by this method.

5.2 If the estimate of the percentage of asbestos in a sample is close to the 1% by weight level, other methods of quantification may be necessary if it is important to determine whether or not asbestos content is more or less than 1% by weight. This distinction may be important because the EPA defines asbestos-containing materials as those materials containing greater than 1% by weight asbestos (Ref. 2 and 3).

5.3 The details of the methods used to determine the optical properties of minerals are not included in this method. The method assumes that the analyst is proficient in making these measurements.

6. INTERFERENCES

6.1 Cellulose may have approximately the same index of refraction as chrysotile-asbestos. For this reason it is frequently confused with chrysotile. However, cellulose fibers frequently pinch and swell along their length, exhibit internal cellular structure, and lack splayed ends: they are not composed of bundles of smaller fibers.

6.2 Cleavage fragments of many natural minerals including amphiboles, talc, gypsum, wollastonite and vermiculite may appear as elongated anisotropic particles. The aspect ratio of these particles may be as great as 20:1. Therefore, aspect ratio alone is not sufficient for the identification of asbestos. Other properties of the asbestiform habit such as curved fibers, fiber bundles exhibiting splayed ends, and fibers with aspect ratios in excess of 20:1 must be observed in order to be sure asbestiform material is present in the sample. However, these properties need not be characteristic of every fiber or fiber bundle in the sample. Therefore, once asbestos is known to be present, other properties such as index of refraction and aspect ratio can be used to identify asbestos and determine which particles will be counted in making a quantitative estimate of the amount of asbestos in the sample.

6.3 Sprayed-on binder materials may coat fibers and affect color or obscure optical characteristics. Fine particles of other materials may also adhere to fibers. Occasionally procedures other than those described in this test method may be helpful if the analyst is unable to observe fibers clearly. Some of these are described in Reference 1.

6.4 Vermiculite may be confused with chrysotile because it has a similar index of refraction and, while it is not

7.7 Ocular lens: 8x minimum

7.8 Eyepiece reticle: cross hair

7.9 compensator (wave retardation plate): 550 nanometer (first-order red or gypsum)

7.10 Microscope slides

7.11 Coverslips

7.12 Mortar and pestle: agate or porcelain

8. REAGENTS

8.1 Index of refraction liquids: $N_D = 1.490\text{-}1.720$ in increments of 0.002 or 0.004.

8.2 Index or refraction liquids for dispersion staining: high dispersion series, $N_D = 1.550$, 1.605, and 1.680. (Optional. Required only if dispersion staining will be used to measure the index of refraction.)

8.3 Reference materials:

8.3.1 Asbestos Materials

a. Commercial asbestos, including amosite, chrysotile, crocidolite, and anthophyllite-asbestos. (UICC Asbestos Reference Sample Set available from UICC MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF6 1XW UK and commercial distributors.)

b. Tremolite-asbestos: available from commercial distributors such as Ward's Natural Science Establishment, Inc., P. O. Box 92912, Rochester, New York, 14692-9012.

c. Actinolite-asbestos: source to be determined (very rare; not used commercially).

8.3.2 Suggested Matrix and Non-asbestos materials.

a. Cellulose

b. Vermiculite: source to be determined.

c. Non-asbestiform amphiboles: available from commercial distributors such as Ward's Natural Science Establishment, Inc., P.O. Box 92912, Rochester, New York 14692-9012.

d. Other silicates such as fibrous talc, wollastonite, gypsum, nemalite (brucite): available from commercial distributors such as Ward's Natural Science Establishment, Inc., P.O. Box 92912, Rochester, New York 14692-9012.

e. Synthetic fibers such as fiberglass and mineral wool.

9. PRECAUTIONS

9.1 This method involves the analysis of material (asbestos) which may be hazardous if inhaled. It does not address the safety problems associated with its use. In addition, it should be noted that some immersion oils manufactured prior to 1978 may contain Polychlorinated Biphenols (PCB). PCB's have been identified as hazardous materials. It is the responsibility of whoever uses this method to establish appropriate safety and health practices to ensure that asbestos is not inhaled and exposure to PCB's does not occur.

10. SAMPLING

10.1 Samples should be taken in the manner prescribed in Reference 2. Information on design of sampling and analysis programs may be found in Reference 3. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

11. GENERAL METHOD DESCRIPTION

11.1 Bulk samples of building materials are first examined with a low power binocular microscope or magnifying glass for homogeneity, the presence or absence of fibrous constituents, preliminary fiber identification and an estimate of fiber content.

11.2 Positive identification of fibers or the confirmation of the absence of fibers is made by analysis of subsamples with the polarized light microscope according to the outline presented in Table I. The optical properties of six types of asbestos are given in Table II. The use of plane polarized light allows the determination of index of refraction parallel to elongation. Morphology and color are observed. Orientation of the two polarizers such that their vibration directions are perpendicular (crossed polars) allows the distinction between anisotropic and isotropic materials to be made. It also allows observation of the birefringence and extinction characteristics of anisotropic particles. When a compensator is inserted into the optical path, the sign of elongation of the particle can be determined. Also, the fibrillar structure of asbestos is most evident under crossed polars.

Dispersion? staining

11.3 Identification of the fibrous constituents is facilitated by comparison of the unknowns to materials in the reference collection.

11.4 A quantitative estimate of the amount of asbestos present is derived from the combination of the estimate made from slide preparations and the estimate of total fiber made from examination of the bulk sample.

12. SAMPLE PREPARATION

12.1 For initial observation, the sample should be placed on a clean glass plate or glassine paper and placed under the binocular microscope or examined with a magnifying glass. Color, the presence or absence of fibers, and homogeneity should be observed and recorded. If only an occasional fiber is observed, one or two should be isolated with forceps and prepared for examination by polarized light microscopy. A preliminary estimate of total fiber content can be made at this time.

12.2 Subsamples for polarized light microscopy are usually best prepared by using forceps to sample at several places from the bulk material. These subsamples are immersed in a refractive index liquid on a microscope slide, teased apart and covered with a cover glass. At a minimum, two slide preparations should be made.

12.3 If the material is obviously layered or comprised of two or more materials that differ in color or texture, slide preparations of each component should be made.

12.4 If the sample is not readily friable or if the sample consists of a coarse-grained matrix, a mortar and pestle can sometimes be used to crush the sample.

12.5 Other methods of sample preparation for homogenization and to remove interferences, such as milling, acid and sodium metaphosphate treatment and ashing, are not normally necessary. They are described in Reference 1.

13. IDENTIFICATION OF ASBESTOS

13.1 Positive identification of asbestos requires the determination of the following optical properties: morphology, color and pleochroism, index of refraction parallel to elongation, birefringence, extinction characteristics and sign of elongation. Techniques for determining these properties are described in References 4 through 8. Characteristics of the asbestiform habit (morphology) are described in References 9 and 10. The sign of elongation is determined by use of a compensator and crossed polars. Index of refraction may be determined by the Becke line method (Reference 4) or by dispersion staining (Reference 8). The optical properties are given in Table II. General optical properties of silicates other than asbestos are found in References 4-7).

14. QUANTIFICATION OF ASBESTOS CONTENT

14.1 A quantitative estimate of the amount of asbestos present is most readily obtained by visual comparison of the bulk sample and slide preparations to other slide preparations and bulk samples with known amounts of asbestos present in them.

14.2 Point counting of slide preparations is not generally recommended. Point counting only produces accurate quantitative data when the material has uniform thickness. In practice, the thickness of asbestos-containing materials placed on a glass slide for petrographic analysis is often highly variable, rendering quantitative volume estimates inaccurate. The method recommended by the EPA for determining the amount of asbestos uses point counting techniques. It is described in Reference 1.

14.3 Estimates of the quantity of asbestos obtained by the method described in 14.1 above are neither volume nor weight-percent estimates. They are based on estimating the projected area from observation of the distribution of particles over the two-dimensional surface of the glass slide and on an observation of the bulk material. A basis for correcting to a weight or volume percent basis has not been established. However, the error introduced by assuming that the estimates are equivalent to weight percent is probably within the precision of the visual estimate technique.

15. DATA PRESENTATION

15.1 The following information should be reported for each sample: color, presence or absence of asbestos, type or types of asbestos present, estimate of the area percentage of each type of asbestos present, area percentage of other fibrous materials present, and identity of other fibrous materials if known.

15.2 If the sample submitted for analysis is inhomogeneous and subsamples of the components were analyzed separately, the data for each subsample should be recorded separately. However, the separate components should be combined in proportion to their abundances and a single analysis should be provided for the sample as a whole.

15.3 Example Sample Analysis Sheet

Analysis of Asbestos in Bulk Materials

Sample Identification:

Analyst:

Date:

Macroscopic Examination:

1. Size and Condition of Sample:
2. Texture: (occurrence of fibrous and other components)
3. Color:
4. Homogeneity:
5. Comments:

Microscopic Examination:

1. Number and Size of Subsamples:
2. Preparation: (incl. grinding, ashing, acid washing, ...)

3. Method of estimation if other than visual estimation:

4. Standards used for quantitation (if any);

5. Index of refraction of the immersion medium.

Sample Identification:

Analysis of fibrous component:

	Component 1	Component 2
a. Morphology		
b. Color		
c. Birefringence		
d. Extinction characteristics		
e. Indexes of refraction (dispersion characteristics)		
f. Sign of Elongation		
g. Estimated range (percent area) of fibrous component		

Comments: (Describe any unusual characteristics or problems with analysis, and if possible, briefly describe nonfibrous matrix components.)

Sample Summary

Sample Identification:

Conclusions

1. Asbestos present: yes no
2. Fibrous-nonasbestos component present: yes no
3. Number of distinct fibrous components:
4. Types of fibers:
5. Estimated range (percent area) of each fiber type:
6. (Optional information on nonfibrous components)

.6. QUALITY ASSURANCE

16.1 Laboratories performing this test method should have a demonstrated profiency in the method. This would include adequate training of the analyst, an internal quality assurance program and participation in the EPA's Bulk Sample Analysis Quality Assurance Program or the National Bureau of Standards Laboratory Accreditation Program for the Analysis of Asbestos. The laboratory should have a complete set of reference materials.

16.2 In order to obtain the accuracy indicated in 17.3, it is suggested that the analyst have completed a college-level course in mineralogy, had formal training in polarized light microscopy and its application to crystalline materials including instruction in the measurement of the index of refraction by the immersion method through Becke line technique and/or dispersion staining, and have experience analyzing asbestos samples. If this training is lacking, two years of participation in the EPA's Bulk Sample Analysis Quality Assurance Program with a 100% success rate is a good indication of proficiency in the application of this method.

16.3 An internal quality assurance program should involve blind samples and replicate analyses. It is also necessary to analyze blank samples to check for contamination of immersion oils, probes, slides and general sample preparation.

16.4 A record of the sample analyses should be kept that includes all the sample and analysis data. An example analysis recording form can be found in section 15.3. While the format of the record is not required, all the information detailed in the sample should be recorded for each sample.

17. PRECISION AND BIAS

17.1 The upper detection limit is 100%. The lower detection limit is less than 1%.

17.2 A preliminary evaluation of a method similar to that outlined in this document is found in Reference 11.

17.3 If used by a properly trained and experienced analyst, the accuracy in the determination of the presence or absence of greater than 1 area % asbestos is greater than 99%. If the analyst does not have the training specified in 16.2, the accuracy may be considerably reduced.

17.4 The error associated with the quantitative estimate of weight or area percent asbestos may be quite large. When the percentage of asbestos in the bulk sample is small, the error in the estimate may exceed 100% relative. Relative errors are particularly large in estimates near 1%. When the percentage of asbestos is large, however, the error is significantly reduced and may be as low as 10% relative or less. The precision and accuracy of the quantitative estimate are highly dependent on the training and experience of the analyst.

REFERENCES:

1. U.S. Environmental Protection Agency, "Interim Method for the Determination of Asbestos in Bulk Insulation Samples", EPA 600/M4-82-020, Dec. 1982.

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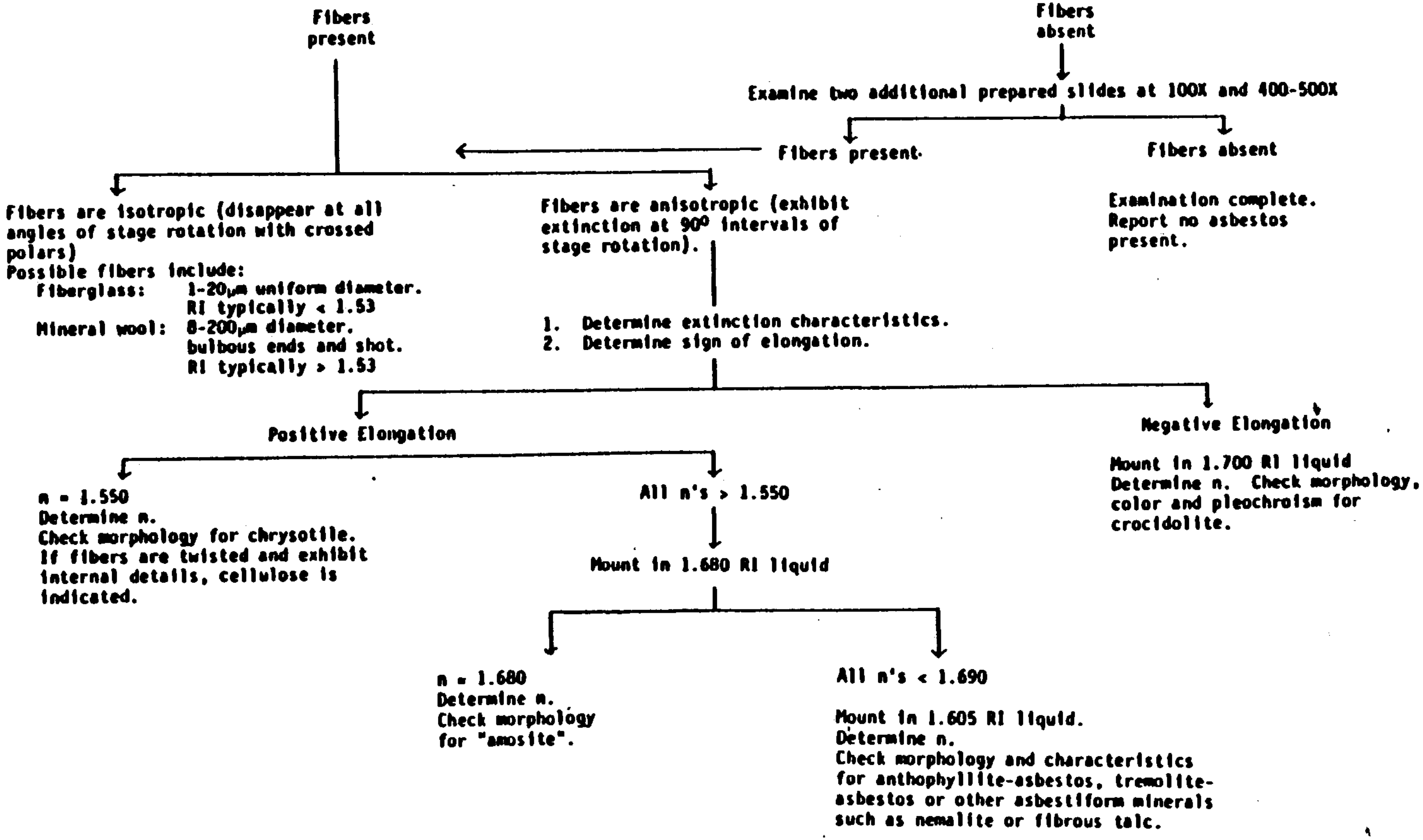
10. Zussman, Jack, "The Mineralogy of Asbestos", in Asbestos: Properties, Applications and Hazards, John Wiley and Sons, 1979, pp. 45-67.

11. U.S. Environmental Protection Agency, "Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method", EPA-600/4-82-021, May, 1982.

TABLE 1: Flow chart for qualitative analysis of bulk samples by polarized light microscopy.

Polarized light microscopy qualitative analysis: For each type of material identified by examination of sample at low magnification. Mount spacially dispersed sample in 1.550 RI liquid. (If using dispersion staining, mount in 1.550 HD.) View at approximately 100X with both plane polarized light and crossed polars. More than one fiber type may be present.



Examination complete.
Report no asbestos present.

Fibers are isotropic (disappear at all angles of stage rotation with crossed polars)
Possible fibers include:
Fiberglass: 1-20μm uniform diameter.
RI typically < 1.53
Mineral wool: 8-200μm diameter.
bulbous ends and shot.
RI typically > 1.53

Fibers are anisotropic (exhibit extinction at 90° intervals of stage rotation).

1. Determine extinction characteristics.
2. Determine sign of elongation.

Positive Elongation

n = 1.550
Determine n.
Check morphology for chrysotile.
If fibers are twisted and exhibit internal details, cellulose is indicated.

All n's > 1.550

Mount in 1.680 RI liquid

n = 1.680
Determine n.
Check morphology for "amosite".

All n's < 1.690

Mount in 1.605 RI liquid.
Determine n.
Check morphology and characteristics for anthophyllite-asbestos, tremolite-asbestos or other asbestiform minerals such as nemalite or fibrous talc.

Negative Elongation

Mount in 1.700 RI liquid
Determine n. Check morphology, color and pleochroism for crocidolite.

TABLE II

MINERAL	MORPHOLOGY & COLOR	REFRACTIVE INDICES (Approximate Values)		BIREFRINGENCE	EXTINCTION	SIGN OF ELONGATION
		‖ to Elongation	⊥ to Elongation			
Chrysotile-asbestos	Wavy fibers with "kinks" common. Large fiber buldles may show splayed ends. Colorless and non-pleochroic. Very common in building materials.	1.55	1.54	0.002-0.014	Parallel	Positive (length slow)
Cummingtonite-grunerite-asbestos (Amosite)	Straight fibers and fiber bundles. Only long fibers show curvature. Fiber bundles usually show splayed ends. Colorless to brown; may be weakly pleochroic. Common in building materials.	1.70	1.67	0.02-0.03	Parallel	Positive (length slow)
Crocidolite	Straight and curved fibers showing splayed ends are common. Blue color characteristic. Pleochroism marked. Uncommon in building materials.	1.70	1.71	0.014-0.016. Interference colors may be masked by blue color.	Parallel	Negative (length fast)
Anthophyllite-asbestos	Straight fibers and fiber bundles showing splayed ends. Colorless to light brown. Pleochroism absent. Rare in building materials.	1.63	1.61	0.013-0.028	Parallel	Positive (length slow)
Tremolite-asbestos and actinolite-asbestos	Straight and curved fibers and fiber bundles. Large bundles show splayed ends. Tremolite is colorless. Actinolite is green and weakly to moderately pleochroic. Both actinolite and tremolite are extremely rare in building materials.	1.62-1.64 (tremolite) 1.64-1.68 (actinolite)	1.60-1.62 (tremolite) 1.62-1.67 (actinolite)	0.02-0.03.	Parallel in most fibers. Narrow fibers may show oblique extinction (c∧Z up to 20°) in some samples.	Positive (length slow)

AN INTRODUCTION TO THE METHODS OF OPTICAL CRYSTALLOGRAPHY

F. Donald Bloss
Virginia Polytechnic Institute and State University

Holt, Rinehart and Winston
New York Chicago San Francisco Toronto London

Interpretation of Becke Lines. In the process of refractive index determination by the immersion method, the grain may be placed in an oil whose refractive index differs so greatly from the grain that the dispersion lines do not intersect in the visible range (Fig. 5–8A). Consequently, whether in oil I or oil II, there would be no color observed in the Becke lines at this grain's edges. When in oil I, a very intense white Becke line would enter the oil as the microscope was racked up. For the grain in oil II, the results would be reversed, the bright white Becke line entering the grain. Lack of color in the Becke line thus indicates that the dispersion curves of grain and oil do not intersect within the visible spectrum.

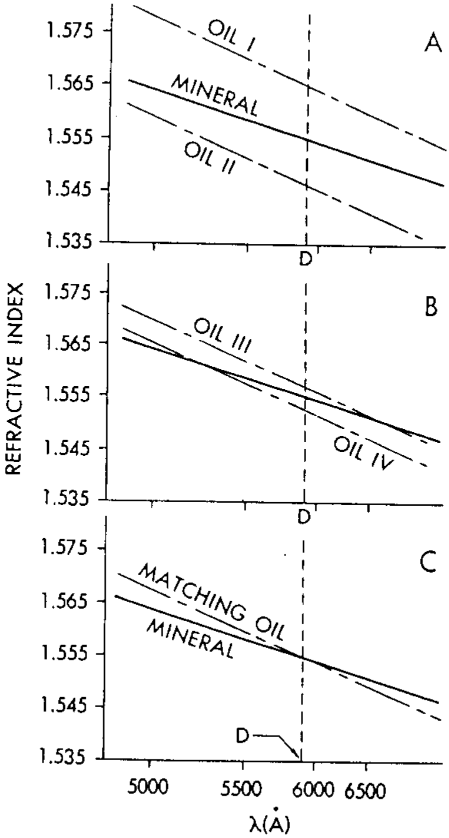


Fig. 5-8. Explanation of the movements and colors of the Becke lines according to whether (and where) the dispersion curves of the grain and oil intersect within the visible spectrum.

Consider grains of this mineral to be respectively immersed in two oils (III and IV in Fig. 5–8B) whose dispersion lines intersect that of the grain, but only at the opposite extremities of the visible spectrum. The Becke lines will now tend to develop slight colorations, although the bright Becke line are little affected. An instructive experiment is to place a grain in an oil whose index is sufficiently high so that its dispersion curve does not quite intersect that of the grain at room temperature (Fig. 5–10). Next gently warm the oil mount on a hot plate (do not boil) until a temperature such as T_5 has been attained; the oil's dispersion curve will have shifted translationally (from T_1 to T_5 in Fig. 5–10) whereas the grain's curve will hardly have shifted at all. Microscopic observation of the grain edges while the mount cools to room temperature—that is, while the oil's dispersion curve steadily shifts back from T_5 to T_4 ⋯ to T_1—will reveal the gamut of dispersion colors summarized in Fig. 5–9.

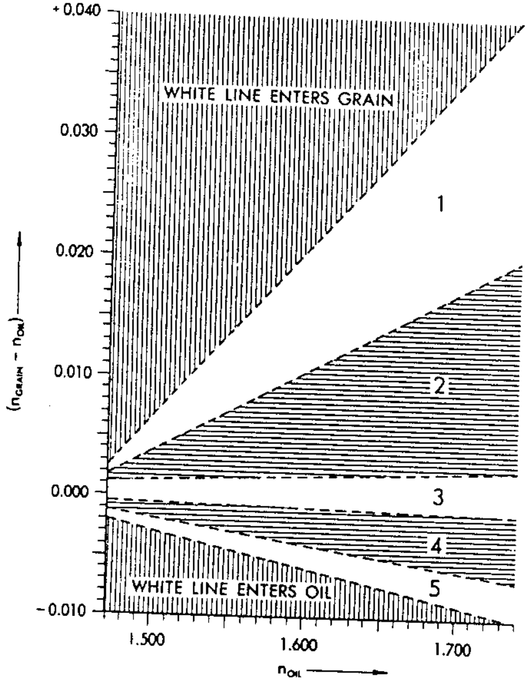


Fig. 5-9. Approximate relationship of the Becke line colors and movements to the refractive index difference (for sodium light) between grain and immersion oil (microscope racked upward). Area 1: whitish-yellow line enters grain; dark blue violet enters oil. Area 2: lemon yellow enters grain; violet blue enters oil. Area 3: orange yellow enters grain; sky blue enters oil. Area 4: reddish orange enters grain; whitish blue enters oil. Area 5: dark reddish brown enters grain; blue white enters oil.

The change of refractive index with temperature, *dn*/*dt*, varies for different oils. For immersion media of index about 1.633, *dn*/*d* are little affected. An instructive experiment is to place a grain in an oil whose index is sufficiently high so that its dispersion curve does not quite intersect that of the grain at room temperature (Fig. 5-10). Next gently warm the oil mount on a hot plate (do not boil) until a temperature such as T_5 has been attained; the oil's dispersion curve will have shifted translationally (from T_1 to T_5 in Fig. 5-10) whereas the grain's curve will

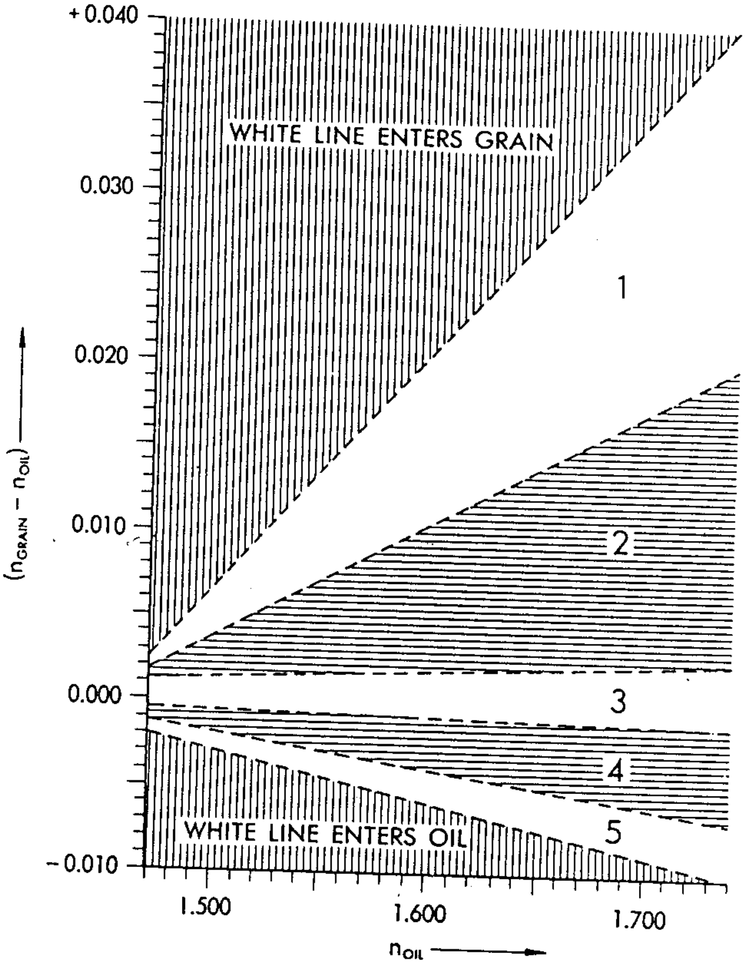


Fig. 5-9. Approximate relationship of the Becke line colors and movements to the refractive index difference (for sodium light) between grain and immersion oil (microscope racked upward). Area 1: whitish-yellow line enters grain; dark blue violet enters oil. Area 2: lemon yellow enters grain; violet blue enters oil. Area 3: orange yellow enters grain; sky blue enters oil. Area 4: reddish orange enters grain; whitish blue enters oil. Area 5: dark reddish brown enters grain; blue white enters oil.

hardly have shifted at all. Microscopic observation of the grain edges while the mount cools to room temperature—that is, while the oil's dispersion curve steadily shifts back from T to T ··· to T will reveal th approximates — 0.0004; in other words, the oil's refractive index decreases 0.0004 for each 1° C rise in the oil's temperature. For oils in the 1.739 range, *dn/dt* is usually about — 0.0007. The value *dn/dt* is generally stated on the label of each oil in the commercially available sets. Precise values for particular oils are listed in the pamphlet available with this text.

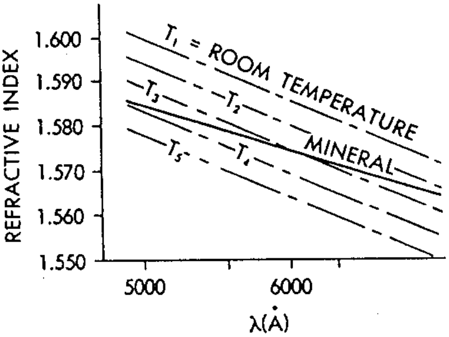


Fig. 5-10. The shift in the dispersion curve of an oil as its temperature is increased from room temperature up to T_6. In contrast, the dispersion curve of a mineral immersed in the oil undergoes no significant shift when subjected to the same temperature increase.

PRACTICAL PROCEDURES

Preparation of Mount

The unknown crystal is first pulverized and the 100 to 200 mesh size is isolated by sieving. (In the case of powdered unknowns, of course, this has already been done). The immersion oil selected should be of refractive index equal to that of the suspected identity of the crystal; if there is no clue as to identity, an oil of index 1.53 should be used. Place a drop of the oil on the slide, dislodging the drop from the glass dropper by tapping it; the dropper should never directly touch the slide, because the immersion media may become contaminated by stray mineral grains that adhere to the dropper on contact. Replace the bottle of immersion oil, *with cap tightly in place,* in the case immediately. Dust a few dozen grains of the unknown crystal onto the oildrop and cover it with a cover glass about ¼-by-¼-inch in size.

Microscopic Observations

The grains of the slide mount can now be examined between crossed nicols, using the low-power objective. If the grains are truly isotropic, they will all remain black—that is, extinct—even if observed while the stage is being rotated a full 360 degrees (see pp. 68-70). If the grains brighten, they are anisotropic and present problems of index measurement that will be discussed later. A slightly more sensitive test for anisotropism is to insert the first-order red accessory plate while conducting the preceding observation. The grain is anisotropic if, during rotation, its color varies from the violet background color produced by the plate for the surrounding oil.

Appendix 3

TEM Specific Requirements

TEM Specific Quality Requirements

Table of Content

Section 1:

Introduction to AHERA Method

The purpose of this manual is to provide an outline of the general operations and quality assurance of Analytical Environmental Services, Inc. for its Transmission Electron Microscopy Laboratory and in particular with respect to the Federal Register 40 CFR part 763, July 1, 2003 or the current US EPA TEM method for the determination of completion of response actions for asbestos abatement projects. This method will be strictly adhered to unless prior approval is obtained from the Laboratory Manager. The Manual also includes descriptions of the Laboratory's quality assurance program, staff, facilities and equipment, calibration methods, test reports, and environmental health and safety procedures. This manual will be reviewed periodically and appropriate revisions and additions made as needed on at least an annual basis.

The management of the laboratory is dedicated to the encouragement of excellence in measurement and to provide the physical and mental environment conducive to its achievement. To further these objectives, the quality assurance program described in the following sections has been established and applies to all scientific and technical work conducted, in principal and in detail, to the extent possible and feasible.

Section 2:
Facilities & Equipment

2.0 Facilities

Analytical Environmental Services, Inc occupies approximately 5000 sq.ft. There are separate rooms for each of the major instruments, a separate PLM Laboratory, a separate TEM laboratory, separate sample preparation areas, sample storage area, and adequate office space for the staff. The entire facility is well lighted and air conditioned.

2.1 Supplies and Reagents

Distilled water
Acetone
Ethanol
Glass slides (25 x 75 mm)
Frosted Glass slides (25 x 75 mm)
Scalpel with surgical steel blades
Tweezers (Dumont #5 or equivalent)
200 mesh, indexed copper grids
Glass petri dishes
Stainless steel meshed screen
Qualitative filter paper
High purity carbon rods
Tungsten wire (0.20um diameter)
Liquid nitrogen
Oxygen

All chemicals are reagent grade or better and ordered on an as-needed basis.

Laboratory blanks are prepared and analyzed regularly to monitor possible sources of contamination.

2.2 Equipment

Instrument: Philips EM420 Transmission Electron Microscope
Serial No: 2327

Instrument: EDAX Octane-T-Plus Energy Dispersive Spectrometer
Serial No: 5480

Equipment: Denton DV-502A Vacuum Evaporator
Serial No: 19664

Equipment: March Plasmod Plasma Asher
Serial No: 1352

Equipment: Captair Ductless Filtration System
Serial No: 806.208

Additional support equipment includes computers, general laboratory equipment, and instruments. The laboratory is also equipped with darkroom facility for the development of negatives and prints.

Section 3:

Standard Operating Procedures

3.1 Introduction

The purpose of the following section is to provide a uniform set of procedures for the performance of analysis of samples, sample integrity and verification of the sample data generated. Adherence to this protocol is essential and will also assist laboratory personnel in recalling and defending their actions under cross - examination if required to present court testimony in litigation. The following quality control operations are performed routinely on all samples submitted for analysis.

3.2 Sample Receipt

When a sample is received at the laboratory it is immediately given to the Sample Coordinator or the TEM Lab Manager. The shipping package is then opened in the hood located in the Sample Prep Room. Each sample container is checked for integrity and wiped with a damp cloth prior to placement in the sample prep hood.

3.2.1 Chain of Custody

If a sample is brought directly to the laboratory by a client, the client must sign a Analytical Environmental Services, Inc chain of custody form, if one is not provided by the client. This includes the name of the client, the client's representative, sample identification, date and time that the client's representative and the laboratory's representative signed the chain of custody form. If the client requests that the sample be returned it must be recorded on the chain of custody form. If a sample is received by mail or other courier service, the Sample Custodian will complete the enclosed chain of custody form if included with the samples. If the chain of custody form or field data sheet is not included with the sample(s), the sampling data sheet may be signed and dated (with the time) and used in place of a chain of custody form.

3.2.2 Sample Log In

Once the samples are closely inspected and the chain-of-custody form completed, they receive Analytical Environmental Services, Inc. identification number and the following information is recorded in the sample log in book: Document Name, Client Sample Number, Client Project Number, Sample Description, Date Received, Accept/Reject, Sample Condition, Date Sample Taken (if available), Type of Sample (Bulk, Air, Water, Tile), Type of Analysis (AHERA, Chatfield, etc.), Date Sample Analyzed, Analyst, Comments.

Each sample and its container are carefully checked by the Sample Custodian for gross features such as tears, breaks, and over all condition of the container. The condition of the sample(s) is then noted in the log-in book. Samples will be rejected for any of the following reasons:

1. Field data sheet is not correctly and completely filled out with the blanks entered on data sheet.

2. Proper number of blanks do not accompany samples.

3. Chain of custody form is not properly signed and dated.

4. Bulk samples are sent with air samples.
5. Shipping container is damaged.

6. There are open cassettes.

7. There are missing cassette end caps.

Samples that are rejected based on the above criteria are logged in as other samples, however the column in the log-in book entitled Accept/Reject should be filled in stating the reason for rejection. The sample should then be placed on hold and the client should be notified. If the rejection criteria can be corrected following a written communication or telephone conversation with the sender then it is appropriate to place the sample back in the tracking system for the completion of analysis. If acceptance criteria cannot be met then the client should be notified by telephone and if requested, the sample(s) returned with a written explanation stating the reason or rejection. A copy of the correspondence should be placed in the project folder and a final note made in the sample log-in book.

3.2.3 Client Folders

Once a sample or set of samples is logged-in, a file folder is opened for the particular sample or set of samples. The folder is labeled with the client's name and the sequential Analytical Environmental Services, Inc. project number. An Analytical Environmental Services, Inc Project Sheet (copy in appendix) is completed and stapled to the outside of the folder. Information and data pertaining to this sample or set of samples is kept in the project folder. The project folder is then placed in the open file box according to the pending analysis. In this case the file would be placed in the TEM Pending Section.

At the completion of the analysis the client project folder with all data and correspondence is taken to the administrative assistant for typing of the report. The report must be proof-read by someone other than the typist before it is mailed to the client. All reports, QA/QC records and any other information relative to the operation of the laboratory are kept indefinitely and in no case can they be discarded prior to five years after their generation.

3.2.4 Sample Storage

When the preparation of a sample is completed, the filter is returned to its original cassette and is appropriately sealed. The samples from a particular project are placed in a labeled storage bag and then into a sample storage box. The box should also be labeled to facilitate sample retrieval if necessary. Samples are kept for a period of 3 months and then disposed of by a certified hazardous waste contractor. Grids are kept for a period of 5 years.

3.3 Sample Preparation

1. Wet wipe the cassettes before placing them in the sample prep hood.

2. Clean glass slides and label them to correspond to samples: prepare one Lab Blank on each slide prepared.

3. Cut one quadrant from each filter using the razor and attach to the slide with adhesive tape.

4. Place each slide in the collapsing chamber for up to 10 minutes until filter has fully cleared.

5. Place all slides in the asher and follow posted instructions for etching 10% of the sample.

6. Carbon coat samples following the instructions provided.

7. Cut three squares from each sample (2 x 2 mm) and place on copper grids (dark shiny side of grid facing down, coated side of sample facing up).

8. Place grids on screens on the Jaffe wick, consisting of filter paper placed upon a stainless steel mesh resting on glass slides. Fill the wick with acetone and after one-hour samples should be ready for analysis.

9. Place cassettes, slides and grids in their respective storage areas.

Note: The above procedure is used for the preparation of MCE filters. As a requirement of NVLAP, the preparation of polycarbonate filters must also be included here. These filters are prepared like the MCE filters with the following exceptions:

1. Filter sections are cut with a scalpel and attached to the slide with adhesive tape.

2. There are no collapsing or etching steps.

3. Carbon coating must be performed slowly and carefully as not to allow buildup of heat.

4. Chloroform is used as the solvent rather than acetone.

5. Filter dissolution may require as long as two days. Be sure to clear the filters in a hood equipped to handle chloroform vapor.

3.4 Sample Analysis

1. Determine the number of grid square openings to be analyzed using the formula in the calculations section of the manual. One half of the total number of openings should be counted on each of the two grids.

2. Place the slide with the sample side up.

3. Fill out the information in the TEM log.

4. Refer to the instructions for turning on the TEM if you are not familiar with them and turn on the microscope. Be sure to run the EDS calibration standard as described in the QA/QC section of this manual.

5. Assess the grid at about 100x:

Reject the grid if:

a. Less than 50% of the grid is covered or less than 50% of the openings are intact.

b. The replica is doubled or folded over across 50% or more of the grid. At least 20 squares should be unobstructed.

c. Greater than 10 % of undissolved filter material remains.

d. Individual grid openings with greater than 5% holes or covered with greater than 10 % particulate matter or having obviously uneven loading shall not be analyzed.

6. Counting procedure:

a. After indexing the grid on the count sheet, increase the magnification to 21 000x.

b. At this magnification, start in the upper left hand corner of the grid and traverse from left to right. Traverses should be parallel and overlap by ten percent. Care should be taken to ensure that no area of the grid square is missed and not analyzed. The larger (5um) circle is used as a boundary and marking a particular recognizable object to determine the next field of view. After a structure is located and analyzed the analyst returns to the same field of view and continues in the original direction until the lower right hand corner is reached.

7. Counting rules:

a. Any continuous grouping of particles in which an asbestos fiber with an aspect rating greater than or equal to 5:1 and length greater than or equal to 0.5um shall be recorded and counted as an asbestos structure.

b. These will be designated as asbestos structures (after analytical techniques have proven them to be asbestos) and are classified as:

Fiber: A structure having a minimum length greater than or equal to 0.5um and an aspect ratio of 5:1 with "substantially parallel" sides.

Bundle: A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

Cluster: A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections. ("Intersection" is defined as a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.)

Matrix: Fiber or fibers with at least one end free and the other end embedded in or hidden by particulate. The exposed part of the fiber must meet the fiber definition.

8. Analytical Techniques:

Asbestos minerals can be subdivided into two types: chrysotile and amphibole. Identification of these minerals is accomplished by determination of their crystal structure through electron diffraction and their crystal chemistry through energy dispersive x-ray analysis. The following is intended as a refresher and not a complete description of how to analyze electron diffraction patterns and x-ray spectra.

3.5 SAED Pattern Identification

An understanding of the mechanics behind measuring and interpretation of diffraction patterns is required from each TEM analyst before this kind of analytical judgment can be made on the sample. Countable structures are classified as asbestos by visual examination of electron diffraction pattern on the fluorescent screen using the calibrated screen markings for layer line reflection measurements and visual recognition of characteristic reflections. Chrysotile has a layer line distance of about 0.53nm and typically shows streaks of the 1 1 0 and 130 reflections due to the cylindrical morphology. Amphiboles are characterized by layer lines formed by closely spaced reflections with a repeat distance of approximately 0.53nm. However, depending on the orientation of the structure, an amphibole has a variety of patterns and should be therefore identified as to type by zone-axis patterns if possible. Measurements of interest from such patterns are row spacing, devalues of reflections and the angle between certain reflections.

3.6 Indexing of Selected Area Electron Diffraction Patterns of Chrysotile

1. Use the Form provided in the Appendix and the following equation:

$$\text{D value (A)} = \frac{\text{Camera Constant (mmA)}}{\text{Distance (mm)}}$$

A. Measure (in millimeters) the distance "r" between the rows. Be sure that you measure perpendicular to the row axis.

B. Then measure the distance between the list of all other observable reflections on the Form. Calculate the d-value using the above equation.

C. Finally measure the angle between the undiffracted beam (center point) and the "002" spot.

Note: Frequently the "002" spots nearest the center are not easily seen. The center spot is usually difficult to locate accurately. In that case measure the distance between the next two spots (the second order spots) remembering that they are twice the "002" distance each so that you must divide the value by "4" to get the correct value.

2. The values accepted for the primary reflections of chrysotile are:

A. Row spacing: 5.17 - 5.45 Angstroms

B. 110: 4 to 5 Angstroms

C. 002: 7.06 - 7.54 Angstroms

3.7 Indexing of Selected Area Diffraction Patterns of Amphiboles

1. Reflection measurements within a horizontal row:
   A. From SAED pattern, determine the row with reflections most closely spaced, and designate this a horizontal row.

B. Draw a fine line to show the row through the origin, and designate this the 0th row.

C. Draw fine lines to show the first and second succeeding horizontal lines.

D. For a few horizontal rows, measure the mean spacing between adjacent reflections (for the minimum vector).
X_1 - Distance between reflections m units apart
where m is chose as an optimum number to minimize measurement errors.

E. The mean horizontal spot distance, (X), equals the summation of X_1 divided by the number, (n), of rows measured.

$X = \sum X_1/n$

F. The d-spacing in angstroms corresponding to this vector is the camera constant divided by X, and is labeled d2.

$d2(a) = CC/X$

2. Perpendicular Distance Between Two Adjacent Horizontal Rows

A. The inter-row spacing, (Z), is the mean separation between horizontal rows, and equals the distance between a number of rows divided by the # of spaces.

$Z = d/n$

B. This distance is an additional vector for comparison that coincides with the slant vector, d1 spacing, when angle $0_{1,2}$ is 90°.

C. The row - spacing (R) equals the camera constant divided by Z.

3. d-value and Corresponding angle $0_{1.2}$

A. Draw a perpendicular to the Oth horizontal row through the origin.

B. Draw a line to the first reflection to the right of the perpendicular in the first row and extend it through succeeding rows. This line, is called the slant vector, and forms,the acute angle $0_{1,2}$.

C. Obtain the mean spacing, (Y), between spots on the slant vector by dividing the maximum distance between reflections by the number of spaces between them, or by calculating from the inter- row spacing.

$Y = R/\sin 0_{1,2}$

D. The d-value in angstroms corresponding to this vector is the camera constant divided by Y and labeled dl.

$dI(a) = CC \text{ x } \sin O_{1,2}/R = CC/Y.$

4. Summary of Data from each SAED Pattern

   A. The use of camera constant in the form used here in calculating dl, d2 and R, which are reciprocal space on the SAED patterns, automatically converts the calculated numbers into real space spacings, which are then compared to those from a suitable standard file.
   B. The parameters of interest are:
      1. d- value of spots in a horizontal row: d2 = CC/X.

      2. d- value of spots in a slant vector: d I = CCIY.

      3. angle $0_{1,2}$ form d between a horizontal row and slant vector.

      4. d- value corresponding to row separation as an additional parameter of interest: CC/Z = R.C.

3.8 X-ray Spectra Identification

To be classified as asbestos, countable structures must display EDX spectra closely matching spectra of known standards or reference materials. A collection of spectra from these materials has been produced by equipment at Analytical Environmental Services, Inc. under similar conditions to actual analysis. By visually comparing these scans to the spectrum in question, elemental ratios of essential elements provide the means for asbestos classification.

Chrysotile: Structures identified as chrysotile must be identified by electron diffraction and x-ray analysis and recorded on the count sheet. Electron diffraction or x-ray analysis alone may be used as identification after a concentration of 70 str/mm2 has been exceeded for a particular sample. Generally this is after 4 or 5 structures have been counted.

Amphibole: Structures classified as amphiboles must be identified by electron diffraction and x-ray analysis and recorded on the count sheet. X-ray analysis alone can be used to Identify amphiboles only after a concentration of 70 str/mm2 has been exceed for a particular sample.

Refer to the standard electron diffraction patterns and x-ray analyses. If any electron diffraction patterns or x-ray spectra are recorded, it must by stated on the count sheet.

Non-asbestos: Electron diffraction or x-ray analysis alone can be used to identify non-asbestos minerals. Record their presence on the count sheet if they are significant.

Note: Separate categories will be maintained for fibers less than 5.0 um and greater than 5.0 um in length.

3.9 Recording rules:

1. Fill in all of the required information in the header of the count sheet.

2. Identify the grid being analyzed as well as the grid opening in the appropriate columns.

3. Record NSD if no structures are detected in the particular grid opening.

4. List any asbestos structures in numerical order.

5. Record the length of the structure by checking the appropriate column.

6. Record at least one image and electron diffraction pattern of a typical asbestos structure per AHERA set.
7. After analyzing the grid, return it to its place in the grid box.

3.10 Stopping Rules

1. If more than 50 asbestos structures are counted in a particular grid opening, the analysis is terminated.

2. After having counted 50 asbestos structures in a minimum of 4 grid openings, the final grid opening is completed and the analysis is terminated.

3. After reaching a point where the 5 inside samples' average concentration will be above 70 str/mm^2, the customer should be notified and the analysis terminated.

4. For blanks, the analysis is always continued until 10 grid openings have been analyzed.

5. In all other samples, the analysis shall be continued until a sensitivity of 0.005 str/cc is reached.

3.11 Calculations

1. To determine the number of grid openings to be counted for an analytical sensitivity of 0.005 str/cc:

$$\frac{385\ \text{mm}^2}{0.005\ \text{str/cc X grid opening area (mm}^2\text{) X vol. Sampled (cc)}}$$

2. To obtain the minimum detection limit (analytical sensitivity):

$$\frac{1\ \text{structure X } 385\ \text{mm}^2}{\text{\# grid openings counted X grid opening area (mm}^2\text{) X vol. sampled (cc)}}$$

3. To obtain area analyzed:
   \# grid openings counted X grid opening area (mm^2)

4. To obtain structures per square millimeter:
   $\frac{\text{\# structures counted}}{\text{area analyzed}}$

5. To obtain structures per cubic centimeter:
   Analytical sensitivity X # structures counted

6. Mass calculations for AHERA and EPA Level II analyses assume that both chrysotile and amphibole fibers have circular cross sections (cylindrical shape) and that the width measurements are one diameter. The density of chrysotile is assumed to be 2.6 g/cm^3, and amphiboles to be 3.0 g/cm^3 . The individual mass is calculated from the equation:
   Mass (ng) = 3.14 x (length, um) x (diameter, um)^2 x (density, g/cm^3) x 10^6

The total mass concentrations of fibers for each type of asbestos is then calculated from the total mass of all the individual fibers of that type.

The individual masses of bundles, clusters and matrices are calculated by assuming a laminar or sheet-like structure with an average thickness of the fiber make-up of the structure. Again, the density of chrysotile is assumed to be 2.6 g/cm^3, and the amphiboles to be 3.0 g/cm^3. The individual masses are calculated from the equation:

Mass, ng = (length, urn) x (width, um) x (thickness, um) x (density, g/cm^3) x 10^6

The total mass for each type of structure and for each type of asbestos is the sum of all the individual masses.

3.12 Clearance criteria

The area passes if the average of the inside samples is less than or equal to 70 str/mm^2. If this is not the case, the blanks are analyzed. If their average is greater than 70 str/mm^2, then there is a problem with contamination probably not associated with the airborne asbestos concentration in the abatement area. The area should be sampled again using a different lot of prescreened filters. If the blanks' average is less than or equal to 70 str/mm^2, the outside samples are analyzed and a Z-test is performed. If the result is less than or equal to 1.65, the area passes and the contractor is released. If not, the area is recleaned and resampled.

3.13 Reporting Protocol

The following information must be reported to the client for each sample analyzed.

1. Asbestos concentration (in structures per square millimeter & structures per cubic centimeter).

2. Analytical sensitivity for the analysis.

3. Number of asbestos structures detected.

4. Type of Asbestos detected.

5. Area of sample analyzed.

6. Volume of air sampled.

7. Signature of laboratory official indicating that the laboratory met the specifications of the method.

8. Pore size of filter if known.

3.14 Test Complaints

Complaints received from a client regarding the results of analysis or invoicing are directed to the Laboratory Manager. The Laboratory Manager will then review the report and establish the nature of the complaint via direct communication with the client or the client's representative. If the complaint is related to an invoicing problem then the manager will have the Administrative Assistant settle the dispute. Appropriate measures will be taken to settle complaints related to results, either by the Laboratory Manager or the Director of

Laboratory Services, if the Manager is unable to settle the dispute. If necessary, a corrective action report is prepared.

Section 4:

Personnel and Training

4.1 Key Personnel at Analytical Environmental Services, Inc

Laboratory Director: Mehmet Yildrim

TEM Quality Assurance Coordinator: Mort Soltani

TEM Laboratory Analyst : Mort Soltani and Matthew Everett

Signatories: Mort Soltani

The Administrative Assistant of the Laboratory maintains personnel folders for each staff member. Records include:

- Position Description/Job Responsibilities
- Resume of Qualifications
- Training
- Assigned Laboratory Procedures
- Results of Periodic Quality Assurance Testing
- Inter-Operator Tests and Inter-Laboratory Tests
- Accuracy and Precision Control Charts
- Correction of Deficiencies

The job descriptions for the TEM laboratory staff are in the Appendix. New technical personnel must conplete an internal training program which is specifically designed for the test methods they will perform.

4.1.5 Conflicts of Interest

Policy

It is AES, Inc. company policy to address conflicts of interest throughout our organization. Precautions are taken to ensure that there are no conflicts of interest within and between staff, the company, and its clients. Conflicts of interest can arise from supervising relatives, outside employment, fraternization between supervisors or co-workers, and/or leave of absence.

Procedures

To help enforce the policy, certain procedures must take place. Employees are instructed per the employee manual about conflicts of interest and what may constitute conflict within the company.

The following procedures are employed to address potential conflicts of interest:

Relatives

1. Employees are instructed that AES permits employment of relatives as long no direct reporting or supervisory/management relationship exists between the two. That is, no employee is permitted to work within the "chain of command" of a relative such that one relative's work responsibilities, salary, or career progress could be influences by the other relative.

2. Employees are instructed that no relatives are permitted to work in the same department or in any positions which AES believes an inherent conflict of interest may exist.

Outside Employment

1. Employees are instructed that they may hold outside jobs as long as it does not interfere with their performance, safety or ability to meet the requirements at AES.
2. Employees are instructed that they may not receive any income or material gain from individuals outside the Company for materials produced or services rendered while performing the jobs with the Company.
3. Employees are instructed that outside employment by certain parties may constitute a conflict of interest and is prohibited. Outside employment by a competitor would be a conflict of interest. Outside employment by a client may constitute a conflict of interest.

Fraternization

1. Employees are instructed that fraternization or becoming romantically involved with other employees may create a conflict of interest. These personal relationships can lead to dissension, create an unprofessional work environment, present concerns regarding supervision, safety, security, or morale.
2. Employees are instructed that an employee involved with a supervisor or fellow employee should immediately and fully disclose the relevant circumstances to their supervisor so that a determination can be made as to whether the relationship presents an actual or potential conflict of interest.

Leave of Absence

1. Employees are instructed that while on authorized personal leave of absence they may not perform work for any employer that is considered by AES to be an actual or potential conflict of interest.
2. Employees are instructed that while on authorized public service leave of absence they may not perform work for any employer that is considered by AES to be an actual or potential conflict of interest.

These examples by no means are all inclusive and AES, Inc. retains the right to review any situation it believes may be a possible conflict of interest. AES may take whatever action appears appropriate according to the circumstances, up to and including transfer or discharge. Failure to disclose facts may lead to disciplinary action, up to and including termination.

4.2 Training

Technical personnel hired at Analytical Environmental Services, Inc.are required to participate in training programs specifically related to their job classification. In-house training is provided transmission electron microscopy.

Prior to a new employee performing analyses on their own, he/she attends a course which consists of lectures, demonstrations and hands-on laboratory practice. Following the course the employee spends an additional week on a 1:1 basis with either the Quality Assurance Coordinator or TEM Laboratory Manager, who closely supervises and critiques their progress. The technician is then closely monitored for the next six months, during his/her probationary period, as to his/her ability to perform analysis independently. The course outline includes:

I. Mineralogy and Chemistry of Asbestos
II. Types of Asbestos Containing Materials
III. Principals of Air Sample Inducing Equipment and Types of Filter Media
IV. Sample Preparation
V. Instrument Operations
  - Theory
  - Applications
  - Limitations
  - Adjustments
  - Calibrations
VI. Analytical Procedures
VII. Counting
VIII. Statistical Evaluation of Data

In addition to the topics covered in the course, the analyst in training is instructed on the procedures of Analytical Environmental Services, Inc. Quality Assurance Manual. Analytical procedures are taught with an emphasis on AHERA and other approved methods. In addition to in-house training, our staff is encouraged and in some cases required to attend advanced training programs and courses.

Asbestos TEM training is intended to introduce the new employee to analysis of asbestos by the TEM method, and is, so far as possible, a "hands-on" course. The course provides theoretical background information, and also practical participation in TEM specimen preparation and fiber counting. However, competence in analysis of asbestos samples by TEM is developed largely by practical experience during which a large number of problems will be encountered and solved. It should be recognized that some other more sophisticated types of analyses related to asbestos may require extensive mineralogical and crystallographic knowledge. It should also be recognized that any asbestos analysis may have considerable legal or commercial implications. Accordingly, it is most important that the analyst understands the limitations inherent in asbestos analysis.

Section 5:
Quality Assurance and Quality Control

5.1 Contamination Control

The analysis of asbestos air samples typically involves analyzing samples with nanogram quantities of asbestos. Consequently, contamination is a critical factor in the analysis of these samples. In order to prevent and test for direct or cross contamination, the following will be adhered to by all laboratory personnel performing air asbestos analysis:

Supplies, equipment and analysts are consistently monitored to reduce the possibility of reporting erroneous results. Analyst accuracy is monitored to maintain a level better than 80% true positives (less than 20 % false negatives) and less than 10 % false positives. Laboratory blanks are prepared with each batch of samples and analyzed at least once per 25 samples analyzed. Duplicate and replicate analyses are performed at a rate such that 10% of all samples are reanalyzed known materials (such as standard reference materials) are reviewed on a regular basis. The reference materials prepared by NIST will be analyzed as required. Criteria for the acceptability of a QC analysis are in the Appendix. Results of all QC measures taken and archived.

5.1.1 Blanks

Blanks are routinely analyzed to check for contamination problems with the filters before sampling occurs. A total of ten grid square openings on two grids are analyzed.

Filter Lot Screening: Analysis of 5% of each lot of new filters received is performed on their arrival when contracted by the sampling organization. Filters are selected at random and analyzed as described above. The results must be less than 53 structures per square millimeter on any individual analysis and less than 18 structures per square millimeter on average. Filter lots not meeting these criteria are returned to the manufacturer. Air samples taken by in-house personnel should be taken on pre-screened filters, and the lot number of the filters should be noted on the Data Sheet.

Field Blanks: As described in the AHERA document, 3 blanks are required per set of clearance samples. (One inside the containment and one outside the containment are opened in the field, and one blank remains sealed until it reaches the ladpratory. The blanks are prepared along with the samples and are analyzed only if the inside samples do not pass. For field blanks, any analysis result greater than 70 structures per square millimeter is considered contaminated.

Laboratory Blanks: All reagents and sample preparation equipment may be checked for contamination by the use of laboratory blanks which are prepared simultanaously with the other samples. If acontamination problem is discovered, individual reagents, tools and laboratory equipment are checked separately to track the source of contamination. No sample results are reported to clients until the contamination problem is resolved. Laboratory blank samples should have a cumulative average of less than 18 structures per square millimeter.

5.1.2 In-House Air Sampling

Air sampling will be conducted quarterly or as needed in the TEM Prep Room and analyzed by transmission electron microscopy. This contamination control check will not only ensure the safety of laboratory staff but in addition will ensure that airborne contamination of samples does not occur. If the analysis of air samples in the TEM laboratory is greater than 0.005 asbestos fibers/cc, it will be reported to the Laboratory Director, and work in the laboratory will stop until the source of contamination is identified and the entire room cleaned by using a HEPA vacuum and a clean damp cloth. Recleaning will continue until the asbestos fiber level is <0.005 f/cc. If asbestos is detected in any monitoring schedule, is to be reported to the Laboratory Manager and QC Supervisor, the source traced and the problem resolved to prevent recurrence.

5.2 Analytical Quality Control

Samples must be routiney resubmitted for analytical quality control. All QC results must be documented in the appropriate QC data book. Specific guidelines for acceptable performance levels have been established for each technician participating in the QC program together with an over-all laboratory performance.

5.2.1 Duplicate Analysis

One sample from every 100 samples previously analyzed is re-prepped and analyzed by the same analyst. Results are tabulated and kept in the QC Log Book. Results must fall within the guidelines specified in the Appendix.

5.2.2 Replicate Analysis

One sample from every 100 samples previously analyzed and reanalyzed by the same microscopist. Results are tabulated and kept in the QC Log Book. Results must fall within the guidelines specified in the Appendix.

5.2.3 Verified Asbestos analysis

This analysis has been combined with inter-laboratory grid exchange verified analysis program outlined in 5.2.7.

5.2.4 Lab Blank analysis

One lab blank for every 100 samples previously analyzed is analyzed to detect laboratory contamination.

5.2.5 Analysis of Standards

A set of standards will be analyzed by each microscopist on an as needed basis. The conformance expectation is 100%. If discrepancies are detected, further training of the microscopist is in order. Results are recorded in the QC record book. These resullts will aid in the identification of unknown materials.

5.2.6 Analysis of NIST SRMs

SRM 1876 standard sample is no longer available.

5.2.7 NIST Proficiency Testing

The TEM laboratory participates in the NIST Proficiency program for asbestos analysis in order to meet the requirements of NVLAP. Each analyst is required to participate independently.

5.2.8 Inter-laboratory Grid Exchange Program (Round Robin)

The TEM laboratory must participate in an inter-laboratory grid exchange program with another NVLAP accredited TEM laboratory at least once per year. The results of this verified analysis can be used to calculate the relative difference R, percent true positives, false positives, and false negatives.

5.2.9 Test Discrepancies

If it is determined that a problem exists based on the criteria given in the Appendix, the sample will be reanalyzed by both the original and the QC analyst. Until the matter is resolved, the analyst is restricted from analyzing regular samples from clients. The Laboratory Director and Laboratory Manager may participate in correction of deficiencies. During this process of correction, the Director or Manager may offer guidance and suggestions to the individual in order to help him or her meet expected criteria. An outside source such as NIST or a consulting laboratory will be called on if necessary.

5.2.10 Validation of Data

Prior to the release of final reports, the signatory shall review the report and the following checks shall be made:

1. Proper sample identification.
2. Calculations are completed.
3. Transcription errors.
4. Typing errors.
5. Consistency of data, signature, etc.

In addition, data validation will be monitored by the Quality Assurance Coordinator who will randomly select 1 out of 100 client folders on a monthly basis to review the report results and verify all computer calculations by hand. The results will be recorded on the TEM Data Validation Form.

5.2.11 QC Frequency

System Check
Alignment Check
EDS Calibration
Camera Constant
Magnification Calibration
Replicate Analysis
Repeat Prep and Analysis of Sample (Same Analyst)
SRM 1876 b
SRM 2063
Data Validation
Verification of SAED
Review of field data sheets
Review of chain-of-custody
Supplies and Reagents
Grid opening size
Screen Cassette Filters

Clean air monitoring
Verified Asbestos
Analysis
Laboratory Blank
Prep
Laboratory Blank
Analysis
Record and verify
EDP

Daily
Daily
Monthly
Weekly
Monthly
1 in 100 samples
1 in 100 samples
Not Available
Annually
100%
Each sample
Each sample
Every Project
As needed

20 openings /20 grids Lot
1 in 50 received
Annually
1 per 100 grid openings
Each AHERA set
1 in 25 samples
1 in 5 positive sample

Calibration of Plasma Asher	Semi-Annually
Resolution of EDS	Annually
Beam Spot Size	Monthly
Detection of Na Peak in Crocidolite	Annually
Inter-laboratory Grid Exchange	Twice a year
Beam Dose Measurement	Monthly

5.3 Equipment Calibration and Maintenance

All equipment including cassettes, pumps, fume hoods, vacuum evaporator and electron microscope must be maintained and in proper operating condition at all times. This requires periodic inspection, cleaning, and preventive maintenance checks. Major and minor failures must be corrected as soon as they are identified.

5.3.1 Transmission Electron Microscope

I. Routine maintenance is provided as needed.
II. Alignment is checked each day before use. In accordance with instructions within this manual for alignment procedures (also within manufactures operating instructions.)
III. Check water level and quality in re-circulation unit weekly (increasing frequency during the summer months, up to daily checks). Add or replace water as needed.
IV. Filament change as needed.

5.3.2 Vacuum Evaporator

I. Maintain under vacuum at all times
II. Clean interior of bell jar every two weeks.
III. Clean diffusion pump and replace diffusion pump oil every 6 months
IV. Clean base plate and bell jar seal once each year.
V. Check gaskets values and valve seats once each year.

5.3.3 Clean Air Hood

I. Change filters as needed.
II. Vacuum interior of hood with HEPA vacuum as needed.
III. Clean interior surface with alcohol after vacuuming. (Use only lint free cloth.)

After maintenance is performed a record of the maintenance shall be recorded in the TEM maintenance log folder located in each TEM laboratory. The information shall include: type of problem; routine/non-routine; date of maintenance, in-house vs. manufacturers; the individual who performed the maintenance or service; and if the instrument was recalibrated. If no re-calibration occurred the reasons for this should be stated. If the instrument is taken out of service, the equipment repair record form must be completed by the analyst who was responsible for taking the instrument out of service.

5.4 Calibration of Analytical Equipment

To maintain accuracy, the analytical equipment must be periodically calibrated according to the requirements of the current methodology. All equipment calibrations must be performed and documented at the specified frequencies in order to allow that unit to be in service.

5.4.1 TEM Magnification

Calibration of magnification is measured on the fluorescent screen at the magnification, voltage. sample position, etc. used for fiber counting. A carbon grating replica grid is placed in the microscope so that the direction of the grating lines lie perpendicular to the pre-measured screen markings. Measured variation of <5% should be achieved. The procedure for this calibration is given in the Appendix.

5.4.2 Camera Constant

The camera constant is measured in the ED mode under the same conditions as ED patterns observation during analysis. A carbon coated grid on which a thin film of pure gold has been evaporated is used to photograph a polycrystalline ring pattern. Known d-values of the gold rings produced are then applied to determine the camera constant. The procedure for this calibration is given in the related SOP. The calibration constant is to be checked at monthly intervals and on an as needed basis especially after service to the microscope. The camera constant should not vary more than 5%.

5.4.3 EDS Calibration

Calibration of EDXS systems is necessary to make comparisons of spectra collected from samples to spectra of reference materials. The manufacturer's instructions describe the procedures for automated calibration of energy scale by collecting data from a copper and aluminum grid. After the automated calibration is complete, a printout should be obtained, initialed and filed in the QC Data book. EDXS calibration is to be checked when the microscope equipped with that system is used for analysis.

5.4.4 Plasma Asher Calibration

The time used for etching 10% of a collapsed MCE filter section is calibrated is determined on a semi-annual basis by a gravimetric method as described in the related SOP.

5.4.5 Grid Measurement System Calibration

Grid Square openings for each lot of grids (typically 1000) are sized prior to use by measuring their area under a light microscope which is used for PCM analysis. Two grids per 100 are measured for the average area (length X width) using the calibrated graticule. The average areas of 20 openings per grid determines the area for each lot.

Section 6:
References

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Section Seven:

Appendices

Appendix 1

Plasma Asher Calibration Procedure

PROCEDURE:

Calibration of the low temperature oxygen plasma asher is carried out to determine how much time is required to etch 10% of a collapsed filter. There are two methods by which this can be accomplished:

I. Place a collapsed MCE filter (prepped as a sample would be prepped) in the center of the asher. Record the time it takes for the filter to ash completely. Use 10% of this time to etch 10% of the filter.

II. Place an uncollapsed 25 mm MCE filter on a slide in the center of the asher. The time it takes to completely ash the filter is noted. This time is then used to etch 10% of a collapsed filter.

Ashing is complete when:

I. The filter shrinks and curls into a ball and suddenly ignites-the chamber will become bright and the RF will go out of tune.

II. The filter stays flat but becomes completely transparent.

To verify this calibration, a gravimetric method is employed:

I. Weigh a clean glass side
II. Collapse an MCE filter, section onto the slide and weigh again.
III. Ash the sample for the time calculated above. A 10% weight loss should have occurrend

Appendix 2

QA/QC Pass/Fall Criteria

As specified in Yamate, et al. (1984), two methods may be used to determine whether or not a QC analysis passes.

1. For less than or equal to 20 structures counted in the original analysis, use the following table to determine the 95% confidence interval for the QC analysis.

Number of Structures Counted in Original Analysis	95% Confidence Limits Lower	Upper
0	0.00	3.69
1	0.25	5.57
2	0.44	7.22
3	0.62	8.77
4	1.09	10.24
5	1.62	11.67
6	2.20	13.06
7	2.81	14.42
8	3.45	15.76
9	4.12	17.08
10	4.80	18.39
11	5.49	19.68
12	6.20	20.96
13	6.92	22.23
14	7.65	23.49
15	8.40	24.74
16	9.15	25.98
17	9.90	27.22
18	10.67	28.45
19	11.44	29.67
20	12.22	30.89

2. For more than 20 structures counted in the original analysis, use the following equation to calculate upper and lower limits: (# structures) ± (1.96 x Square root of the # of structures)

Appendix 3

Camera Constant Determination

The camera constant is determined monthly and should not significantly differ from previous readings.

1. An electron diffraction pattern is obtained from a eucentric, focused portion of the gold standard using the same condition as ED pattern observation during analysis. For calibration purposes, insert the objective aperture to the second position arid carefully note how the pattern is eclipsed by the aperture's diameter.

2. After processing, the radii of the first 4 rings are measured several times each and averaged.

3. Use the known interplianar spacings of gold as indicated on the Camera Constant Determination Sheet and calculate a value for each ring. An average of these four values is used.

4. Record the results on the control chart for the camera constant and archive the negative.

Appendix 4

Magnification Calibration

TEM magnification is calibrated monthly and should not differ significantly from previous measurements.

1. Insert the diffraction grating replica into the microscope and be sure it is eucentric and the beam is aligned properly.

2. Perform the required on-screen measurements as indicated on the Magnification Calibration Sheet.

3. Take a photo of the grating at 21000x and process the negative.

4. Perform the required measurements for Plate Magnification as indicated on the Magnification Calibration Sheet.

5. Record this information on the Magnification Calibration Control Chart and and upload a copy to the portal.

Appendix 5

Sizing Grid Square Openings

In most cases, pre-sized grids are ordered for use in AHERA analyses. If these are not available, size a new batch of grids as follows:

1. Grid openings are assessed on a phase contrast scope at 400x.
2. A stage micrometer is calibrated against an eyepiece graticoule which is then used to measure the openings.
3. Twenty grid openings on twenty randomly chosen grids are then measured per lot of 1000 grids.
4. The measurements are then averaged to obtain the area used for all calculations involving samples prepared using that batch of grids.
5. Results of these measurements are entered in the appropriate logbook.
6. If precalibrated grids are used, place the measurements provided by the supplier in the Log Book.

Appendix 6

Operating Procedure for Vacuum Coater (Denton DV-502A)

I. Start Up Procedure

Bring System Up From "Off" Condition

ALL valves are closed; ALL switches are off.

1. Turn on system power. The SYSTEM READY light should come ON.
2. **TURN ON MECHANICAL PUMP.**
3. Turn Thermocouple Gauge selector switch to the FORELINE position.
4. Thermocouple needle should indicate 50 millitorr very quickly. When it does, **OPEN BACKING VALVE**.
5. Turn on diffusion pump cooling water.
6. When FORELINE reads below 50 millitorr, **TURN ON DIFFUSION PUMP**. Wait 20 minutes for the diffusion pump to reach operating temperature. FORELINE pressure should go to less than 50 millitorr.
7. Adjust water flow to the diffusion pump so the exit temperature is approximately 100F.
8. If using LN2, it can be added at this time.

II. Operating Procedure

i. Bell Jar Loading

1. Open the CHAMBER vent valve and bring the bell jar to atmosphere.
2. Lift off bell jar.
3. Load carbon source. Adjust to desired position.
4. Position samples in bell jar.
5. Replace bell jar being careful not to hit baseplate fittings.

ii. Bell Jar Roughing

1. Check that both vent valves are closed.
2. **CLOSE BACKING VALVE**.
3. **OPEN ROUGHING VALVE**.
4. Turn "TC Select" knob to CHAMBER.

iii. Pumping Bell Jar to High-Vacuum

Check that CHAMBER pressure has been roughed to between 100 and 150 milltiorr. ONLY then:

1. **CLOSE ROUGHING VALVE**.
2. **OPEN BACKING VALVE**.
3. **OPEN MAIN VALVE** by grasping ball handle and raising upward. The bell jar is now pumping to vacuum.

iv. Evaporating

When the bell jar reaches necessary vacuum ($2x10^{-5}$ millitorr):

1. Place FILAMENT/GLOW switch in FILAMENT position.
2. Check that FILAMENT ADJUST knob is in OFF position.
3. Turn on FILAMENT/GLOW POWER switch.
4. Turn FILAMENT ADJUST knob to bring filament to red color (approx. 10 amps). Hold at this power level. Check HIGH-VACUUM gauge to see if vacuum is holding. Check ammeter to obtain ampere reading. This is outgassing the filament and evaporant. Do this for about 20 seconds or until the pressure starts dropping again. Raise power to the appropriate level to complete the evaporation (approx. 30 amps for 30-45 seconds).

v. Bell Jar Venting

1. **CLOSE MAIN VALVE**.
2. **OPEN CHAMBER VENT VALVE**. Check vacuum on FORELINE. It should remain stable. A rise in reading indicates the main valve is leaking through. If the FORELINE pressure refuses to go back to zero, contact factory for recommendations.
3. Retrieve samples and close bell jar.

vi. Roughing Bell Jar for Shutdown

1. Place bell jar on the baseplate, making certain the gasket and plate are both clean and free of debris. **CLOSE CHAMBER VENT VALVE**.
2. **CLOSE BACKING VALVE**.
3. **OPEN ROUGHING VALVE** and allow system to pump to between 100 and 150 millitorr.
4. **CLOSE ROUGHING VALVE.**
5. **OPEN BACKING VALVE**.
6. **OPEN MAIN VALVE** for about 30 seconds to bring the bell jar to high vacuum.
7. **CLOSE MAIN VALVE.**

III. Shutting System Down

1. If the system is open to air, place the bell jar on the baseplate and rough down to 100 microns. Pump to high vacuum as above if time permits. (The bell jar should ALWAYS be left under vacuum when not in use.)
2. **CLOSE MAIN VALVE** if open.
3. With **ONLY** the backing valve open, **TURN OFF DIFFUSION PUMP**. Allow 10 to 20 minutes for the diffusion pump to cool.
4. **CLOSE BACKING VALVE**.
5. **TURN OFF MECHANICAL PUMP**.
6. **VENT MECHANICAL PUMP**.
7. Turn off system power.
8. Close diffusion pump cooling water valve.

Notes:

1. **NEVER** have both the backing and roughing valves open at the same time. Always close one before opening the other.
2. **NEVER** open the main valve unless:
    a. The chamber pressure is between 100 and 150 millitorr or less;
    b. The backing valve is open.
3. **NEVER** open chamber vent with main valve open **and** the diffusion pump hot.
4. **NEVER** open mechanical pump vent with the backing valve

Appendix 7

TEM Job Descriptions

10.1 TEM Laboratory Manager:

This individual is responsible for the overall operation of the TEM laboratory. He/she shall perform or oversee the preparation and analysis of all the samples submitted to the laboratory. All QA/QC activities, training, and acquiring and maintenance of the certifications shall be performed by the TEM Laboratory Manager

10.2 TEM Analyst:

The TEM analyst prepares and analyzes routine TEM samples in accordance with this manual and all the pertinent regulations. The analyst ensures that any equipment used is properly calibrated, perform routine calibrations and document any QA/QC activities performed. Any problems or questions related to their responsibilities are directed to the TEM Laboratory Manager. In addition, the TEM analyst is responsible for the log in and data processing of all TEM samples received by the laboratory.

Appendix 8

A copy of typical TEM analysis sheet is attached.

GEO-ANALYTICAL SERVICES, INC.
TRANSMISSION ELECTRON MICROSCOPY DATA SHEET

TEMID:	**4594**	SampleID:	**1**	Analyst:	
Client Name:	Kemron Environmental Services	Location:	Inside work area	Date Analyzed:	
Instrument:	Philips 400T	Grid ID:		# op. read:	
Magnification:	22,000X	Grid Open. Area:	0.0121 mm2	Alignment checked:	Yes No
Acc. Voltage:	80kV	Volume:	1200	Grid Criteria:	Accepted Rejected

Grid ID	GO	Str #	Str Type	Length		ED Observation				EDXA Profile						Asbestos Type	Comments
				<5um	>5um	CHR	AMPH	AMBIG	Photo#	Na	Mg	Si	Ca	Fe	Spectra#		

Grid Criteria	Particle Loading	(<10%) Yes No	Overlapping/Folded	(<50%) Yes No	Opaques	(<5%) Yes No
	Intact Portion	(>50%) Yes No	Holes	(<5%) Yes No	Undissolved Filter	(<50%) Yes No

Appendix 9

A copy of an individual sample report is attached.

ANALYTICAL ENVIRONMENTAL SERVICES, INC.
3080 Presidential Drive
Atlanta, GA 30340
Tel: (770) 457-8177
Fax: (770) 457-8188

AES

NVLAP Lab Code 102082-0

AES Project ID: T20371

Page 1 of 1 Total Samples

TRANSMISSION ELECTRON MICROSCOPY ANALYSIS REPORT

Project Name: [redacted]

Client Name: [redacted]

AES TEM ID: 25183

Date Received: 5/7/2016

Client Sample ID: 101Z

Date Analyzed: 5/9/2016

Sample Location: KITCHEN

ANALYTICAL INFORMATION

Magnification:	17000X	Number of Openings:	4
Total Volume:	1812 liters	Average Opening Area:	0.0121mm2
Analytical Sensitivity:	0.004s/cc	Total Area Analyzed:	0.0484mm2
Filter Type: MCE 0.45 μ			

STRUCTURE INFORMATION

Chrysotile:	**Amosite:**	**Crocidolite:**	**Other:**
0	0	0	0

Asbestos Concentration:	<	**0.004s/cc**
	<	**20.661s/mm2**

These test results apply only to those samples actually tested, as submitted by the client. This sample has been analyzed in accordance with the AHERA protocol, using a Philips 400T transmission electron microscope. For asbestos concentration of less than analytical sensitivity (no asbestos detected), the corresponding one-sided upper 95% confidence limit is 2.99 times the reported analytical sensitivity.

A copy of the analysis count sheet for this sample which may contain additional information about break down of structure length and SAED micrograph numbers will be provided upon request.

It is certified by the signature below that the laboratory identified is accredited by the National Insititute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Lab No. 102082-0, for Transmission Electron Microscopy (TEM) analysis. This report must not be used by the client to claim product endorsement by NVLAP or any agency of the US Government.

Microanalyst:

Mortesa Soltani

Appendix 10

A copy of a typical TEM summary report is attached.

Date: 5/7/2016

AES Project ID: T20371

AES

ANALYTICAL ENVIRONMENTAL SERVICES, INC.
Transmission Electron Microscopy Summary Report

NVLAP Lab Code 102082-0

Company Name: [redacted]
Project Name: [redacted]
Attention:

			Structure Data				Asbestos Concentration				
AES ID	Client ID	Location	Chr	Am	Cr	Oth	Sensitivity		s/cc		s/mm2
25183	101Z	KITCHEN	0	0	0	0	0.004	<	0.004	<	20.661

These test results apply only to those samples actually tested, as submitted by the client. Results reported are dependent on the sample collection procedures and the volume of air sampled. The laboratory is not responsible for the sample collection procedures and they are not covered by the laboratory accreditation. Samples have been analyzed in accordance with the AHERA protocol. It is certified by the signature below that Analytical Environmental Services, Inc. is accredited by the National Institute of Standards and Technology's NVLAP, Lab No. 102082-0.

Microanalyst:

Mortesa Soltani

Page 1 of 1

Appendix 11

Test Discrepancies

In the case of any test discrepancies the Summary of Contamination Assessment and Resolution Form is used and filled out. See Section 7.A12 for an example of this form. These forms are kept in a notebook in the laboratory.

The assessments of contamination during sample preparation consist of:

1. Prescreen examination of reagents prior to being used to sample preparation.
2. Assessment of sample collection using field blank analyses.
3. Assessment of sample preparation using lab blank analysis.
4. Assessment of sample preparation environment.
5. Assessment of plasmta asher and carbon coater.

A summary of these assessments is performed and recorded monthly and stored in the respective notebooks in the library. The resolution for the contamination detected in each assessment is recorded on the Summary of Contamination Assessment And Resolution form.

Appendix 12

Analytical Method for EPA Level II Analysis

Al6.1 Choosing a Grid Opening

Place the grid into the sample holder such that the sides of the indexed grid are parallel to the sides of the sample holder. At low magnification (100X) randomly choose a grid opening ard note it on the sample analysis sheet (see Section 7.A12 for an example of an analysis sheet).

Al6.2 Counting and Sizing Rules

1. The analysis, morphology, and SAED are performed at a tilt angle of 0 degrees.

2. Since asbestos fibers are found isolated as well as with each other or with other particles in varying arrangements, the fibrous particulates are characterized as asbestos structures:
   Fiber: a particle with an aspect ratio of 3:1 or greater with substantially parallel sides.

   Bundle: a particulate composed of fibers in a parallel arrangement, with each fiber closer than the ameter of one fiber.

   Cluster: a particulate with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

   Matrix: a fiber or fibers with one end free and the other end embedded or hidden by a particulate.

   Combinations of structures, such as matrix and cluster, matrix and bundle, etc. are categorized by the dominant fiber quality.

3. Counting rules
   A. Particulates meeting the definition of fiber are isolated by themselves.

   B. Count as single structure if separation is equal to or greater than the diameter of single fiber.

   C. Count as single structures if three ends can be seen.

   D. Count as single entities if four ends can be seen.

   E. In general, fibers that touch or cross are counted separately.

   F. Two or more fibers are counted as a bundle if the distances between fibers are less than the diameter of a single fiber, or if the ends cannot be resolved.

   G. Fibrils attached longitudinally to a fiber are counted as part of the fiber and the size (width) is estimated based on the fiber-to-fibril relationship.

   H. A fiber partially hidden by the grid is counted, but labeled an X-fiber in the structure column.

4. Sizing rules for asbestos structures
   A. For fibers width and lengths are obtained by orienting the fibers to the

inscribed circled the fluorescent screen. Since estimates are within ±l mmm, small diameter fiber have greater margins of error. Fibers less than I mm at the fluorescent screen level are characterized as being 1 mm. X-fibers are sized by measuring their entire visible portion.

B. Bundles and clusters are sized by estimating their average length.

C. Matrices are sized by adding the best estimates of individual fiber components.

5. The method of sizing
   - A. An asbestos structure is recognized, and its location on the screen relative to the inscribed ci les and center rectangle, is memorized.
   - B. The structure is moved to the center for SAED observation and sizing.
   - C. Sizing is performed using the inscribed circles, the central rectangle and the scale under the rectangle. If a structure extends beyond the screen width, it is superimposed across the series of concentric circles until the entire structure has been measured.
   - D. The structure is returned to its original location and scanning continues.

A16.3 Analytical Procedure

1. The grid is examined at 100X.

2. Particulate loading is examined at 100X.

3. A grid opening is selected at random and noted on the count sheet.

4. A series of parallel traverses is made across the grid opening at the film magnification of 21000X. Starting at one corner, and using the tilting section of the fluorescent screen as a "gate" or "chute", the grid opening is traversed. On reaching the end of a traverse, the image is moved the width of one "gate" and the traverse is reversed. These parallel traverses are made until the entire grid opening has been scanned.

5. Asbestos structures are identified morphologically and counted.

6. The asbestos structure is categorized as fiber, X-fiber, bundle, cluster, matrix, and sized.

7. The structure is centered and focused, and the SAED pattern obtained. SAED pattern from single fibers of asbestos mineral fall into distinct groups.
   - A. The chrysotile asbestos pattern has characteristic streaks on layer lines other than the central line, and some streaking also on the central line. Spots of normal sharpness are present on the central layer line and on alternate lines (2nd, 4th, etc.) The repeat distance between layer lines is about 0.53nm. A picture of the typical chrysotile diffraction pattern is shown in figure A16.7.A.
   - B. Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines of about 0.53nm. Streaking in layer lines is occasionally present due to crystal structure defects. Layer lines are perpendicular to the fiber.
   - C. Transmission electron micrographs and SAED patterns obtained with asbestos standard samples should be used as guides to fiber identification.

See the Morphology, SAED, and Spectra of Chrysotile and Amphiboles notebook and Identification of Interfering Minerals notebook.

D. After a structure has been identified as amphibole by SAED, the next step is to get an EDXA spectrum.

8. From visual examination of the SAED pattern, the structure is classified as belonging to one of the following groups: chrysotile, amphibole, ambiguous, no identification. SAED patterns cannon be inspected for some fibers. Reasons for the absence of a recognizable diffraction pattern include contamination of the fiber, interference from nearby particles, fibers that are too small or too thick, and nonsuitable orientation of the fiber. Some chrysotile fibers are destroyed in the electron beam, resulting in patterns that fade away within seconds of being formed. Some patterns are very faint and can be seen only under the binocular microscope. In general, the shortest available camera length must be used, and the objective lens current may need to be adjusted to give optimum pattern visibility for correct identification.

9. The specimen holder is tilted to 34 degrees for optimum X-ray detection. The categorized asbestos structure is maintained in its centered position for X-ray analysis by means of the z-control. See Section 7.A19 for the procedure for centering the Z-axis.

10. The spot size of the electron beam is reduced and stigmated to overlap the fiber.

11. The EDS is used to obtain a spectrum of the X-rays generated by the asbestos structure.

12. The profile of the spectrum is compared with profiles obtained from asbestos standards: the best match identifies and categorizes the structure. The image of the spectrum may the printed out, or the peak heights (Na, Mg, Si, Ca, Fe) recorded for normalizing at a later time.

13. The specimen holder is returned to 0 degrees tilt to examine other asbestos structures.

14. Scanning is continued until all structures are identified, measured, analyzed, and categorized in the grid opening.

15. Additional grid openings are selected, scanned, and counted in the same manner until either the total number of structures counted exceeds 100 per known area, or a minimum of 10 openings have been scanned, whichever comes first.

16. The TEM data is recorded in a systematic form .

Appendix 13

Analytical Method for AHERA Analysis

A17.1 Choosing a Grid Opening

Place the grid into the sample holder such that the sides of the indexed grid are parallel to the sides of the sample holder. At low magnification (10OX) randomly choose a grid opening and note it on the sample analysis sheet (see Section 7.A12 for an example of an analysis sheet).

Al 7.2 Counting and Sizing Rules

1. The analysis, morphology, and SAED are performed at a tilt angle of 0 degrees.

2. Since asbestos fibers are found isolated as well as with each other or with other particles in varying arrangements, the fibrous particulates are characterized as asbestos structures:

   Fiber: a particle with an aspect ratio of 5:1 or greater with substantially parallel sides.

   Bundle: a particulate composed of fibers in a parallel arrangement, with each fiber closer than the diameter of one fiber.

   Cluster: a particulate with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

   Matrix: a fiber or fibers with one end free and the other end embedded or hidden by a particulate.

   Combinations of structures, such as matrix and cluster, matrix and bundle, etc. are categorized by the dominant fiber quality.

3. Counting rules
   A. Particulates meeting the definition of fiber are isolated by themselves.

   B. Count as single structures if separation is equal toor greater than the diameter of a single fiber.

   C. Count as single structures if three ends can be seen.

   D. Count as single entities if four ends can be seen.

   E. In general, fibers that touch or cross are counted separately.

   F. Two or more fibers are counted as a bundle if the distances between fibers are less than the diameter of a single fiber, or if the ends cannot be resolved.

   G. Fibrils attached longitudinally to a fiber are counted as part of the fiber and the size (width) is estimated based on the fiber-to-fibril relationship.

H. A fiber partially hidden by the grid is counted, but labeled an X-fiber in the structure column.

4. Sizing rules for asbestos structures
    A. For fibers, widths and lengths are obtained by orienting the fibers to the inscribed circled on the fluorescent screen. Since estimates are within ±1mmm, small diameter fiber have greater margins of error. Fibers less than 1 mm at the fluorescent screen level are characterized as being 1 mm. X-fibers are sized by measuring their entire visible portion.

    B. Bundles and clusters are sized by estimating their average length.

    C. Matrices are sized by adding the best estimates of individual fiber components.

5. The method of sizing
    A. An asbestos structure is recognized, and its location on the screen relative to the inscribed circles and center rectangle, is memorized.

    B. The structure is moved to the center for SAED observation and sizing.

    C. Sizing is performed using the inscribed circles, the central rectangle and the scale under the rectangle. If a structure extends beyond the screen width, it is superimposed across the series of concentric circles until the entire structure has been measured.

    D. The structure is returned to its original location and scanning continues.

A17.3 Analytical Procedure

1. The grid is examined at 100X.

2. Particulate loading is examined at 1000X.

3. A grid opening is selected at random and noted on the count sheet.

4. A series of parallel traverses is made across the grid opening at the film magnification of 21000X. Starting at one corner, and using the tilting section of the fluorescent screen as a "gate" or "chute", the grid opening is traversed. On

    reaching the end of a traverse, the image is moved the width of one "gate" and the traverse is reversed. These parallel traverses are made until the entire grid opening has been scanned.

5. Asbestos structures are identified morphologically and counted.

6. The asbestos structure is categorized as fiber, X-fiber, bundle, cluster, matrix, and sized.

7. The structure is centered and focused, and the SAED pattern obtained. SAED pattern from single fibers of asbestos mineral fall into distinct groups. See Section 7.A5.3.

    A. The chrysotile asbestos pattern has characteristic streaks on layer lines other than the central line, and some streaking also on the central line. Spots of normal sharpness are present on the central layer line and on alternate lines (2nd, 4th, etc.) The repeat distance between layer lines is about 0.53nm. A picture of the typical chrysotile diffraction pattern is shown in figure A16.7.A.

B. Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines of about 0.53nm. Streaking in layer lines is occasionally present due to crystal structure defects. Layer lines are perpendicular to the fiber.

C. Transmission electron micrographs and SAED patterns obtained with asbestos standard samples should be used as guides to fiber identification. See the Morpholooy, SAED, and Spectra of Chrysotile and Amphiboles notebook and Identification of Interfering Minerals notebook.

D. After a structure has been identified as amphibole by SAED, the next step is to get an EDXA spectrum.

8. From visual examination of the SAED pattern, the structure is classified as belonging to one of the following groups: chrysotile, amphibole, ambiguous, no identification. SAED patterns cannot be inspected for some fibers. Reasons for the absence of a recognizable diffraction pattern include contamination of the fiber, interference from nearby particles, fibers that are too small or too thick, and nonsuitable orientation of the fiber. Some chrysotile fibers are destroyed in the electron beam, resulting in patterns that fade away within seconds of being formed. Some patterns are very faint and can be seen only under the binocular microseope. Ln general, the shortest availablecamera length must be used, and the objective lens current may need to be adjusted to give optimum pattern visibility for correct identification.

9. The EDS spectra is obtained. See Section 7.A5.5.

10. The spot size of the electron beam is reduced and stigmated to overlap the fiber.

11. The EDS is used to obtain a spectrum of the X-rays generated by the asbestos structure. See section 7.A19 for a detailed procedure.

12. The profile of the spectrum is compared with profiles obtained from asbestos standards, the best match identifies and categorizes the structure. The image of the spectrum may the printed out, or the peak heights (Na, Mg, Si, Ca, Fe) recorded for normalizing at a later time.

13. The specimen holder is returned to 0 degrees tilt to examine other asbestos structures.

14. Scanning is continued until all structures are identified, measured, analyzed, and categorized in the grid opening.

15. Additional grid openings are selected and analyzed until an analytical sensitivity

of 0.005 s/cc is attained. See Section 3.11 for the calculations.

16. Positive identification of asbestos structures is required. At least one diffraction pattern of each structure type from every five samples must be recorded and compared with a standard diff raction pattern. For each asbestos structure reported, the analytical functions, such as morphology, SAED, and/or EDS, used for identification is recorded on the count sheet.

17. Structures classified as chrysotile must be identified by diffraction pattern and/or X-ray analysis and recorded on the count sheet. X-ray analysis alone can be used as sole identification only after 70s/mm^2 has been exceeded for a particular sample.

18. Structures classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. X-ray analysis alone can be used for sole identification only after 70s/mm^2 has been exceeded for a particular sample.

19. If a diffraction pattern is recorded on film, the micrograph number recorded in the Micrograph Log must be recorded on the count sheet.

20. If and electron diffraction is attempted and an appropriate spectrum is not obtained, the structure is recorded as non-asbestos.

21. If an X-ray analysis is attempted but not observed, the structure is recorded as non-asbestos.

22. If an X-ray analysis spectrum is stored, the file and disk number is recorded on the count sheet.

23. The TEM data is recorded in a systematic form .

A17.4 Stoppirig Rules

1. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.

2. After having counted 60 asbestiform structures in a minimum of 4 openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.

3. Blank sample analysis is continued until 1 0 grid openings have been scanned.

4. In all other samples the analysis will be continued until an analytical sensitivity of 0.005 s/oc is reached.

Appendix 14

Differentiating Between Chrysotile, Amphiboles, and Non-Asbestos Structures

A19.1 Chrysotile

1. Morphology: tubular. See Table A19.1.1 for classification of fibers with tubular morphology.

2. EDAX: Na:Mg:Si:Ca:Fe = 0:7:10:0:0

3. Qualitative SAED: Foe identification as chrysotile, the (002) reflections must exhibit d-spacings of approximately 0.73nm, the pattern should have a row spacing of 0.53nm, ard the should be streaking of the (110) and (130) reflections. A copy of a typical chrysotile SAED pattern is found in Table A19.1.2

4. Quantitative SAED: This is not necessary for the analysis of chrysotile, since the fibrils are scrolled and all orientations will give the same SAED.

5. Chemical formula: $Mg_3,(Si_2O_5)(OH)_4$

TableA19.1.1. Classifcation of Fibers with Tubular Morphology

TM	Tubular morphology not sufficiently characterisfic for classification as chrysofile
GM	Characterisfic chrysotile morphology
CD	Chrysofile SAED pattern
CQ	Chrysotile composition by quantitative EDXA
CMQ	Chrysotile morphology and compostion by quantitative EDXA
CDQ	Chrysotile SAED pattern and composibon by quanfitafive EDXA
NAM	Non-asbestos mineral

A19.2 Amosite

1. Morphology: blocky with flat ends and having parallel or stepped sides. See Table A19.2.1 for classification of fibers without tubular morphology.

2. EDAX: Na:Mg-Si:Ca:Fe = 0:2:10:0:7

3. Qualitative SAED: The SAED pattern obtained from a randomly oriented amphibole asbestos fiber is not specific for amphibole minerals. Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines of about 0.53nm. Streaking in layer lines is occasionally present due to crystal structure defects.

4. Quantitative (or Zone Axis) SAED: See Morphology, SAED, and Spectra of Chrysotile and Amphibales notebook and Section 7.A20.

5. Chemical formula: $(Fe, Mg)_7(Si_8O_{22})(OH)_2$

Table A19.2. 1. Classification of Fibers Without Tubular Morphology

UF	Unitentifled fiber
AD	Arnphibole by random orientation SAED (shows layer pattern of 0.53nm spacing)
AX	Amphibole by qualitative EOXik Spectrum has elemental components consistent with amphibole
AOX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by quanitative EDXA AZ by zone axis SAED
ADQ	Amphibole by random orientation SAED and quanitative EOXA
AZQ	Amphibole by zone axis SAED and quanitative EDXA
AZZ	Amphibole by two zone axis SAED patterns with consistent inter-axial angle
AZZQ	Amphibole by two zone axis SAED patterns with consistent inter-axial angle and quanitative EDXA
NAM	Non-asbestos mineral

A19.3 Tremolite-Actinolite

1. Morphology: blocky with flat ends and having parallel or stepped sides.

2. EDAX: Na:Mg,.Si:Ca:Fe = 0:4:10:3:<l

3. Qualitative SAED: The SAED pattern obtained from a randomly oriented amphibole asbestos fiber is not specific for amphibole minerals. Amphibole asbestos fiber pattern show layer lines formed by very closely spaced dots, and have repeat dista ices between layer lines of about 0.53nm. Streaking in layer lines is occasionlly present due to crystal structure defects

4. Quantitative (or Zone Axis) SAED: See Morphology, SAED, and Spectra of Chrysotile and Amphiboles notebook and Section 7.A20.

5. Chemical formula: $Ca2(Mg, Fe)_5 (Si_8O_{22})(OH)_2$

A19.4 Anthophyllite

1 Morphology: blocky With flat ends and having parallel or stepped sides.

2 EDAX: Na:Mg:Si:Ca:Fe-=0:3:10:0:1

3 Qualitative SAED: The SAED pattern obtained from a randomly oriented amphibole asbestos fi Der is not specific for amphibole minerals. Amphibole asbestos fiber pattern show layer lines formed by very closely spaced dots, and have repeat dista ices between layer lines of about 0.53nm. Streaking in layer lines is occasionally present due to crystal structure defects

4 Quantitative (or Zone Axis) SAED: See Morphology, SAED, and Spectra of Chrysotile and Amphiboles notebook and Section 7.A20.

5 Chemical formula: $(Mg, Fe)_7(Si_8O_{22})(OH)_2$

A19.5 Crocidolite

1 Morphology: blocky with flat ends and having parallel or stepped sides.

2 EDAX: Na:Mg:Si:Ca:Fe= 1:1:10:0:6

3 Qualitative SAED: The SAED pattern obtained from a randomly oriented amphibole asbestos fiber is not specific for amphibole minerals. Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines of about 0.53nm. Streaking in layer lines is occasionally present due to crystal structure defects

4 Quantitative (or Zone Axis) SAED: See Morphology, SAED, and Spectra of Chrysotile and Amphiocies notebook and Section 7.A20.

5 Chemical formula: $Na_2Fe^{+2}{}_3Fe^{+3}{}_2(Si_8O_{22})(OH)_2$

Appendix 15

Zone Axis Analysis

Quantitative (or Zone Axis) SAED: requires alignment of the fiber so that a principal crystallographic axis is parallel to the electron beam. The pattern is then recorded and its consistency with known mineral structures is checked by comparing to computer data or by using a computer program. The SAED pattern obtained from one zone axis may not be sufficiently specific to identify the mineral fiber, but it is often possible to tilt the fiber to another angle and to record a different zone axis pattern. The angle between the two axes can also be checked for consistency with the structure of a suspected mineral. It is of extreme importance that, when obtaining an SAED pattern for either recording or visual evaluation, the sample height be properly adjusted to the eucentric point and the image be focused in the plane of the selected area aperture.

If a zone axis SAED analysis is to be attempted on the fiber, the sample must be in the appropriate holder. The holder should be able to do single axis tilting. Rotate the sample until the fiber image indicated that the fiber is oriented with Its length coincident with the tilt axis of the goniometer, and adjust the sample height until thefiber is at the eucentric position. Tilt the fiber until a pattern appears which is symmetrical, two dimensional array of spots.

Five spots, closest to the center spot, along two intersecting lines of the zone axis pattern must be selected for measurement. The distance of these spots from the center spot and the four angles are the input for the computer program or hand calculationln. The required distances must therefore be obtained by measuring between pairs of spots symmetrically disposed about the center spot, preferable separated by several repeat distances. The distances must be measured within a precision of 0.3mm, and the angles better than 2.5 degrees.

If a computer program is not available, the following method must be used for hand calculation of zone axis. Using the camera constant, calculate the d-spacingsforthefivespots. Use JCPDS files of amphiboles to designate (hkl) indices to each spot. Use the lattice parameters of the suspected amphibole and the related equations to generate and verify the zone axis. However, this method is extremely ttedious and time consuming and should be attempted only as the last option. The alternative to this method is to match the data obtained from hand calculation (Miller indices and angle) to the data generated by the computer program at NVLAP and kept in the related notebook.

Appendix 16

Revised Procedure for Determination of Precession of TEM Analyst and Laboratory

The method for calculation of precession of analyst and laboratory from duplicate and replicate analysis has been modified using New York ELAP procedure. The determination of accuracy will not be affected by this revised method.

Definition of Accuracy:
The accuracy of a measurement is the difference between the average of several measurements and the true value or the degree of agreement of a measured value with the true or expected value of the quantity of concern. The accuracy of TEM asbestos analysis is determined by analysis of reference materials, verified analysis, and NIST proficiency testing.

Definition of Precision:
The closeness of agreement between randomly selected individual measurements or test results. It is also defined as repeatability of the results of a particular experiment or the degree of agreement among repeated measurements of the same quantity. This is determined by replicate and duplicate analysis of same TEM sample.

A. Duplicate analysis:
The reanalysis of the same grid openings by different analysts will be used to determine both the laboratory's overall precession and to detect bias on the part of different analysts.
Relative difference (R) values will be calculated for each pair of re-analyses and will be statistically evaluated for four ranges of average structure counts: <1, 1 to 4.9, 5 to 20, and >20. For duplicate analysis, R-values will be calculated for each analyst comparing his/her results to results from other analysts using the following equation:

$$R= (A-B)/((A+B)/2)$$

Where:
A=Result from the original analyst
B= Result from other analyst for same sample/grid openings

Range of Mean of Recount	Acceptable Agreement
<5 Structures	1 structure
5 to 20 Structures	2 Structures
>20 Structures	3 Structures

B. Replicate Analysis:

The reanalysis of the same grid openings by the same analyst will be used to determine the analyst's precession. Relative difference (R) will be calculated for each pair of reanalysis and will be statistically evaluated for four ranges of average structure counts: <1, 1 to 4.9, 5 to 20, and >20.

For replicate analysis, R-values will be calculated for that analyst, comparing his/her results to his/her previous results using the following equation:

$$R= / (A-B)/((A+B)/2)/$$

Where:
A=First result from the analyst being checked
B= Second result from same analyst
Note: The value of R is an absolute value

The cumulative value of (R) will be determined for each analyst and laboratory and control charts (R-charts) will be constructed to show the performance of each analyst and the laboratory.

Reference:
Environmental Laboratory Approval Program, Certification Manual, New York State Dept. of Health, Item No. 236, 3/1/1997.

Appendix 17

Laboratory Policy Regarding Departures from Standard Procedures and Methods

According to NVLAP Handbook 150-13 there is a need for a written policy regarding deviations and departures from documented procedures and methods. The following are the most common deviations:

1. Indirect preparation of an overloaded filter will be performed only upon approval from the client.
2. Termination of the analysis before 50 asbestos fibers are counted must be by approval from the client.
3. The client will be notified if a sample is coated with encapsulant.

Appendix 18

Review of requests, tenders and contracts (NVLAP Section 4.4)

Review of New Work

The Laboratory Manager is primarily responsible for determining the capacity of the facility and its resources to handle new work, although other senior members of management may be called upon to provide expertise and input as needed. This determination consists of a comprehensive appraisal of the client's projected needs. Factors assessed are the ability of the laboratory to comply with the requirements of its accreditations while maintaining the expected level of legal defensibility and analytical validity of all reported data.

Prior to the acceptance of any new requests, tenders, or contracts by Analytical Environmental Services, Inc., the appropriateness of facilities and resources is considered utilizing the information in the following sections. If the facility and/or resources are inadequate to perform the work, the Laboratory Manager may exercise his discretion to refuse to perform all or part of a particular project. The Client Services Manager will be informed of this decision and the Project Managers will inform the client.

Facilities

The facility must be suitable for the proper receipt and storage of the number and type of samples proposed to be accepted.

The specified methods must be adequately defined, documented, and understood.

Resources

Stipulated methods, sample preparations, final reports, data packages, and deliverables are reviewed to determine the availability of suitable instrumentation and personnel.

The laboratory must be must be capable of meeting all analytical requirements for the selected test methods.

The laboratory shall advise and obtain approval from the client before subcontracting work to another laboratory.

Technical and Management Capability

The review of capability must establish that the laboratory possesses the necessary physical personnel, information, and resources to perform the tests in question. Additionally, the laboratory personnel must have the skills and expertise required for performing these tests.

The laboratory shall have adequate personnel at all times during the performance of analytical testing to ensure that clients receive data which meets the terms and conditions of the work agreement.

The review may consider the results of previous work of a similar nature or, where new testing is being implemented, the results of inter-laboratory testing, trial tests, proficiency samples, MDL studies, etc.

Discrepancies

Any differences between the request or tender and the capability of the laboratory to fulfill the proposed work are resolved before any testing begins.

Modifications are allowed upon consent of the client. Each contract shall be acceptable to both the laboratory and the client.

Problems encountered during any stage of reviewing the testing are addressed and resolved to the satisfaction of both the laboratory and the client.

Records

The laboratory maintains any records for the initial review of new work entering the laboratory, including any significant changes in the proposed work plan.

Communication logs (telephone calls, on-site visits, meetings, e-mails, etc.) are used to record all pertinent discussions concerning the client’s requirements. Logs must include the date, time, brief details of the exchange, resolution of any complaints, and identification of the parties involved.

Subcontracted work is fully described and documented in advance of receipt of the work from the client.

Once work has been accepted, the Customer Service Manager is responsible for setting up the client in the LIMS system, setting up an account with the client, and monitoring the project to ensure that all of the client’s requirements are met.

Appendix 19

Standard Procedure for Evaluation and Updating QA Manual

According to NVLAP Handbook 150-13, the TEM QA manual must be reviewed on an annual basis by the QA officer and update or revised if necessary. The manual will also must be revised and updated when there is a change in the regulation governing the analytical method which the laboratory is being accredited for; there are additions or removal of instruments from the list of equipments, any change in the key personnel or signatories or QA procedure changes. It is the responsibility of the QA officer to complete the process by documenting the changes made. The QA officer then signs and dates the revision.

Appendix 20

Organization and Client Confidentiality (NVLAP Section 4.1.5)

a) At AES, Inc. analysts and technicians are insulated from work-related undue pressures that would compromise the quality of their work. Management is aware and considerate of these internal pressures such as management burdens and project deadlines, and of external stresses such as customer complaints and priority requests for analysis

Management policy is to remain supportive of laboratory personnel and aware of their workloads and the demands placed upon them. Precautions are taken to ensure that there are no conflicts of interest between staff and clients. For example, priority requests, complaints, or status of work inquiries are directed through supervisors, managers, or administrative personnel.

b) A section that ensures the protection of client information and transmission of results has been developed. An Interim Change Notice has been completed and will be added to the laboratory QAM at the next revision. The Interim Change Notice states:
"AES, Inc., will not intentionally divulge to any person (other than a client or person designated by a client in writing) any information regarding the services provided by AES or any information disclosed to AES by the client.

Reports should be issued only with a 'Notice of Confidentiality' clause, whether it is sent via facsimile, electronic or any other means. A copy of the report, cover letter, invoice, sample receipt checklist, COC and case narrative (if printed) should be made and placed in the folder before mailing the report.

Test results are reported according to client requirements. If a client requests to have reports or information sent by fax, the client is notified in advance of the transmission, whenever possible, and all documents include a cover sheet with the following statement:

NOTICE OF CONFIDENTIALITY
The information contained in this facsimile message may be legally privileged and is confidential information intended only for the use of the individual or entity named above. If the reader of this message is not the intended recipient, you are hereby notified that any use, dissemination, distribution or copy of this facsimile message is strictly prohibited. If you have received this facsimile message in error, please contact us by telephone at (770) 457-8177 and return the facsimile message to us at the address above via the US postal service."

c) <u>Legal and Ethical Policy</u>
For a quality assurance program to succeed, it is imperative that all employees adhere to procedures which detect and prevent improper, unethical, or illegal actions which could in any way compromise the reliability and integrity of the laboratory. Training in legal and ethical responsibilities is mandatory. Records are maintained that document, through individual signatures, that every employee understands the consequences of improper, unethical, or illegal actions.

Appendix 21

Standard Procedure for TEM Data Processing

The section (h) (I) (ii) of NVLAP Handbook 150-13 discusses the need for capture procedure, processing, manipulation, recording, storage or retrieval of calibration or test data using the computers.

As a rule, data transfer should be kept to a minimum to prevent errors or loss of data. The preservation of critical data is assured by scanning and saving the reports submitted to the clients in the central computer system of the company.

Appendix 22

NVLAP Accreditation - Use of NVLAP Logo (NVLAP Annex A)

1. The term and logo shall not be used in a manner that brings NVLAP into disrepute or misrepresents a laboratory's scope of accreditation or accredited status.

2. When the term *NVLAP* is used to reference a laboratory's accredited status, it shall be accompanied by the NVLAP Lab Code.

3. When the NVLAP logo is used to reference a laboratory's accredited status, it shall be accompanied by the NVLAP Lab Code in an approved caption. The caption shall appear below and in close proximity to the logo.

4. The form of the NVLAP logo must conform to the following guidelines:

- The logo shall stand by itself and shall not be combined with any other logo, symbol, or graphic.
- The aspect ratio (height to width) shall be 1 to 2.25.
- The logo and caption shall be of a size that allows the caption to be easily read. The size of the caption shall not exceed the size of the logo itself.
- The logo shall appear in black, blue or other color approved by NVLAP, and may be filled or unfilled. In the case of a filled logo, the same color shall be used for the outline and the fill.

5. The name of at least one Approved Signatory shall appear on a test or calibration report that displays the NVLAP logo or references NVLAP accreditation.

6. When the term and logo are used on test or calibration reports, such use shall be limited to reports in which some or all of the data are from tests or calibrations performed by the laboratory under its scope of accreditation.

- A test or calibration report that contains both data covered by the accreditation and data not covered by the accreditation shall clearly identify the data that are not covered by the accreditation.
- The report must prominently display the following statement at the beginning of the report: "This report contains data that are not covered by the NVLAP accreditation."

7. When the term and logo are used on test or calibration reports that also include work done by subcontracted laboratories, such use shall be limited to reports in which some or all of the data are from tests or calibrations performed by the laboratory under its scope of accreditation.

- A test or calibration report that contains both data covered by the accreditation and data provided by a subcontractor shall clearly identify the data that were provided by the subcontracted laboratory.

- The report must prominently display the following statement at the beginning of the report: "This report contains data that were produced under subcontract by Laboratory X." If the subcontracted laboratory is accredited by NVLAP, then its Lab Code should also be stated.

- If the subcontracted laboratory is accredited by a body other than NVLAP, then the name of the accreditation body and the laboratory's number will be stated. If the subcontracted laboratory is not accredited, then this must be stated.

8. Each test and calibration report bearing the term or logo shall include a statement that the report must not be used by the client to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the federal government.

9. When used in a contract or proposal, the term and logo shall be accompanied by a description of the laboratory's scope of accreditation and current accreditation status.

Appendix 23

Standard Procedures for Using Non-Standard Methods

Whenever the use of a non-standard procedure or method is required to complete preparation or analysis of samples, approval from the client will be obtained in advance and a full summary of the method is disclosed to the client as described in NVLAP Handbook 150-13. These include detection limit, accuracy, and limitation of the method. In general these non-standard methods are employed for analysis of asbestos bulk samples, water or waste water, qualitative bulk sample analysis, and particle identification.

Appendix 24

Subcontracting of tests and calibrations (NVLAP Section 4.5)

4.5 An Interim Change Notice has been completed and will be added to the laboratory QAM at the next revision. The Interim Change Notice includes the following updates:

4.5.1 All subcontract laboratories are required to supply the Quality Assurance Manager, upon written request, with adequate proof of accreditation in applicable state, NVLAP, or other programs, depending upon the client and origination of the samples. Documents shall be requested from all subcontract laboratories. The requested documents will include, but may not be limited to, a current Quality Assurance Manual, the scope of approved testing, proof of insurance, and NVLAP and/or applicable state accrediting authority certificates.

4.5.2 The client must be contacted in writing of the intent to subtract any portion of the testing to another party.

4.5.3 When samples are received which have testing requirements that cannot be performed in-house, the samples must be sub-contracted to another laboratory. The subcontract laboratory's scope will be checked to make sure it possess the proper accreditation for the requested test (See 2.19 - Subcontracting Analyses). The results from the subcontracted laboratory must be reported utilizing a copy of the original report received from the subcontract laboratory. By approving the subcontractor, the client has made the selection therefore assumes responsibility.

4.5.4 The following is a list of laboratories which may be utilized for the subcontracting of AES samples: Material Analytical Services (MAS) 770-866-3209

Testing and calibration results obtained from subcontractors (NVLAP Section 5.10.6)

The results from the subcontracted laboratory must be reported utilizing a copy of the original report received from the subcontract laboratory.

Appendix 25

Standard Procedures for Purchase, Reception, and Storing of Consumable materials

All the supplies have to be examined prior to being used in the laboratory to ensure their quality. Any item that does not meet the required specifications as described in NVLAP handbook 150-13 is rejected.

Several important reagents and grades are given in the following:

Acetone, Chloroform	Reagent Grade
Distilled water	Asbestos free
Oxygen	99.5 % purity
MCE and polycarbonate filters	asbestos free
TEM Grids	copper
Carbon rods	spectrochemically pure

All the consumables and supplies must be stored in such a way to prevent cross contamination.

Appendix 26

Standard Procedures for Determination of Analyst Accuracy in Classifying Bundles and Fibrils ≥ 1 micron in Length

The requirements for assuring that each analyst achieves at least 90% accuracy in distinguishing bundles from single fibrils is described in the NVLAP Handbook 150-13.

Several grids with different loading of chrysotile fibers and bundles are prepared from bulk SRM 1866. The grids with a moderate loading are to be selected and used for analysis by a different analyst to determine accuracy.

Appendix 27

Standard Procedures for Deterring the Accuracy and Precision of Measuring Fibers approximately 0.5 micron long

The NVLAP handbook 150-13 requires that the length measuring system at 0.5 micron level be calibrated on a regular basis. In addition, repeat analysis by the same analyst and a different analyst is to be used to determine the measurement accuracy of the original analyst.

The measure marks on the fluorescent screen must be calibrated on monthly basis with special attention to the 1 micron scale. A dispersion of fibers approximately 0.5 micron long is prepared from SRM 1866. This may require preparation of several grids with different loading and select the ones that exhibit the highest amount of 0.5 micron fibers.

Appendix 28

Standard Procedure for Creating and use of Control Charts

The control charts are the basic tools for quality assurance. They provide graphical means to demonstrate statistical control, monitor measurement process, diagnose measurement problems, etc. At AES the control chars for various calibration values of TEM are prepared using EXCEL spreadsheet software. The control charts must utilize a central baseline (mean) and two warning limits around it (2-signma) within which most 95% values should lie.

It is responsibility of TEM quality control officer to monitor the system calibration, update the control charts, ensure that the overall system is in statistical control, and resolve any control problem as soon as possible.

Appendix 29

Standard Operating Procedure for Detection of Na-Peak in Standard Crocidolite (Quantitative Method)

Introduction:

The quantitative information available from a x-ray spectrum is the energy at which the x-ray emission lines appear in the spectrum. The quantitative information is the intensity of the emitted radiation. This intensity is normally expressed as the number of counts per second, I (cps), from the detector or total number of counts, N, obtained in a fixed period of time. Because x-ray emission is a random event process, the precision of intensity measurements can be predicted. These events follow Poisson statistics that enable calculation of the standard deviation of the number of counts:

$$\mathbf{\Sigma = (N_t)^{1/2}}$$

Where σ is the standard deviation in the counts, and N_t is the number of counts collected in the time, t.

As the elemental concentration, C, approaches the order of 0.5 wt % in x-ray microanalysis, the value of N_t is no longer much larger than N_b (background). This concentration range, below 0.5 wt %, is often referred to as the trace element analysis range of lower limit of detection (LLC). For the light elements or x-ray lines with energy less than or equal to 1.0 kev such as sodium (1.040 kev) the trace element range (LLC) begins at about 1.0 wt %.

The analysis requirement in trace element analysis is to detect significant difference between the sample and the background generated from the sample. Therefore accurate background measurement becomes important particularly at low concentartions. The critical issues are low peak-to-background ratios and difficulty of finding suitable background areas adjacent to the peak being measured.

The practical approach suggested by Leibhafsky et al and others is that an element can be considered to be present if the value of N_{net} exceeds the twice the background standard deviation for a 95% probability and three times of background standard deviation for a 99% probability.

The following SOP must be followed to show that Na K-lines are measured above the Cu L-lines background as described in section 5.7 b of NIST booklet 150-13.

Procedure:

1. Follow steps 1 through 6 described in appendix 34A to acquire a spectrum of crocidolite.
2. Move the cursor to 1.040 kev (Na k-line) and record the number of counts as N.
3. Move the cursor to the left of copper L-line about 0.8 kev and record the counts as N_b.
4. Acquire new spectrum and repeat steps 1 through 3 until enough data is collected (normally 5 data points).
5. Calculate the mean of Na counts and record it as N.
6. Calculate the mean of background and record it as N_b.
7. Calculate the standard deviation of background mean $(N_b)^{1/2}$
8. Calculate net Na count as $N_{net}= N-N_b$.
9. Calculate values for two and three times of background standard deviation.
10. Determine if the presence of Na peak is statistically significant as described above.

References:

J.I. Goldstein et al., Scanning Electron Microscopy and X-ray Microanalysis, Plenum Press, New York, 1981, pp. 393-397 and pp. 430-435.

Appendix 30

Standard Procedure for Calibration of Magnification of Optical Microscope used to Measure Grid Opening Area

The PCM microscope which is used to measure the average grid opening area of every new TEM grid lot will be calibrated using an external calibration reticule.

The PCM microscope used for this purpose has a reticule mounted in one of its eyepieces. This reticule has a diameter of 100 microns divided into 20 divisions each 5 microns wide.

In practice, the variation in the calibration measurements (2s) must be less than 5% of the mean of each calibration value.

Appendix 31

Standard procedure for Internal TEM Audit and Review

The TEM laboratory manager shall conduct the internal audit annually.

Internal performance audits are a means for the quality assurance of officer to determine the applicability, effectiveness, and utilization of procedure. Only designated personnel perform the audits.

An audit is performed using NVLAP specific operations checklist (HB 150-13) as well as the NIST HB 150 Checklist. The results of audit are discussed with the QA/QC officer and if necessary corrective actions are recommended.

The laboratory manager, following the QA/QC reviews all analytical data. The laboratory manager is responsible for checking the data integrity, correct usage of test methods, and the instruments.

Appendix 32

Management Reviews (NVLAP Section 4.14)

Annual Reports

The annual report to management includes the following information.

SOP. The report indicates any changes to existing SOPs or any new SOPs.

Corrective Action Reports. The report contains information about any corrective action reports that may have been written during the time period since the last QA report.

Audits. The QA report includes the results of any audits performed during the time period since the last report.

PE samples. The report includes the results of PE samples analyzed since the last report. The PE report indicates the status of performance as it relates to NVLAP.

Certifications. Any changes or additions to the laboratory's certifications are addressed in the reports.

The annual report is reviewed and signed by the President of Operations, Laboratory Manager, and the Bulk & Air Asbestos Manager. A copy of this report is kept for 5 years.

Appendix 33

Quality System (NVLAP Section 4.2.2)

4.2.2 Quality Policy

The objective of Analytical Environmental Services, Inc. is to generate high quality data in a cost-effective manner, which is accurate, impartial, reliable, and adequate for its intended use. The management of AES is committed to following accepted laboratory practices to achieve high quality of testing services, and strives to ensure both the analytical validity and legal defensibility of all reported data.

e) AES management is committed to establish, implement, and maintain a quality system appropriate to the scope of all laboratory activities, including the type, range, and volume of testing. Management shall document the policies, systems, programs, procedures, and instructions to the extent necessary (*compliance with NVLAP Handbook 150*) to enable AES to assure the quality of the test results generated.

Quality system documentation is communicated to, understood by, and made available to personnel through AES management by means of training and educational instruction. All laboratory staff concerned with analytical testing activities must familiarize themselves with the quality documentation and implement the policies and principles in their work.

It is the policy of AES to continually improve quality systems and provide support to improvement efforts.

Appendix 34

Document Control (NVLAP Section 4.3)

Document Control

Regardless of which analytical procedures are used in the laboratory, the methodology shall consist of carefully documented Standard Operating Procedures (SOPs) and approved methods which may be periodically modified, updated or replaced entirely due to advances in technology or changes in regulatory protocols. Some clients may require pre-approval of method revisions before modifications are used to generate data. Documentation of analytical procedures for generating laboratory data shall be clear, concise, adequately referenced, and reflect the actual steps employed by the analyst.

Procedures

Methodologies employed in the laboratory are documented by the creation of an SOP. This document provides the analyst with the information necessary to perform the analysis. Every SOP is created in accordance with this QA document. It follows the intent of the method it is patterned after, but provides any additional information essential to the specific instrument instructions, specific quality concerns, etc.

If an SOP is not available for a specific analysis, the analyst will follow EPA, Standard Methods, NIOSH, recognized industry standards, or other regulatory methodology as required. No deviations of any kind are allowed.

All analytical procedures must provide documentation so that the complete process used to produce data can be reconstructed.

All deviations from an approved analytical procedure are authorized and documented by the Technical Director.

All changes to an approved procedure require, at a minimum, an Interim Change Notice. A complete revision and re-issuance of the SOP may be required. SOPs are reviewed at least annually.

A list of all current SOPs including their review and revision status is maintained electronically on AES_server\L\Current SOP\SOP Masterlist. Current SOPs are maintained electronically in the AES_server\L\Current SOP Laboratory SOP\Current Revisions\. These documents are also available from the AES Portal Server in the Technical Management folder. All controlled documents are in “Read Only” format and password protected. The Vice-President of Operations, QA Manager, Technical Director and their appointees are the only laboratory employees with edit access to these folders.

Appendix 35

Service to the client (NVLAP Section 4.7)

The laboratory affords its clients and representatives cooperation by clarifying their request and monitoring the laboratory's performance relative to the work performed.

The Bulk & Air Asbestos Manager accomplishes this the following way:

- Ensures effective and accurate communication between the client and the laboratory.
- Handles all client requests and needs.
- Utilizes any corporate documents to consult with clients about client questions or concerns.
- Responsible for notifying the Customer Service Manager of any client activities that entail services that are not currently performed by AES.
- Assesses client requests in light of current workload with consultation with the Customer Service Manager.
- Develops and maintains client records and requirements.
- Ensures that the laboratory is aware of, and completes, all client requests and requirements.
- Responsible for meeting with the Marketing Manager, Customer Service Manager, and President on a periodic basis for marketing purposes.
- Communicates proper sampling, shipping, and receiving procedures to clients.
- Documents all client interaction and maintains all client information in the Project Management System.
- Reviews and approves data reports prior to their release to the clients.
- Ensures client specific reporting and quality control requirements are met.

Appendix 36

Control of nonconforming testing and/or calibration work
(NVLAP Section 4.9-4.11)

Deficiencies or non-conformances in analytical procedures, materials, components or methodology may lead to the release of incorrect analytical results to the customer. Once a deficiency or non-conformance has been identified, corrective actions must be implemented to insure proper data qualification and narration on the final client report and, when possible, prevent the deficiency being repeated. To document and track the non-conformance, a Corrective Action Report (CAR) is issued through the LIMS system. An example of a Corrective Action Report is at the end of this section.

Standard Procedure for Defining, Implementing, and Closing a Corrective Action Report (CAR).

> Non-conformance: A non-conformance is defined as any situation that is either outside acceptable limits (data) or does not comply with the procedure/method in some way. The following situations are considered non-conformances and the completion of a CAR report is required. (E.g. Contamination in the Blank)
>
> Procedure for the issuing, completing, and closing of an analytical or technical systems related CAR.
>
>> When a non-conformance occurs, the employee performing the work or the initial data reviewer must complete a CAR in the LIMS system. as indicated below.
>>
>> When completing a CAR, include all accompanying data, information, etc in a "Data Package" along with the NCR and submit this to the Technical Director or Quality Assurance Manager for review.
>
> General Procedures and Responsibilities for Corrective Action Reports Involving Deficiencies:
>
>> When the PLM Laboratory Quality Assurance Manager or Bulk Air & Asbestos Manager issues a corrective action report (CAR) for a non-conformance classified as a deficiency, the Laboratory Manager, Assistant Laboratory Manager, or Technical Director will be informed immediately.
>>
>> The QA Manager will track the completion of the corrective actions required to correct the deficiency. The assigned personnel are responsible for completing the corrective action within the specified time frame.
>>
>> The chain of custody and the Sample Receipt Forms are used to document non-conformance during log-in.

Method Suspension or Restriction

In some cases, it may be necessary to suspend or restrict the use of a method that constitutes significant risk and or liability to AES. Suspension or restriction procedures can be initiated by the Quality Assurance Manager, Technical Director, Laboratory Manager, or VP of Operations.

Prior to suspension or restriction, confidentiality is respected, the problem and the required corrective action is stated in writing on the associated CAR and presented to the Laboratory Manager.

The Laboratory Manager, Technical Director, Quality Assurance Manager, and the affected supervisor are notified.

The Laboratory Manager arranges for the appropriate operations people to speak with the Quality Assurance Manager or Technical Director the day of notification. This meeting is held to confirm that there is a problem, and that suspension or restriction of the method is required.

The suspension or restriction meeting will conclude with a discussion of the steps necessary to bring the method or test fully back on line if the method is suspended or restricted. The Quality Assurance Manager will also specify any documentation necessary to verify that corrective action has occurred.

After suspension or restriction, the laboratory will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. It is the responsibility of the Laboratory Manager to hold all reports. Clients will not generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Upon completion of the required corrective actions per the CAR, laboratory management will determine if the affected systems are back in control and reports can be released. If systems are still deemed out of control, further corrective actions are required. A team, with all principals involved can devise a start up plan to cover all steps from client notification through compliance of method and release of reports.

If the PLM Laboratory Quality Assurance Manager or Bulk Air & Asbestos Manager recommends client notification regarding affects on past or current data

quality, all associated information is forwarded to the Laboratory Manager and VP of Operations. These will review the data and determine appropriate actions.

Client notifications are the responsibility of the Laboratory Manager and VP of Operations

Appendix 37

Estimation of Uncertainty (NVLAP Section 5.4.6)

A reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

It is defined by the monthly checks, proficiency exam results and error rates.

Appendix 38

March Plasmod Asher Operating Procedure

Procedures for using the March Plasmod Asher to prepare TEM samples for analysis. This step is a prerequisite for carbon coating and should follow mounting the samples on glass slides and collapsing the filters with acetone.

1. There are 3 main parts to the March Plasmod Asher:
   1.1 Vacuum Pump
   1.2 RF Generator
   1.3 Oxygen Flow Controller w/ Oxygen Tank

2. To use the March Plasmod Asher:
   2.1.1 Press the AC button on both the RF Generator and Oxygen Flow Controller and allow both units to warm up
      2.1.1.1 Make sure that the 'Level' indicator is on 'B'
   2.1.2 Turn on the vacuum pump by flipping the switch on the vacuum pump that is connected to the RF Generator
   2.1.3 Open the door on the RF Generator and very carefully pull out the quartz reaction chamber from the instrument. Keep the reaction chamber positioned so that the holes for Oxygen to flow through remain on the bottom.
   2.1.4 Place the slides to be ashed face-up and across the tube, perpendicular to the direction the reaction chamber was removed from the instrument. Carefully return the quartz reaction chamber into the metal outer reaction chamber.
   2.1.5 Align the rubber gasket and apply light upward pressure to the flat outer surface of the glass reaction chamber and **press the vacuum button** on the RF Generator.
   2.1.6 Close metal door.
   2.1.7 Watch the vacuum gauge, located on the Oxygen Flow Controller, to ensure that the seal is good. The vacuum gauge needle should slowly move toward the 100µm mark but must be at least between the 10^3 µm mark and the 100µm mark.
      2.1.7.1 During this time the vacuum is removing air, moisture and nitrogen from inside of the reaction chamber.

2.1.8 Open the main valve on top of the oxygen tank and the regulator valve sticking out from the side just before the oxygen line begins. The pressure gauge on the oxygen tank should read between 5-6 psi.

2.1.9 Press the “Gas 1” button on the Oxygen Flow Controller. The flow for “Gas 1” should read 4 L/min. At this time the vacuum gauge should read between 1.3-1.4 Torr. Wait at least 3-5 minutes to allow the oxygen time to replace the nitrogen in the reaction chamber.

2.1.10 Once the vacuum and oxygen levels are correct, set the timer to the duration determined from calibration and start the timer. **Then press the RF button** on the RF Generator.

2.1.10.1 The plasma should become more blueish than pinkish as ashing progresses: Blue=Oxygen plasma and Pink=Nitrogen plasma

2.1.10.2 Current ideal level should be set at B on the RF Generator

2.1.11 Once the 2 ½ minutes on the timer has been started, press the Meter button on the RF Generator and adjust the tuning knob so that the Power Efficiency is showing in the midrange.

2.1.12 Once the timer stops, **press the buttons to turn off the RF, Meter, Vacuum and Gas 1**. Then flip the switch to turn off the vacuum pump. At this time you can press the AC buttons on both the RF Generator and Oxygen Flow Controller to shut down the entire system.

Section Eight:
Attachments

ATTACHMENT 1

40CFR763

[Code of Federal Regulations]
[Title 40, Volume 28]
[Revised as of July 1, 2003]
From the U.S. Government Printing Office via GPO Access
[CITE: **40CFR763**]

[Page 708-803]

TITLE 40--PROTECTION OF ENVIRONMENT

CHAPTER I--ENVIRONMENTAL PROTECTION AGENCY (CONTINUED)

PART 763--**ASBESTOS**

Subparts A-C [Reserved]

Subpart D [Reserved]

Subpart E--**Asbestos**-Containing Materials in Schools

Subpart F [Reserved]

Subpart G--**Asbestos** Worker Protection

Subpart H [Reserved]

Subpart I--Prohibition of the Manufacture, Importation, Processing, and Distribution in Commerce of Certain **Asbestos**-Containing Products;

Labeling Requirements

Authority: 15 U.S.C. 2605, 2607(c), 2643, and 2646.

Subparts A-D [Reserved]

Subpart E--**Asbestos**-Containing Materials in Schools

Source: 52 FR 41846, Oct. 30, 1987, unless otherwise noted.

Sec. 763.80 Scope and purpose.

(a) This rule requires local education agencies to identify friable and nonfriable **asbestos**-containing material (ACM) in public and private elementary and secondary schools by visually inspecting school buildings for such materials, sampling such materials if they are not assumed to be ACM, and having samples analyzed by appropriate techniques referred to in this rule. The rule requires local education agencies to submit management plans to the Governor of their State by October 12, 1988, begin to implement the plans by July 9, 1989, and complete implementation of the plans in a timely fashion. In addition, local education agencies are required to use persons who have been accredited to conduct inspections, reinspections, develop management plans, or perform response actions. The rule also includes recordkeeping requirements. Local education agencies may contractually delegate their duties under this rule, but they remain responsible for the proper performance of those duties.

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Local education agencies are encouraged to consult with EPA Regional **Asbestos** Coordinators, or if applicable, a State's lead agency designated by the State Governor, for assistance in complying with this rule.

(b) Local education agencies must provide for the transportation and disposal of **asbestos** in accordance with EPA's ``**Asbestos** Waste Management Guidance.'' For convenience, applicable sections of this guidance are reprinted as Appendix D of this subpart. There are regulations in place, however, that affect transportation and disposal of **asbestos** waste generated by this rule. The transportation of **asbestos** waste is covered by the Department of Transportation (49 CFR part 173, subpart J) and disposal is covered by the National Emissions Standards for Hazardous Air Pollutants (NESHAP) (40 CFR part 61, subpart M).

Sec. 763.83 Definitions.

For purposes of this subpart:

Act means the Toxic Substances Control Act (TSCA), 15 U.S.C. 2601, et seq.

Accessible when referring to ACM means that the material is subject to disturbance by school building occupants or custodial or maintenance personnel in the course of their normal activities.

Accredited or accreditation when referring to a person or laboratory means that such person or laboratory is accredited in accordance with section 206 of Title II of the Act.

Air erosion means the passage of air over friable ACBM which may result in the release of **asbestos** fibers.

Asbestos means the asbestiform varieties of: Chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonitegrunerite); anthophyllite; tremolite; and actinolite.

Asbestos-containing material (ACM) when referring to school buildings means any material or product which contains more than 1 percent **asbestos**.

Asbestos-containing building material (ACBM) means surfacing ACM, thermal system insulation ACM, or miscellaneous ACM that is found in or on interior structural members or other parts of a school building.

Asbestos debris means pieces of ACBM that can be identified by color, texture, or composition, or means dust, if the dust is determined by an accredited inspector to be ACM.

Damaged friable miscellaneous ACM means friable miscellaneous ACM which has deteriorated or sustained physical injury such that the internal structure (cohesion) of the material is inadequate or, if applicable, which has delaminated such that its bond to the substrate (adhesion) is inadequate or which for any other reason lacks fiber cohesion or adhesion qualities. Such damage or deterioration may be illustrated by the separation of ACM into layers; separation of ACM from the substrate; flaking, blistering, or crumbling of the ACM surface; water damage; significant or repeated water stains, scrapes, gouges, mars or other signs of physical injury on the ACM. **Asbestos** debris originating from the ACBM in question may also indicate damage.

Damaged friable surfacing ACM means friable surfacing ACM which has deteriorated or sustained physical injury such that the internal structure (cohesion) of the material is inadequate or which has delaminated such that its bond to the substrate (adhesion) is inadequate, or which, for any other reason, lacks fiber cohesion or adhesion qualities. Such damage or deterioration may be illustrated by the separation of ACM into layers; separation of ACM from the substrate; flaking, blistering, or crumbling of the ACM surface; water damage; significant or repeated water stains, scrapes, gouges, mars or other signs of physical injury on the ACM. **Asbestos** debris originating from the ACBM in question may also indicate damage.

Damaged or significantly damaged thermal system insulation ACM means thermal system insulation ACM on pipes, boilers, tanks, ducts, and other thermal system insulation equipment where the insulation has lost its structural integrity, or its covering, in whole or in part, is crushed, water-stained, gouged, punctured, missing, or not intact such that it is not able to contain fibers. Damage may be further illustrated by occasional punctures, gouges or other signs of physical injury to ACM; occasional water damage on

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the protective coverings/jackets; or exposed ACM ends or joints. **Asbestos** debris originating from the ACBM in question may also indicate damage.

Encapsulation means the treatment of ACBM with a material that surrounds or embeds **asbestos** fibers in an adhesive matrix to prevent the release of fibers, as the encapsulant creates a membrane over the surface (bridging encapsulant) or penetrates the material and binds its components together (penetrating encapsulant).

Enclosure means an airtight, impermeable, permanent barrier around ACBM to prevent the release of **asbestos** fibers into the air.

Fiber release episode means any uncontrolled or unintentional disturbance of ACBM resulting in visible emission.

Friable when referring to material in a school building means that the material, when dry, may be crumbled, pulverized, or reduced to powder by hand pressure, and includes previously nonfriable material after such previously nonfriable material becomes damaged to the extent that when dry it may be crumbled, pulverized, or reduced to powder by hand pressure.

Functional space means a room, group of rooms, or homogeneous area (including crawl spaces or the space between a dropped ceiling and the floor or roof deck above), such as classroom(s), a cafeteria, gymnasium, hallway(s), designated by a person accredited to prepare management plans, design abatement projects, or conduct response actions.

High-efficiency particulate air (HEPA) refers to a filtering system capable of trapping and retaining at least 99.97 percent of all monodispersed particles 0.3 [mu]m in diameter or larger.

Homogeneous area means an area of surfacing material, thermal system insulation material, or miscellaneous material that is uniform in color and texture.

Local education agency means:

(1) Any local educational agency as defined in section 198 of the Elementary and Secondary Education Act of 1965 (20 U.S.C. 3381).

(2) The owner of any nonpublic, nonprofit elementary, or secondary school building.

(3) The governing authority of any school operated under the defense dependent's education system provided for under the Defense Dependents' Education Act of 1978 (20 U.S.C. 921, et seq.).

Miscellaneous ACM means miscellaneous material that is ACM in a school building.

Miscellaneous material means interior building material on structural components, structural members or fixtures, such as floor and ceiling tiles, and does not include surfacing material or thermal system insulation.

Nonfriable means material in a school building which when dry may not be crumbled, pulverized, or reduced to powder by hand pressure.

Operations and maintenance program means a program of work practices to maintain friable ACBM in good condition, ensure clean up of **asbestos** fibers previously released, and prevent further release by minimizing and controlling friable ACBM disturbance or damage.

Potential damage means circumstances in which:

(1) Friable ACBM is in an area regularly used by building occupants, including maintenance personnel, in the course of their normal activities.

(2) There are indications that there is a reasonable likelihood that the material or its covering will become damaged, deteriorated, or delaminated due to factors such as changes in building use, changes in operations and maintenance practices, changes in occupancy, or recurrent damage.

Potential significant damage means circumstances in which:

(1) Friable ACBM is in an area regularly used by building occupants, including maintenance personnel, in the course of their normal activities.

(2) There are indications that there is a reasonable likelihood that the material or its covering will become significantly damaged, deteriorated, or delaminated due to factors such as changes in building use, changes in operations and maintenance practices, changes in

occupancy, or recurrent damage.

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(3) The material is subject to major or continuing disturbance, due to factors including, but not limited to, accessibility or, under certain circumstances, vibration or air erosion.

Preventive measures means actions taken to reduce disturbance of ACBM or otherwise eliminate the reasonable likelihood of the material's becoming damaged or significantly damaged.

Removal means the taking out or the stripping of substantially all ACBM from a damaged area, a functional space, or a homogeneous area in a school building.

Repair means returning damaged ACBM to an undamaged condition or to an intact state so as to prevent fiber release.

Response action means a method, including removal, encapsulation, enclosure, repair, operations and maintenance, that protects human health and the environment from friable ACBM.

Routine maintenance area means an area, such as a boiler room or mechanical room, that is not normally frequented by students and in which maintenance employees or contract workers regularly conduct maintenance activities.

School means any elementary or secondary school as defined in section 198 of the Elementary and Secondary Education Act of 1965 (20 U.S.C. 2854).

School building means:

(1) Any structure suitable for use as a classroom, including a school facility such as a laboratory, library, school eating facility, or facility used for the preparation of food.

(2) Any gymnasium or other facility which is specially designed for athletic or recreational activities for an academic course in physical education.

(3) Any other facility used for the instruction or housing of students or for the administration of educational or research programs.

(4) Any maintenance, storage, or utility facility, including any hallway, essential to the operation of any facility described in this definition of ``school building'' under paragraphs (1), (2), or (3).

(5) Any portico or covered exterior hallway or walkway.

(6) Any exterior portion of a mechanical system used to condition interior space.

Significantly damaged friable miscellaneous ACM means damaged friable miscellaneous ACM where the damage is extensive and severe.

Significantly damaged friable surfacing ACM means damaged friable surfacing ACM in a functional space where the damage is extensive and severe.

State means a State, the District of Columbia, the Commonwealth of Puerto Rico, Guam, American Samoa, the Northern Marianas, the Trust Territory of the Pacific Islands, and the Virgin Islands.

Surfacing ACM means surfacing material that is ACM.

Surfacing material means material in a school building that is sprayed-on, troweled-on, or otherwise applied to surfaces, such as acoustical plaster on ceilings and fireproofing materials on structural members, or other materials on surfaces for acoustical, fireproofing, or other purposes.

Thermal system insulation means material in a school building applied to pipes, fittings, boilers, breeching, tanks, ducts, or other interior structural components to prevent heat loss or gain, or water condensation, or for other purposes.

Thermal system insulation ACM means thermal system insulation that is ACM.

Vibration means the periodic motion of friable ACBM which may result in the release of **asbestos** fibers.

Sec. 763.84 General local education agency responsibilities.

Each local education agency shall:

(a) Ensure that the activities of any persons who perform inspections, reinspections, and periodic surveillance, develop and update management plans, and develop and implement response actions, including operations and maintenance, are carried out in accordance with subpart E of this part.

(b) Ensure that all custodial and maintenance employees are properly trained as required by this subpart E and other applicable Federal and/or

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State regulations (e.g., the Occupational Safety and Health Administration **asbestos** standard for construction, the EPA worker protection rule, or applicable State regulations).

(c) Ensure that workers and building occupants, or their legal guardians, are informed at least once each school year about inspections, response actions, and post-response action activities, including periodic reinspection and surveillance activities that are planned or in progress.

(d) Ensure that short-term workers (e.g., telephone repair workers, utility workers, or exterminators) who may come in contact with **asbestos** in a school are provided information regarding the locations of ACBM and suspected ACBM assumed to be ACM.

(e) Ensure that warning labels are posted in accordance with Sec. 763.95.

(f) Ensure that management plans are available for inspection and notification of such availability has been provided as specified in the management plan under Sec. 763.93(g).

(g)(1) Designate a person to ensure that requirements under this section are properly implemented.

(2) Ensure that the designated person receives adequate training to perform duties assigned under this section. Such training shall provide, as necessary, basic knowledge of:

(i) Health effects of **asbestos**.

(ii) Detection, identification, and assessment of ACM.

(iii) Options for controlling ACBM.

(iv) **Asbestos** management programs.

(v) Relevant Federal and State regulations concerning **asbestos**, including those in this subpart E and those of the Occupational Safety and Health Administration, U.S. Department of Labor, the U.S. Department of Transportation and the U.S. Environmental Protection Agency.

(h) Consider whether any conflict of interest may arise from the interrelationship among accredited personnel and whether that should influence the selection of accredited personnel to perform activities under this subpart.

Sec. 763.85 Inspection and reinspections.

(a) Inspection. (1) Except as provided in paragraph (a)(2) of this section, before October 12, 1988, local education agencies shall inspect each school building that they lease, own, or otherwise use as a school building to identify all locations of friable and nonfriable ACBM.

(2) Any building leased or acquired on or after October 12, 1988, that is to be used as a school building shall be inspected as described

under paragraphs (a) (3) and (4) of this section prior to use as a school building. In the event that emergency use of an uninspected building as a school building is necessitated, such buildings shall be inspected within 30 days after commencement of such use.

(3) Each inspection shall be made by an accredited inspector.

(4) For each area of a school building, except as excluded under Sec. 763.99, each person performing an inspection shall:

(i) Visually inspect the area to identify the locations of all suspected ACBM.

(ii) Touch all suspected ACBM to determine whether they are friable.

(iii) Identify all homogeneous areas of friable suspected ACBM and all homogeneous areas of nonfriable suspected ACBM.

(iv) Assume that some or all of the homogeneous areas are ACM, and, for each homogeneous area that is not assumed to be ACM, collect and submit for analysis bulk samples under Secs. 763.86 and 763.87.

(v) Assess, under Sec. 763.88, friable material in areas where samples are collected, friable material in areas that are assumed to be ACBM, and friable ACBM identified during a previous inspection.

(vi) Record the following and submit to the person designated under Sec. 763.84 a copy of such record for inclusion in the management plan within 30 days of the inspection:

(A) An inspection report with the date of the inspection signed by each accredited person making the inspection, State of accreditation, and if applicable, his or her accreditation number.

(B) An inventory of the locations of the homogeneous areas where samples are collected, exact location where each bulk sample is collected, dates

that samples are collected, homogeneous areas where friable suspected ACBM is assumed to be ACM, and homogeneous areas where nonfriable suspected ACBM is assumed to be ACM.

(C) A description of the manner used to determine sampling locations, the name and signature of each accredited inspector who collected the samples, State of accreditation, and, if applicable, his or her accreditation number.

(D) A list of whether the homogeneous areas identified under paragraph (a)(4)(vi)(B) of this section, are surfacing material, thermal system insulation, or miscellaneous material.

(E) Assessments made of friable material, the name and signature of each accredited inspector making the assessment, State of accreditation, and if applicable, his or her accreditation number.

(b) Reinspection. (1) At least once every 3 years after a management plan is in effect, each local education agency shall conduct a reinspection of all friable and nonfriable known or assumed ACBM in each school building that they lease, own, or otherwise use as a school building.

(2) Each inspection shall be made by an accredited inspector.

(3) For each area of a school building, each person performing a reinspection shall:

(i) Visually reinspect, and reassess, under Sec. 763.88, the condition of all friable known or assumed ACBM.

(ii) Visually inspect material that was previously considered nonfriable ACBM and touch the material to determine whether it has become friable since the last inspection or reinspection.

(iii) Identify any homogeneous areas with material that has become friable since the last inspection or reinspection.

(iv) For each homogeneous area of newly friable material that is already assumed to be ACBM, bulk samples may be collected and submitted for analysis in accordance with Secs. 763.86 and 763.87.

(v) Assess, under Sec. 763.88, the condition of the newly friable material in areas where samples are collected, and newly friable materials in areas that are assumed to be ACBM.

(vi) Reassess, under Sec. 763.88, the condition of friable known or assumed ACBM previously identified.

(vii) Record the following and submit to the person designated under Sec. 763.84 a copy of such record for inclusion in the management plan within 30 days of the reinspection:

(A) The date of the reinspection, the name and signature of the person making the reinspection, State of accreditation, and if applicable, his or her accreditation number, and any changes in the condition of known or assumed ACBM.

(B) The exact locations where samples are collected during the reinspection, a description of the manner used to determine sampling locations, the name and signature of each accredited inspector who collected the samples, State of accreditation, and, if applicable, his or her accreditation number.

(C) Any assessments or reassessments made of friable material, the name and signature of the accredited inspector making the assessments, State of accreditation, and if applicable, his or her accreditation number.

(c) General. Thermal system insulation that has retained its structural integrity and that has an undamaged protective jacket or wrap that prevents fiber release shall be treated as nonfriable and therefore is subject only to periodic surveillance and preventive measures as necessary.

Sec. 763.86 Sampling.

(a) Surfacing material. An accredited inspector shall collect, in a statistically random manner that is representative of the homogeneous area, bulk samples from each homogeneous area of friable surfacing material that is not assumed to be ACM, and shall collect the samples as follows:

(1) At least three bulk samples shall be collected from each homogeneous area that is 1,000 ft\2\ or less, except as provided in Sec. 763.87(c)(2).

(2) At least five bulk samples shall be collected from each homogeneous area that is greater than 1,000 ft\2\ but less than or equal to 5,000 ft\2\, except as provided in Sec. 763.87(c)(2).

(3) At least seven bulk samples shall be collected from each homogeneous

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area that is greater than 5,000 ft\2\, except as provided in Sec. 763.87(c)(2).

(b) Thermal system insulation. (1) Except as provided in paragraphs (b) (2) through (4) of this section and Sec. 763.87(c), an accredited inspector shall collect, in a randomly distributed manner, at least three bulk samples from each homogeneous area of thermal system insulation that is not assumed to be ACM.

(2) Collect at least one bulk sample from each homogeneous area of patched thermal system insulation that is not assumed to be ACM if the patched section is less than 6 linear or square feet.

(3) In a manner sufficient to determine whether the material is ACM or not ACM, collect bulk samples from each insulated mechanical system that is not assumed to be ACM where cement or plaster is used on fittings such as tees, elbows, or valves, except as provided under

Sec. 763.87(c)(2).

(4) Bulk samples are not required to be collected from any homogeneous area where the accredited inspector has determined that the thermal system insulation is fiberglass, foam glass, rubber, or other non-ACBM.

(c) Miscellaneous material. In a manner sufficient to determine whether material is ACM or not ACM, an accredited inspector shall collect bulk samples from each homogeneous area of friable miscellaneous material that is not assumed to be ACM.

(d) Nonfriable suspected ACBM. If any homogeneous area of nonfriable suspected ACBM is not assumed to be ACM, then an accredited inspector shall collect, in a manner sufficient to determine whether the material is ACM or not ACM, bulk samples from the homogeneous area of nonfriable suspected ACBM that is not assumed to be ACM.

Sec. 763.87 Analysis.

(a) Local education agencies shall have bulk samples, collected under Sec. 763.86 and submitted for analysis, analyzed for **asbestos** using laboratories accredited by the National Bureau of Standards (NBS). Local education agencies shall use laboratories which have received interim accreditation for polarized light microscopy (PLM) analysis under the EPA Interim **Asbestos** Bulk Sample Analysis Quality Assurance Program until the NBS PLM laboratory accreditation program for PLM is operational.

(b) Bulk samples shall not be composited for analysis and shall be analyzed for **asbestos** content by PLM, using the ``Interim Method for the Determination of **Asbestos** in Bulk Insulation Samples'' found at appendix E to subpart E of this part.

(c)(1) A homogeneous area is considered not to contain ACM only if the results of all samples required to be collected from the area show **asbestos** in amounts of 1 percent or less.

(2) A homogeneous area shall be determined to contain ACM based on a finding that the results of at least one sample collected from that area shows that **asbestos** is present in an amount greater than 1 percent.

(d) The name and address of each laboratory performing an analysis, the date of analysis, and the name and signature of the person performing the analysis shall be submitted to the person designated under Sec. 763.84 for inclusion into the management plan within 30 days of the analysis.

[52 FR 41846, Oct. 30, 1987, as amended at 60 FR 31922, June 19, 1995]

Sec. 763.88 Assessment.

(a)(1) For each inspection and reinspection conducted under Sec. 763.85 (a) and (c) and previous inspections specified under Sec. 763.99, the local education agency shall have an accredited inspector provide a written assessment of all friable known or assumed ACBM in the school building.

(2) Each accredited inspector providing a written assessment shall sign and date the assessment, provide his or her State of accreditation, and if applicable, accreditation number, and submit a copy of the assessment to the person designated under Sec. 763.84 for inclusion in the management plan within 30 days of the assessment.

(b) The inspector shall classify and give reasons in the written assessment for classifying the ACBM and suspected ACBM assumed to be ACM in the school building into one of the following categories:

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(1) Damaged or significantly damaged thermal system insulation ACM.

(2) Damaged friable surfacing ACM.

(3) Significantly damaged friable surfacing ACM.

(4) Damaged or significantly damaged friable miscellaneous ACM.

(5) ACBM with potential for damage.

(6) ACBM with potential for significant damage.

(7) Any remaining friable ACBM or friable suspected ACBM.

(c) Assessment may include the following considerations:

(1) Location and the amount of the material, both in total quantity and as a percentage of the functional space.

(2) Condition of the material, specifying:

(i) Type of damage or significant damage (e.g., flaking, blistering, water damage, or other signs of physical damage).

(ii) Severity of damage (e.g., major flaking, severely torn jackets, as opposed to occasional flaking, minor tears to jackets).

(iii) Extent or spread of damage over large areas or large percentages of the homogeneous area.

(3) Whether the material is accessible.

(4) The material's potential for disturbance.

(5) Known or suspected causes of damage or significant damage (e.g., air erosion, vandalism, vibration, water).

(6) Preventive measures which might eliminate the reasonable likelihood of undamaged ACM from becoming significantly damaged.

(d) The local education agency shall select a person accredited to develop management plans to review the results of each inspection, reinspection, and assessment for the school building and to conduct any other necessary activities in order to recommend in writing to the local education agency appropriate response actions. The accredited person shall sign and date the recommendation, provide his or her State of accreditation, and, if applicable, provide his or her accreditation number, and submit a copy of the recommendation to the person designated under Sec. 763.84 for inclusion in the management plan.

Sec. 763.90 Response actions.

(a) The local education agency shall select and implement in a timely manner the appropriate response actions in this section consistent with the assessment conducted in Sec. 763.88. The response actions selected shall be sufficient to protect human health and the environment. The local education agency may then select, from the response actions which protect human health and the environment, that action which is the least burdensome method. Nothing in this section shall be construed to prohibit removal of ACBM from a school building at any time, should removal be the preferred response action of the local education agency.

(b) If damaged or significantly damaged thermal system insulation ACM is present in a building, the local education agency shall:

(1) At least repair the damaged area.

(2) Remove the damaged material if it is not feasible, due to technological factors, to repair the damage.

(3) Maintain all thermal system insulation ACM and its covering in an intact state and undamaged condition.

(c)(1) If damaged friable surfacing ACM or damaged friable miscellaneous ACM is present in a building, the local education agency shall select from among the following response actions: encapsulation, enclosure, removal, or repair of the damaged material.

(2) In selecting the response action from among those which meet the definitional standards in Sec. 763.83, the local education agency shall determine which of these response actions protects human health and the

environment. For purposes of determining which of these response actions are the least burdensome, the local education agency may then consider local circumstances, including occupancy and use patterns within the school building, and its economic concerns, including short- and long-term costs.

(d) If significantly damaged friable surfacing ACM or significantly damaged friable miscellaneous ACM is present in a building the local education agency shall:

(1) Immediately isolate the functional space and restrict access, unless

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isolation is not necessary to protect human health and the environment.

(2) Remove the material in the functional space or, depending upon whether enclosure or encapsulation would be sufficient to protect human health and the environment, enclose or encapsulate.

(e) If any friable surfacing ACM, thermal system insulation ACM, or friable miscellaneous ACM that has potential for damage is present in a building, the local education agency shall at least implement an operations and maintenance (O&M) program, as described under Sec. 763.91.

(f) If any friable surfacing ACM, thermal system insulation ACM, or friable miscellaneous ACM that has potential for significant damage is present in a building, the local education agency shall:

(1) Implement an O&M program, as described under Sec. 763.91.

(2) Institute preventive measures appropriate to eliminate the reasonable likelihood that the ACM or its covering will become significantly damaged, deteriorated, or delaminated.

(3) Remove the material as soon as possible if appropriate preventive measures cannot be effectively implemented, or unless other response actions are determined to protect human health and the environment. Immediately isolate the area and restrict access if necessary to avoid an imminent and substantial endangerment to human health or the environment.

(g) Response actions including removal, encapsulation, enclosure, or repair, other than small-scale, short-duration repairs, shall be designed and conducted by persons accredited to design and conduct response actions.

(h) The requirements of this subpart E in no way supersede the worker protection and work practice requirements under 29 CFR 1926.58 (Occupational Safety and Health Administration (OSHA) **asbestos** worker protection standards for construction), 40 CFR part 763, subpart G (EPA **asbestos** worker protection standards for public employees), and 40 CFR part 61, subpart M (National Emission Standards for Hazardous Air Pollutants--**Asbestos**).

(i) Completion of response actions. (1) At the conclusion of any action to remove, encapsulate, or enclose ACBM or material assumed to be ACBM, a person designated by the local education agency shall visually inspect each functional space where such action was conducted to determine whether the action has been properly completed.

(2)(i) A person designated by the local education agency shall collect air samples using aggressive sampling as described in appendix A to this subpart E to monitor air for clearance after each removal, encapsulation, and enclosure project involving ACBM, except for projects that are of small-scale, short-duration.

(ii) Local education agencies shall have air samples collected under this section analyzed for **asbestos** using laboratories accredited by the National Bureau of Standards to conduct such analysis using transmission electron microscopy (TEM) or, under circumstances permitted in this section, laboratories enrolled in the American Industrial Hygiene Association Proficiency Analytical Testing Program for phase contrast microscopy (PCM).

(iii) Until the National Bureau of Standards TEM laboratory accreditation program is operational, local educational agencies shall use laboratories that use the protocol described in appendix A to subpart E of this part.

(3) Except as provided in paragraphs (i)(4), and (i)(5), of this section, an action to remove, encapsulate, or enclose ACBM shall be considered complete when the average concentration of **asbestos** of five air samples collected within the affected functional space and analyzed by the TEM method in appendix A of this subpart E, is not statistically significantly different, as determined by the Z-test calculation found in appendix A of this subpart E, from the average **asbestos** concentration of five air samples collected at the same time outside the affected functional space and analyzed in the same manner, and the average **asbestos** concentration of the three field blanks described in appendix A of this subpart E is below the filter background level, as defined in appendix A of this subpart E, of 70 structures per square millimeter (70 s/mm \2\).

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(4) An action may also be considered complete if the volume of air drawn for each of the five samples collected within the affected functional space is equal to or greater than 1,199 L of air for a 25 mm filter or equal to or greater than 2,799 L of air for a 37 mm filter, and the average concentration of **asbestos** as analyzed by the TEM method in appendix A of this subpart E, for the five air samples does not exceed the filter background level, as defined in appendix A, of 70 structures per square millimeter (70 s/mm \2\). If the average concentration of **asbestos** of the five air samples within the affected functional space exceeds 70 s/mm \2\, or if the volume of air in each of the samples is less than 1,199 L of air for a 25 mm filter or less than 2,799 L of air for a 37 mm filter, the action shall be considered complete only when the requirements of paragraph (i)(3) or (i)(5), of this section are met.

(5) At any time, a local education agency may analyze air monitoring samples collected for clearance purposes by phase contrast microscopy (PCM) to confirm completion of removal, encapsulation, or enclosure of ACBM that is greater than small-scale, short-duration and less than or equal to 160 square feet or 260 linear feet. The action shall be considered complete when the results of samples collected in the affected functional space and analyzed by phase contrast microscopy using the National Institute for Occupational Safety and Health (NIOSH) Method 7400 entitled ``Fibers'' published in the NIOSH Manual of Analytical Methods, 3rd Edition, Second Supplement, August 1987, show that the concentration of fibers for each of the five samples is less than or equal to a limit of quantitation for PCM (0.01 fibers per cubic centimeter (0.01 f/cm \3\) of air). The method is available for public inspection at the Office of the Federal Register, 800 North Capitol Street, NW, Suite 700, Washington, DC, 20408, and the Non-Confidential Information Center (NCIC) (7407), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Room B-607 NEM, 401 M St., SW., Washington, DC 20460, between the hours of 12 p.m. and 4 p.m. weekdays excluding legal holidays. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. The method is incorporated as it exists on the effective date of this rule, and a notice of any change to the method will be published in the Federal Register.

(6) To determine the amount of ACBM affected under paragraph (i)(5) of this section, the local education agency shall add the total square or linear footage of ACBM within the containment barriers used to isolate the functional space for the action to remove, encapsulate, or enclose the ACBM. Contiguous portions of material subject to such action conducted concurrently or at approximately the same time within the same school building shall not be separated to qualify under paragraph (i)(5), of this section.

[52 FR 41846, Oct. 30, 1987, as amended at 53 FR 12525, Apr. 15, 1988; 60 FR 31922, June 19, 1995; 60 FR 34465, July 3, 1995]

Sec. 763.91 Operations and maintenance.

(a) Applicability. The local education agency shall implement an operations, maintenance, and repair (O&M) program under this section whenever any friable ACBM is present or assumed to be present in a building that it leases, owns, or otherwise uses as a school building. Any material identified as nonfriable ACBM or nonfriable assumed ACBM must be treated as friable ACBM for purposes of this section when the material is about to become friable as a result of activities performed in the school building.

(b) Worker protection. Local education agencies must comply with either the OSHA **Asbestos** Construction Standard at 29 CFR 1926.1101, or the **Asbestos** Worker Protection Rule at 40 CFR 763.120, whichever is applicable.

(c) Cleaning--(1) Initial cleaning. Unless the building has been cleaned using equivalent methods within the previous 6 months, all areas of a school building where friable ACBM, damaged or significantly damaged thermal system insulation ACM, or friable suspected ACBM assumed to be ACM are present shall be cleaned at least once after the completion of the inspection required by Sec. 763.85(a) and before the initiation of any response action, other

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than O&M activities or repair, according to the following procedures:

(i) HEPA-vacuum or steam-clean all carpets.

(ii) HEPA-vacuum or wet-clean all other floors and all other horizontal surfaces.

(iii) Dispose of all debris, filters, mopheads, and cloths in sealed, leak-tight containers.

(2) Additional cleaning. The accredited management planner shall make a written recommendation to the local education agency whether additional cleaning is needed, and if so, the methods and frequency of such cleaning.

(d) Operations and maintenance activities. The local education agency shall ensure that the procedures described below to protect building occupants shall be followed for any operations and maintenance activities disturbing friable ACBM:

(1) Restrict entry into the area by persons other than those necessary to perform the maintenance project, either by physically isolating the area or by scheduling.

(2) Post signs to prevent entry by unauthorized persons.

(3) Shut off or temporarily modify the air-handling system and restrict other sources of air movement.

(4) Use work practices or other controls, such as, wet methods, protective clothing, HEPA-vacuums, mini-enclosures, glove bags, as necessary to inhibit the spread of any released fibers.

(5) Clean all fixtures or other components in the immediate work

area.

(6) Place the **asbestos** debris and other cleaning materials in a sealed, leak-tight container.

(e) Maintenance activities other than small-scale, short-duration. The response action for any maintenance activities disturbing friable ACBM, other than small-scale, short-duration maintenance activities, shall be designed by persons accredited to design response actions and conducted by persons accredited to conduct response actions.

(f) Fiber release episodes--(1) Minor fiber release episode. The local education agency shall ensure that the procedures described below are followed in the event of a minor fiber release episode (i.e., the falling or dislodging of 3 square or linear feet or less of friable ACBM): 5

(i) Thoroughly saturate the debris using wet methods.

(ii) Clean the area, as described in paragraph (e) of this section.

(iii) Place the **asbestos** debris in a sealed, leak-tight container.

(iv) Repair the area of damaged ACM with materials such as **asbestos**-free spackling, plaster, cement, or insulation, or seal with latex paint or an encapsulant, or immediately have the appropriate response action implemented as required by Sec. 763.90.

(2) Major fiber release episode. The local education agency shall ensure that the procedures described below are followed in the event of a major fiber release episode (i.e., the falling or dislodging of more than 3 square or linear feet of friable ACBM):

(i) Restrict entry into the area and post signs to prevent entry into the area by persons other than those necessary to perform the response action.

(ii) Shut off or temporarily modify the air-handling system to prevent the distribution of fibers to other areas in the building.

(iii) The response action for any major fiber release episode must be designed by persons accredited to design response actions and conducted by persons accredited to conduct response actions.

[52 FR 41846, Oct. 30, 1987, as amended at 65 FR 69216, Nov. 15, 2000]

Sec. 763.92 Training and periodic surveillance.

(a) Training. (1) The local education agency shall ensure, prior to the implementation of the O&M provisions of the management plan, that all members of its maintenance and custodial staff (custodians, electricians, heating/air conditioning engineers, plumbers, etc.) who may work in a building that contains ACBM receive awareness training of at least 2 hours, whether or not they are required to work with ACBM. New custodial and maintenance employees shall be trained within 60 days after commencement of employment. Training shall include, but not be limited to:

(i) Information regarding **asbestos** and its various uses and forms.

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(ii) Information on the health effects associated with **asbestos** exposure.

(iii) Locations of ACBM identified throughout each school building in which they work.

(iv) Recognition of damage, deterioration, and delamination of ACBM.

(v) Name and telephone number of the person designated to carry out general local education agency responsibilities under Sec. 763.84 and the availability and location of the management plan.

(2) The local education agency shall ensure that all members of its maintenance and custodial staff who conduct any activities that will

result in the disturbance of ACBM shall receive training described in paragraph (a)(1) of this section and 14 hours of additional training. Additional training shall include, but not be limited to:

(i) Descriptions of the proper methods of handling ACBM.

(ii) Information on the use of respiratory protection as contained in the EPA/NIOSH Guide to Respiratory Protection for the **Asbestos** Abatement Industry, September 1986 (EPA 560/OPPTS-86-001), available from the Director, Environmental Assistance Division (7408), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Room E-543B, 1200 Pennsylvania Ave., NW., Washington, DC 20460, Telephone: (202) 554-1404, TDD: (202) 544-0551 and other personal protection measures.

(iii) The provisions of this section and Sec. 763.91, Appendices A, C, and D of this subpart E of this part, EPA regulations contained in 40 CFR part 763, subpart G, and in 40 CFR part 61, subpart M, and OSHA regulations contained in 29 CFR 1926.58.

(iv) Hands-on training in the use of respiratory protection, other personal protection measures, and good work practices.

(3) Local education agency maintenance and custodial staff who have attended EPA-approved **asbestos** training or received equivalent training for O&M and periodic surveillance activities involving **asbestos** shall be considered trained for the purposes of this section.

(b) Periodic surveillance. (1) At least once every 6 months after a management plan is in effect, each local education agency shall conduct periodic surveillance in each building that it leases, owns, or otherwise uses as a school building that contains ACBM or is assumed to contain ACBM.

(2) Each person performing periodic surveillance shall:

(i) Visually inspect all areas that are identified in the management plan as ACBM or assumed ACBM.

(ii) Record the date of the surveillance, his or her name, and any changes in the condition of the materials.

(iii) Submit to the person designated to carry out general local education agency responsibilities under Sec. 763.84 a copy of such record for inclusion in the management plan.

[52 FR 41846, Oct. 30, 1987, as amended at 60 FR 34465, July 3, 1995; 65 FR 69216, Nov. 15, 2000]

Sec. 763.93 Management plans.

(a)(1) On or before October 12, 1988, each local education agency shall develop an **asbestos** management plan for each school, including all buildings that they lease, own, or otherwise use as school buildings, and submit the plan to an Agency designated by the Governor of the State in which the local education agency is located. The plan may be submitted in stages that cover a portion of the school buildings under the authority of the local education agency.

(2) If a building to be used as part of a school is leased or otherwise acquired after October 12, 1988, the local education agency shall include the new building in the management plan for the school prior to its use as a school building. The revised portions of the management plan shall be submitted to the Agency designated by the Governor.

(3) If a local education agency begins to use a building as a school after October 12, 1988, the local education agency shall submit a management plan for the school to the Agency designated by the Governor prior to its use as a school.

(b) On or before October 17, 1987, the Governor of each State shall notify local education agencies in the State

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regarding where to submit their management plans. States may establish administrative procedures for reviewing management plans. If the Governor does not disapprove a management plan within 90 days after receipt of the plan, the local education agency shall implement the plan.

(c) Each local education agency must begin implementation of its management plan on or before July 9, 1989, and complete implementation in a timely fashion.

(d) Each local education agency shall maintain and update its management plan to keep it current with ongoing operations and maintenance, periodic surveillance, inspection, reinspection, and response action activities. All provisions required to be included in the management plan under this section shall be retained as part of the management plan, as well as any information that has been revised to bring the plan up-to-date.

(e) The management plan shall be developed by an accredited management planner and shall include:

(1) A list of the name and address of each school building and whether the school building contains friable ACBM, nonfriable ACBM, and friable and nonfriable suspected ACBM assumed to be ACM.

(2) For each inspection conducted before the December 14, 1987:

(i) The date of the inspection.

(ii) A blueprint, diagram, or written description of each school building that identifies clearly each location and approximate square or linear footage of any homogeneous or sampling area where material was sampled for ACM, and, if possible, the exact locations where bulk samples were collected, and the dates of collection.

(iii) A copy of the analyses of any bulk samples, dates of analyses, and a copy of any other laboratory reports pertaining to the analyses.

(iv) A description of any response actions or preventive measures taken to reduce **asbestos** exposure, including if possible, the names and addresses of all contractors involved, start and completion dates of the work, and results of any air samples analyzed during and upon completion of the work.

(v) A description of assessments, required to be made under Sec. 763.88, of material that was identified before December 14, 1987, as friable ACBM or friable suspected ACBM assumed to be ACM, and the name and signature, State of accreditation, and if applicable, accreditation number of each accredited person making the assessments.

(3) For each inspection and reinspection conducted under Sec. 763.85:

(i) The date of the inspection or reinspection and the name and signature, State of accreditation and, if applicable, the accreditation number of each accredited inspector performing the inspection or reinspection.

(ii) A blueprint, diagram, or written description of each school building that identifies clearly each location and approximate square or linear footage of homogeneous areas where material was sampled for ACM, the exact location where each bulk sample was collected, date of collection, homogeneous areas where friable suspected ACBM is assumed to be ACM, and where nonfriable suspected ACBM is assumed to be ACM.

(iii) A description of the manner used to determine sampling locations, and the name and signature of each accredited inspector collecting samples, the State of accreditation, and if applicable, his or her accreditation number.

(iv) A copy of the analyses of any bulk samples collected and analyzed, the name and address of any laboratory that analyzed bulk samples, a statement that the laboratory meets the applicable requirements of Sec. 763.87(a) the date of analysis, and the name and signature of the person performing the analysis.

(v) A description of assessments, required to be made under Sec. 763.88, of all ACBM and suspected ACBM assumed to be ACM, and the name, signature, State of accreditation, and if applicable, accreditation number of each accredited person making the assessments.

(4) The name, address, and telephone number of the person designated under Sec. 763.84 to ensure that the duties of the local education agency are carried out, and the course name, and dates and hours of training taken by that person to carry out the duties.

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(5) The recommendations made to the local education agency regarding response actions, under Sec. 763.88(d), the name, signature, State of accreditation of each person making the recommendations, and if applicable, his or her accreditation number.

(6) A detailed description of preventive measures and response actions to be taken, including methods to be used, for any friable ACBM, the locations where such measures and action will be taken, reasons for selecting the response action or preventive measure, and a schedule for beginning and completing each preventive measure and response action.

(7) With respect to the person or persons who inspected for ACBM and who will design or carry out response actions, except for operations and maintenance, with respect to the ACBM, one of the following statements:

(i) If the State has adopted a contractor accreditation program under section 206(b) of Title II of the Act, a statement that the person(s) is accredited under such plan.

(ii) A statement that the local education agency used (or will use) persons who have been accredited by another State which has adopted a contractor accreditation plan under section 206(b) of Title II of the Act or is accredited by an EPA-approved course under section 206(c) of Title II of the Act.

(8) A detailed description in the form of a blueprint, diagram, or in writing of any ACBM or suspected ACBM assumed to be ACM which remains in the school once response actions are undertaken pursuant to Sec. 763.90. This description shall be updated as response actions are completed.

(9) A plan for reinspection under Sec. 763.85, a plan for operations and maintenance activities under Sec. 763.91, and a plan for periodic surveillance under Sec. 763.92, a description of the recommendation made by the management planner regarding additional cleaning under Sec. 763.91(c)(2) as part of an operations and maintenance program, and the response of the local education agency to that recommendation.

(10) A description of steps taken to inform workers and building occupants, or their legal guardians, about inspections, reinspections, response actions, and post-response action activities, including periodic reinspection and surveillance activities that are planned or in progress.

(11) An evaluation of the resources needed to complete response actions successfully and carry out reinspection, operations and maintenance activities, periodic surveillance and training.

(12) With respect to each consultant who contributed to the management plan, the name of the consultant and one of the following statements:

(i) If the State has adopted a contractor accreditation plan under section 206(b) of Title II of the Act, a statement that the consultant is accredited under such plan.

(ii) A statement that the contractor is accredited by another State which has adopted a contractor accreditation plan under section 206(b) of Title II of the Act, or is accredited by an EPA-approved course developed under section 206(c) of Title II of the Act.

(f) A local education agency may require each management plan to contain a statement signed by an accredited management plan developer that such person has prepared or assisted in the preparation of such plan or has reviewed such plan, and that such plan is in compliance with this subpart E. Such statement may not be signed by a person who, in addition to preparing or assisting in preparing the management plan, also implements (or will implement) the management plan.

(g)(1) Upon submission of a management plan to the Governor for review, a local education agency shall keep a copy of the plan in its administrative office. The management plans shall be available, without cost or restriction, for inspection by representatives of EPA and the State, the public, including teachers, other school personnel and their representatives, and parents. The local education agency may charge a reasonable cost to make copies of management plans.

(2) Each local education agency shall maintain in its administrative office a complete, updated copy of a management plan for each school under its administrative control or direction. The management plans shall be available,

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during normal business hours, without cost or restriction, for inspection by representatives of EPA and the State, the public, including teachers, other school personnel and their representatives, and parents. The local education agency may charge a reasonable cost to make copies of management plans.

(3) Each school shall maintain in its administrative office a complete, updated copy of the management plan for that school. Management plans shall be available for inspection, without cost or restriction, to workers before work begins in any area of a school building. The school shall make management plans available for inspection to representatives of EPA and the State, the public, including parents, teachers, and other school personnel and their representatives within 5 working days after receiving a request for inspection. The school may charge a reasonable cost to make copies of the management plan.

(4) Upon submission of its management plan to the Governor and at least once each school year, the local education agency shall notify in writing parent, teacher, and employee organizations of the availability of management plans and shall include in the management plan a description of the steps taken to notify such organizations, and a dated copy of the notification. In the absence of any such organizations for parents, teachers, or employees, the local education agency shall provide written notice to that relevant group of the availability of management plans and shall include in the management plan a description of the steps taken to notify such groups, and a dated copy of the notification.

(h) Records required under Sec. 763.94 shall be made by local education agencies and maintained as part of the management plan.

(i) Each management plan must contain a true and correct statement, signed by the individual designated by the local education agency under Sec. 763.84, which certifies that the general, local education agency responsibilities, as stipulated by Sec. 763.84, have been met or will be met.

Sec. 763.94 Recordkeeping.

(a) Records required under this section shall be maintained in a centralized location in the administrative office of both the school and the local education agency as part of the management plan. For each homogeneous area where all ACBM has been removed, the local education agency shall ensure that such records are retained for 3 years after the next reinspection required under Sec. 763.85(b)(1), or for an equivalent period.

(b) For each preventive measure and response action taken for friable and nonfriable ACBM and friable and nonfriable suspected ACBM assumed to be ACM, the local education agency shall provide:

(1) A detailed written description of the measure or action, including methods used, the location where the measure or action was taken, reasons for selecting the measure or action, start and completion dates of the work, names and addresses of all contractors involved, and if applicable, their State of accreditation, and accreditation numbers, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(2) The name and signature of any person collecting any air sample required to be collected at the completion of certain response actions specified by Sec. 763.90(i), the locations where samples were collected, date of collection, the name and address of the laboratory analyzing the samples, the date of analysis, the results of the analysis, the method of analysis, the name and signature of the person performing the analysis, and a statement that the laboratory meets the applicable requirements of Sec. 763.90(i)(2)(ii).

(c) For each person required to be trained under Sec. 763.92(a) (1) and (2), the local education agency shall provide the person's name and job title, the date that training was completed by that person, the location of the training, and the number of hours completed in such training.

(d) For each time that periodic surveillance under Sec. 763.92(b) is performed, the local education agency shall record the name of each person performing

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the surveillance, the date of the surveillance, and any changes in the conditions of the materials.

(e) For each time that cleaning under Sec. 763.91(c) is performed, the local education agency shall record the name of each person performing the cleaning, the date of such cleaning, the locations cleaned, and the methods used to perform such cleaning.

(f) For each time that operations and maintenance activities under Sec. 763.91(d) are performed, the local education agency shall record the name of each person performing the activity, the start and completion dates of the activity, the locations where such activity occurred, a description of the activity including preventive measures used, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(g) For each time that major **asbestos** activity under Sec. 763.91(e) is performed, the local education agency shall provide the name and signature, State of accreditation, and if applicable, the accreditation number of each person performing the activity, the start and completion dates of the activity, the locations where such activity occurred, a description of the activity including preventive measures used, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(h) For each fiber release episode under Sec. 763.91(f), the local education agency shall provide the date and location of the episode, the method of repair, preventive measures or response action taken, the name of each person performing the work, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(Approved by the Office of Management and Budget under control number 2070-0091)

Sec. 763.95 Warning labels.

(a) The local education agency shall attach a warning label immediately adjacent to any friable and nonfriable ACBM and suspected ACBM assumed to be ACM located in routine maintenance areas (such as boiler rooms) at each school building. This shall include:

(1) Friable ACBM that was responded to by a means other than removal.

(2) ACBM for which no response action was carried out.

(b) All labels shall be prominently displayed in readily visible locations and shall remain posted until the ACBM that is labeled is removed.

(c) The warning label shall read, in print which is readily visible because of large size or bright color, as follows:

CAUTION: **ASBESTOS**. HAZARDOUS. DO NOT DISTURB WITHOUT PROPER TRAINING AND EQUIPMENT.

Sec. 763.97 Compliance and enforcement.

(a) Compliance with Title II of the Act. (1) Section 207(a) of Title II of the Act (15 U.S.C. 2647) makes it unlawful for any local education agency to:

(i) Fail to conduct inspections pursuant to section 203(b) of Title II of the Act, including failure to follow procedures and failure to use accredited personnel and laboratories.

(ii) Knowingly submit false information to the Governor regarding any inspection pursuant to regulations under section 203(i) of Title II of the Act.

(iii) Fail to develop a management plan pursuant to regulations under section 203(i) of Title II of the Act.

(2) Section 207(a) of Title II of the Act (15 U.S.C. 2647) also provides that any local education agency which violates any provision of section 207 shall be liable for a civil penalty of not more than $5,000 for each day during which the violation continues. For the purposes of this subpart, a ``violation'' means a failure to comply with respect to a single school building.

(b) Compliance with Title I of the Act. (1) Section 15(1)(D) of Title I of the Act (15 U.S.C. 2614) makes it unlawful for any person to fail or refuse to comply with any requirement of Title II or any rule promulgated or order issued under Title II. Therefore, any person who violates any requirement of this subpart is in violation of section 15 of Title I of the Act.

(2) Section 15(3) of Title I of the Act (15 U.S.C. 2614) makes it unlawful for any person to fail or refuse to establish or maintain records, submit reports, notices or other information, or permit access to or copying of records, as required by this Act or a rule thereunder.

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(3) Section 15(4) (15 U.S.C. 2614) of Title I of the Act makes it unlawful for any person to fail or refuse to permit entry or inspection as required by section 11 of Title I of the Act.

(4) Section 16(a) of Title I of the Act (15 U.S.C. 2615) provides

that any person who violates any provision of section 15 of Title I of the Act shall be liable to the United States for a civil penalty in an amount not to exceed $25,000 for each such violation. Each day such a violation continues shall, for purposes of this paragraph, constitute a separate violation of section 15. A local education agency is not liable for any civil penalty under Title I of the Act for failing or refusing to comply with any rule promulgated or order issued under Title II of the Act.

(c) Criminal penalties. If any violation committed by any person (including a local education agency) is knowing or willful, criminal penalties may be assessed under section 16(b) of Title I of the Act.

(d) Injunctive relief. The Agency may obtain injunctive relief under section 208(b) of Title II of the Act to respond to a hazard which poses an imminent and substantial endangerment to human health or the environment or section 17 (15 U.S.C. 2616) of Title I of the Act to restrain any violation of section 15 of Title I of the Act or to compel the taking of any action required by or under Title I of the Act.

(e) Citizen complaints. Any citizen who wishes to file a complaint pursuant to section 207(d) of Title II of the Act should direct the complaint to the Governor of the State or the EPA **Asbestos** Ombudsman, 1200 Pennsylvania Ave., NW., Washington, DC 20460. The citizen complaint should be in writing and identified as a citizen complaint pursuant to section 207(d) of Title II of TSCA. The EPA **Asbestos** Ombudsman or the Governor shall investigate and respond to the complaint within a reasonable period of time if the allegations provide a reasonable basis to believe that a violation of the Act has occurred.

(f) Inspections. EPA may conduct inspections and review management plans under section 11 of Title I of the Act (15 U.S.C. 2610) to ensure compliance.

Sec. 763.98 Waiver; delegation to State.

(a) General. (1) Upon request from a State Governor and after notice and comment and an opportunity for a public hearing in accordance with paragraphs (b) and (c) of this section, EPA may waive some or all of the requirements of this subpart E if the State has established and is implementing or intends to implement a program of **asbestos** inspection and management that contains requirements that are at least as stringent as the requirements of this subpart E.

(2) A waiver from any requirement of this subpart E shall apply only to the specific provision for which a waiver has been granted under this section. All requirements of this subpart E shall apply until a waiver is granted under this section.

(b) Request. Each request by a Governor to waive any requirement of this subpart E shall be sent with three complete copies of the request to the Regional Administrator for the EPA Region in which the State is located and shall include:

(1) A copy of the State provisions or proposed provisions relating to its program of **asbestos** inspection and management in schools for which the request is made.

(2)(i) The name of the State agency that is or will be responsible for administering and enforcing the requirements for which a waiver is requested, the names and job titles of responsible officials in that agency, and phone numbers where the officials can be contacted.

(ii) In the event that more than one agency is or will be responsible for administering and enforcing the requirements for which a waiver is requested, a description of the functions to be performed by each agency, how the program will be coordinated by the lead agency to ensure consistency and effective administration in the **asbestos** inspection and management program within the State, the names and job titles of responsible officials in the agencies, and phone numbers where the officials can be contacted. The lead agency will serve as the central contact point for the EPA.

(3) Detailed reasons, supporting papers, and the rationale for concluding

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that the State's **asbestos** inspection and management program provisions for which the request is made are at least as stringent as the requirements of this subpart E.

(4) A discussion of any special situations, problems, and needs pertaining to the waiver request accompanied by an explanation of how the State intends to handle them.

(5) A statement of the resources that the State intends to devote to the administration and enforcement of the provisions relating to the waiver request.

(6) Copies of any specific or enabling State laws (enacted and pending enactment) and regulations (promulgated and pending promulgation) relating to the request, including provisions for assessing criminal and/or civil penalties.

(7) Assurance from the Governor, the Attorney General, or the legal counsel of the lead agency that the lead agency or other cooperating agencies have the legal authority necessary to carry out the requirements relating to the request.

(c) General notice--hearing. (1) Within 30 days after receipt of a request for a waiver, EPA will determine the completeness of the request. If EPA does not request further information within the 30-day period, the request will be deemed complete.

(2) Within 30 days after EPA determines that a request is complete, EPA will issue for publication in the Federal Register a notice that announces receipt of the request, describes the information submitted under paragraph (b) of this section, and solicits written comment from interested members of the public. Comments must be submitted within 60 days.

(3) If, during the comment period, EPA receives a written objection to a Governor's request and a request for a public hearing detailing specific objections to the granting of a waiver, EPA will schedule a public hearing to be held in the affected State after the close of the comment period and will announce the public hearing date in the Federal Register before the date of the hearing. Each comment shall include the name and address of the person submitting the comment.

(d) Criteria. EPA may waive some or all of the requirements of subpart E of this part if:

(1) The State's lead agency and other cooperating agencies have the legal authority necessary to carry out the provisions of **asbestos** inspection and management in schools relating to the waiver request.

(2) The State's program of **asbestos** inspection and management in schools relating to the waiver request and implementation of the program are or will be at least as stringent as the requirements of this subpart E.

(3) The State has an enforcement mechanism to allow it to implement the program described in the waiver request.

(4) The lead agency and any cooperating agencies have or will have qualified personnel to carry out the provisions relating to the waiver request.

(5) The State will devote adequate resources to the administration and enforcement of the **asbestos** inspection and management provisions relating to the waiver request.

(6) When specified by EPA, the State gives satisfactory assurances

that necessary steps, including specific actions it proposes to take and a time schedule for their accomplishment, will be taken within a reasonable time to conform with applicable criteria under paragraphs (d)(2) through (4) of this section.

(e) Decision. EPA will issue for publication in the Federal Register a notice announcing its decision to grant or deny, in whole or in part, a Governor's request for a waiver from some or all of the requirements of this subpart E within 30 days after the close of the comment period or within 30 days following a public hearing, whichever is applicable. The notice will include the Agency's reasons and rationale for granting or denying the Governor's request. The 30-day period may be extended if mutually agreed upon by EPA and the State.

(f) Modifications. When any substantial change is made in the administration or enforcement of a State program for which a waiver was granted under this section, a responsible official in the lead agency shall submit such changes to EPA.

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(g) Reports. The lead agency in each State that has been granted a waiver by EPA from any requirement of subpart E of this part shall submit a report to the Regional Administrator for the Region in which the State is located at least once every 12 months to include the following information:

(1) A summary of the State's implementation and enforcement activities during the last reporting period relating to provisions waived under this section, including enforcement actions taken.

(2) Any changes in the administration or enforcement of the State program implemented during the last reporting period.

(3) Other reports as may be required by EPA to carry out effective oversight of any requirement of this subpart E that was waived under this section.

(h) Oversight. EPA may periodically evaluate the adequacy of a State's implementation and enforcement of and resources devoted to carrying out requirements relating to the waiver. This evaluation may include, but is not limited to, site visits to local education agencies without prior notice to the State.

(i) Informal conference. (1) EPA may request that an informal conference be held between appropriate State and EPA officials when EPA has reason to believe that a State has failed to:

(i) Substantially comply with the terms of any provision that was waived under this section.

(ii) Meet the criteria under paragraph (d) of this section, including the failure to carry out enforcement activities or act on violations of the State program.

(2) EPA will:

(i) Specify to the State those aspects of the State's program believed to be inadequate.

(ii) Specify to the State the facts that underlie the belief of inadequacy.

(3) If EPA finds, on the basis of information submitted by the State at the conference, that deficiencies did not exist or were corrected by the State, no further action is required.

(4) Where EPA finds that deficiencies in the State program exist, a plan to correct the deficiencies shall be negotiated between the State and EPA. The plan shall detail the deficiencies found in the State program, specify the steps the State has taken or will take to remedy the deficiencies, and establish a schedule for each remedial action to be initiated.

(j) Rescission. (1) If the State fails to meet with EPA or fails to correct deficiencies raised at the informal conference, EPA will deliver to the Governor of the State and a responsible official in the lead agency a written notice of its intent to rescind, in whole or part, the waiver.

(2) EPA will issue for publication in the Federal Register a notice that announces the rescission of the waiver, describes those aspects of the State's program determined to be inadequate, and specifies the facts that underlie the findings of inadequacy.

Sec. 763.99 Exclusions.

(a) A local education agency shall not be required to perform an inspection under Sec. 763.85(a) in any sampling area as defined in 40 CFR 763.103 or homogeneous area of a school building where:

(1) An accredited inspector has determined that, based on sampling records, friable ACBM was identified in that homogeneous or sampling area during an inspection conducted before December 14, 1987. The inspector shall sign and date a statement to that effect with his or her State of accreditation and if applicable, accreditation number and, within 30 days after such determination, submit a copy of the statement to the person designated under Sec. 763.84 for inclusion in the management plan. However, an accredited inspector shall assess the friable ACBM under Sec. 763.88.

(2) An accredited inspector has determined that, based on sampling records, nonfriable ACBM was identified in that homogeneous or sampling area during an inspection conducted before December 14, 1987. The inspector shall sign and date a statement to that effect with his or her State of accreditation and if applicable, accreditation number and, within 30 days after such determination, submit a copy of the statement to the person designated under Sec. 763.84 for inclusion in the management plan. However, an accredited inspector shall identify whether material that was nonfriable has become friable

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since that previous inspection and shall assess the newly-friable ACBM under Sec. 763.88.

(3) Based on sampling records and inspection records, an accredited inspector has determined that no ACBM is present in the homogeneous or sampling area and the records show that the area was sampled, before December 14, 1987 in substantial compliance with Sec. 763.85(a), which for purposes of this section means in a random manner and with a sufficient number of samples to reasonably ensure that the area is not ACBM.

(i) The accredited inspector shall sign and date a statement, with his or her State of accreditation and if applicable, accreditation number that the homogeneous or sampling area determined not to be ACBM was sampled in substantial compliance with Sec. 763.85(a).

(ii) Within 30 days after the inspector's determination, the local education agency shall submit a copy of the inspector's statement to the EPA Regional Office and shall include the statement in the management plan for that school.

(4) The lead agency responsible for **asbestos** inspection in a State that has been granted a waiver from Sec. 763.85(a) has determined that, based on sampling records and inspection records, no ACBM is present in the homogeneous or sampling area and the records show that the area was sampled before December 14, 1987, in substantial compliance with Sec. 763.85(a). Such determination shall be included in the management plan for that school.

(5) An accredited inspector has determined that, based on records of

an inspection conducted before December 14, 1987, suspected ACBM identified in that homogeneous or sampling area is assumed to be ACM. The inspector shall sign and date a statement to that effect, with his or her State of accreditation and if applicable, accreditation number and, within 30 days of such determination, submit a copy of the statement to the person designated under Sec. 763.84 for inclusion in the management plan. However, an accredited inspector shall identify whether material that was nonfriable suspected ACBM assumed to be ACM has become friable since the previous inspection and shall assess the newly friable material and previously identified friable suspected ACBM assumed to be ACM under Sec. 763.88.

(6) Based on inspection records and contractor and clearance records, an accredited inspector has determined that no ACBM is present in the homogeneous or sampling area where **asbestos** removal operations have been conducted before December 14, 1987, and shall sign and date a statement to that effect and include his or her State of accreditation and, if applicable, accreditation number. The local education agency shall submit a copy of the statement to the EPA Regional Office and shall include the statement in the management plan for that school.

(7) An architect or project engineer responsible for the construction of a new school building built after October 12, 1988, or an accredited inspector signs a statement that no ACBM was specified as a building material in any construction document for the building, or, to the best of his or her knowledge, no ACBM was used as a building material in the building. The local education agency shall submit a copy of the signed statement of the architect, project engineer, or accredited inspector to the EPA Regional Office and shall include the statement in the management plan for that school.

(b) The exclusion, under paragraphs (a) (1) through (4) of this section, from conducting the inspection under Sec. 763.85(a) shall apply only to homogeneous or sampling areas of a school building that were inspected and sampled before October 17, 1987. The local education agency shall conduct an inspection under Sec. 763.85(a) of all areas inspected before October 17, 1987, that were not sampled or were not assumed to be ACM.

(c) If ACBM is subsequently found in a homogeneous or sampling area of a local education agency that had been identified as receiving an exclusion by an accredited inspector under paragraphs (a) (3), (4), (5) of this section, or an architect, project engineer or accredited inspector under paragraph

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(a)(7) of this section, the local education agency shall have 180 days following the date of identification of ACBM to comply with this subpart E.

Appendix A to Subpart E of Part 763--Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions

I. Introduction

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for **asbestos** by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis.

EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

II. Mandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. Analytical sensitivity--Airborne **asbestos** concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm\3\.
2. Asbestiform--A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.
3. Aspect ratio--A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.
4. Bundle--A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
5. Clean area--A controlled environment which is maintained and monitored to assure a low probability of **asbestos** contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm\2\ in an area of 0.057 mm\2\ (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm\2\ for any single preparation for that same area.
6. Cluster--A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
7. ED--Electron diffraction.
8. EDXA--Energy dispersive X-ray analysis.
9. Fiber--A structure greater than or equal to 0.5 [mu]m in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.
10. Grid--An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.
11. Intersection--Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.
12. Laboratory sample coordinator--That person responsible for the conduct of sample handling and the certification of the testing procedures.
13. Filter background level--The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm\2\.
14. Matrix--Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
15. NSD--No structure detected.
16. Operator--A person responsible for the TEM instrumental analysis of the sample.

17. PCM--Phase contrast microscopy.
18. SAED--Selected area electron diffraction.
19. SEM--Scanning electron microscope.
20. STEM--Scanning transmission electron microscope.
21. Structure--a microscopic bundle, cluster, fiber, or matrix which may contain **asbestos**.
22. S/cm\3\--Structures per cubic centimeter.
23. S/mm\2\--Structures per square millimeter.
24. TEM--Transmission electron microscope.

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B. Sampling

1. The sampling agency must have written quality control procedures and documents which verify compliance.
2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3, and 5 of Unit II.J.).
3. Sampling for airborne **asbestos** following an abatement action must use commercially available cassettes.
4. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of **asbestos** which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm\2\ in an area of 0.057 mm\2\ (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm\2\ for that same area is acceptable for this method.
5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to 0.4 [mu]m or mixed cellulose ester having a pore size less than or equal to 0.45 [mu]m.
6. Place these filters in series with a 5.0 [mu]m backup filter (to serve as a diffuser) and a support pad. See the following Figure 1:

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7. Reloading of used cassettes is not permitted.
8. Orient the cassette downward at approximately 45 degrees from the horizontal.
9. Maintain a log of all pertinent sampling information.

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10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.
11. Record all calibration information.
12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.
14. The final plastic barrier around the abatement area remains in place for the sampling period.
15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.B.7.d.)
16. Select an appropriate flow rate equal to or greater than 1 liter

per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

17. A minimum of 13 samples are to be collected for each testing site consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each abatement area.

ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

19. The following Table I specifies volume ranges to be used:

[[Page 732]]

[GRAPHIC] [TIFF OMITTED] TC01AP92.002

20. Ensure that the sampler is turned upright before interrupting the pump flow.

21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

22. Ensure that the samples are stored in a secure and representative location.

23. Do not change containers if portions of these filters are taken for other purposes.

24. A summary of Sample Data Quality Objectives is shown in the following Table II:

[[Page 733]]

[GRAPHIC] [TIFF OMITTED] TC01AP92.003

C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples.

D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

E. Sample Preparation

1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.

2. Wet-wipe the exterior of the cassettes to minimize contamination

possibilities before taking them into the clean room facility.

3. Perform sample preparation in a well-equipped clean facility.

<ls-thn-eq>Note: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18 s/mm\2\ in an area of 0.057 mm\2\ (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm\2\ for that same area.

4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.

5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.

a. TEM Grid Opening Area measurement must be done as follows:

i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.

ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field diameters. From the data, calculate the field area for an average grid opening.

iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G. or other equivalent methods may be used.

c. TEM specimen preparation from mixed cellulose ester (MCE) filters.

i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)

ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a

[[Page 734]]

known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate asher and operating conditions will then be set such that a 1-2 [mu]m (10 percent) layer of collapsed surface will be removed.

iii. Procedures as described in Unit III. or other equivalent methods may be used to prepare samples.

F. TEM Method

1. An 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment

or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.

2. Determination of Camera Constant and ED Pattern Analysis. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in **asbestos** analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.

3. Magnification Calibration. The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate a eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.

5. Microscope settings: 80-120 kV, grid assessment 250-1,000X, then 15,000-20,000X screen magnification for analysis.

6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

7. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.

8. Reject the grid if:

a. Less than 50 percent of the grid openings covered by the replica are intact.

b. The replica is doubled or folded.

c. The replica is too dark because of incomplete dissolution of the filter.

9. Recording Rules.

a. Any continuous grouping of particles in which an **asbestos** fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 [mu]m is detected shall be recorded on the count sheet. These will be designated **asbestos** structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. See the following Figure 2:

[[Page 735]]

[GRAPHIC] [TIFF OMITTED] TC01AP92.004

[[Page 736]]

[GRAPHIC] [TIFF OMITTED] TC01AP92.005

i. Fiber. A structure having a minimum length greater than or equal to 0.5 [mu]m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. Bundle. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. Cluster. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

iv. Matrix. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

b. Separate categories will be maintained for fibers less than 5 [mu]m and for fibers equal to or greater than 5 [mu]m in length.

c. Record NSD when no structures are detected in the field.

d. Visual identification of electron diffraction (ED) patterns is required for each **asbestos** structure counted which would cause the

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analysis to exceed the 70 s/mm\2\ concentration. (Generally this means the first four fibers identified as **asbestos** must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.

f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm\2\ concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

g. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm\2\ concentration, the fact that they are not **asbestos** must be confirmed by EDXA or measurement of a zone axis diffraction pattern.

h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70 s/mm\2\ have been exceeded for a particular sample.

i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70 s/mm\2\ have been exceeded for a particular sample.)

j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.

k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.

l. If an EDXA spectrum was attempted but not observed, record N on the count sheet.

m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.

10. Classification Rules.

a. Fiber. A structure having a minimum length greater than or equal to 0.5 [mu]m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

b. Bundle. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

c. Cluster. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

d. Matrix. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

G. Sample Analytical Sequence

1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.

2. Carry out visual inspection of work site prior to air monitoring.

3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix.

H. Reporting

1. The following information must be reported to the client for each sample analyzed:

a. Concentration in structures per square millimeter and structures per cubic centimeter.

b. Analytical sensitivity used for the analysis.

c. Number of **asbestos** structures.

d. Area analyzed.

e. Volume of air sampled (which must be initially supplied to lab by client).

f. Copy of the count sheet must be included with the report.

g. Signature of laboratory official to indicate that the laboratory met specifications of the method.

h. Report form must contain official laboratory identification (e.g., letterhead).

i. Type of **asbestos**.

I. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne **asbestos** requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the

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operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:
[GRAPHIC] [TIFF OMITTED] TC01AP92.006

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
2. Check all laboratory reagents and supplies for acceptable **asbestos** background levels.
3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.
4. Prepare multiple grids of each sample.

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5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm \2\ per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.
6. Perform a system check on the transmission electron microscope daily.
7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.I.
8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.I.
9. Validate all data entries.
10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.I.
11. Record an electron diffraction pattern of one **asbestos** structure from every five samples that contain **asbestos**. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual

identification based on his measured patterns.

12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures.

J. References

For additional background information on this method, the following references should be consulted.

1. ``Guidance for Controlling **Asbestos**-Containing Materials in Buildings,'' EPA 560/5-85-024, June 1985.
2. ``Measuring Airborne **Asbestos** Following an Abatement Action,'' USEPA, Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
3. Small, John and E. Steel. **Asbestos** Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.
7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London, ``Proposed Analytical Method for Determination of **Asbestos** in Air.''
8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., ``Standard Operating Procedure for Determination of Airborne **Asbestos** Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters,'' WERL SOP 87-1, March 5, 1987.
9. NIOSH Method 7402 for **Asbestos** Fibers, 12-11-86 Draft.
10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, ``Methodology for the Measurement of Airborne **Asbestos** by Electron Microscopy,'' Draft report, USEPA Contract 68-02-3266, July 1984.
11. ``Guidance to the Preparation of Quality Assurance Project Plans,'' USEPA, Office of Pollution Prevention and Toxics, 1984.

III. Nonmandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. Analytical sensitivity--Airborne **asbestos** concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 s/cm\3\.
2. Asbestiform--A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.
3. Aspect ratio--A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.
4. Bundle--A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
5. Clean area--A controlled environment which is maintained and monitored to assure a low probability of **asbestos** contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has

an average of less than 18 structures/mm\2\ in an area of 0.057 mm\2\ (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm\2\ for no more than one single preparation for that same area.

6. Cluster--A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. ED--Electron diffraction.

8. EDXA--Energy dispersive X-ray analysis.

9. Fiber--A structure greater than or equal to 0.5 [mu]m in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

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10. Grid--An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. Intersection--Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. Laboratory sample coordinator--That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. Filter background level--The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on blanks (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm\2\.

14. Matrix--Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. NSD--No structure detected.

16. Operator--A person responsible for the TEM instrumental analysis of the sample.

17. PCM--Phase contrast microscopy.

18. SAED--Selected area electron diffraction.

19. SEM--Scanning electron microscope.

20. STEM--Scanning transmission electron microscope.

21. Structure--a microscopic bundle, cluster, fiber, or matrix which may contain **asbestos**.

22. S/cm\3\--Structures per cubic centimeter.

23. S/mm\2\--Structures per square millimeter.

24. TEM--Transmission electron microscope.

B. Sampling

1. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (See References 1, 2, and 5 of Unit III.L.) Special precautions should be taken to avoid contamination of the sample. For example, materials that have not been prescreened for their **asbestos** background content should not be used; also, sample handling procedures which do not take cross contamination possibilities into account should not be used.

2. Material and supply checks for **asbestos** contamination should be made on all critical supplies, reagents, and procedures before their use in a monitoring study.

3. Quality control and quality assurance steps are needed to identify problem areas and isolate the cause of the contamination (see Reference 5 of Unit III.L.). Control checks shall be permanently recorded to document the quality of the information produced. The

sampling firm must have written quality control procedures and documents which verify compliance. Independent audits by a qualified consultant or firm should be performed once a year. All documentation of compliance should be retained indefinitely to provide a guarantee of quality. A summary of Sample Data Quality Objectives is shown in Table II of Unit II.B.

4. Sampling materials.

a. Sample for airborne **asbestos** following an abatement action using commercially available cassettes.

b. Use either a cowling or a filter-retaining middle piece. Conductive material may reduce the potential for particulates to adhere to the walls of the cowl.

c. Cassettes must be verified as ``clean'' prior to use in the field. If packaged filters are used for loading or preloaded cassettes are purchased from the manufacturer or a distributor, the manufacturer's name and lot number should be entered on all field data sheets provided to the laboratory, and are required to be listed on all reports from the laboratory.

d. Assemble the cassettes in a clean facility (See definition of clean area under Unit III.A.).

e. Reloading of used cassettes is not permitted.

f. Use sample collection filters which are either polycarbonate having a pore size of less than or equal to 0.4 [mu]m or mixed cellulose ester having a pore size of less than or equal to 0.45 [mu]m.

g. Place these filters in series with a backup filter with a pore size of 5.0 [mu]m (to serve as a diffuser) and a support pad. See the following Figure 1:

[[Page 741]]

[GRAPHIC] [TIFF OMITTED] TC01AP92.007

h. When polycarbonate filters are used, position the highly reflective face such that the incoming particulate is received on this surface.

i. Seal the cassettes to prevent leakage around the filter edges or between cassette part joints. A mechanical press may be useful to achieve a reproducible leak-free seal.

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Shrink fit gel-bands may be used for this purpose and are available from filter manufacturers and their authorized distributors.

j. Use wrinkle-free loaded cassettes in the sampling operation.

5. Pump setup.

a. Calibrate the sampling pump over the range of flow rates and loads anticipated for the monitoring period with this flow measuring device in series. Perform this calibration using guidance from EPA Method 2A each time the unit is sent to the field (See Reference 6 of Unit III.L.).

b. Configure the sampling system to preclude pump vibrations from being transmitted to the cassette by using a sampling stand separate from the pump station and making connections with flexible tubing.

c. Maintain continuous smooth flow conditions by damping out any pump action fluctuations if necessary.

d. Check the sampling system for leaks with the end cap still in place and the pump operating before initiating sample collection. Trace and stop the source of any flow indicated by the flowmeter under these conditions.

e. Select an appropriate flow rate equal to or greater than 1 L/min or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

f. Orient the cassette downward at approximately 45 degrees from the horizontal.

g. Maintain a log of all pertinent sampling information, such as pump identification number, calibration data, sample location, date, sample identification number, flow rates at the beginning, middle, and end, start and stop times, and other useful information or comments. Use of a sampling log form is recommended. See the following Figure 2:

[[Page 743]]

[GRAPHIC] [TIFF OMITTED] TC01AP92.008

h. Initiate a chain of custody procedure at the start of each sampling, if this is requested by the client.

i. Maintain a close check of all aspects of the sampling operation on a regular basis.

j. Continue sampling until at least the minimum volume is collected, as specified in the following Table I:

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[GRAPHIC] [TIFF OMITTED] TC01AP92.009

k. At the conclusion of sampling, turn the cassette upward before stopping the flow to minimize possible particle loss. If the sampling is resumed, restart the flow before reorienting the cassette downward. Note the condition of the filter at the conclusion of sampling.

l. Double check to see that all information has been recorded on the data collection forms and that the cassette is securely closed and appropriately identified using a waterproof label. Protect cassettes in individual clean resealed polyethylene bags. Bags are to be used for storing cassette caps when they are removed for sampling purposes. Caps and plugs should only be removed or replaced using clean hands or clean disposable plastic gloves.

m. Do not change containers if portions of these filters are taken for other purposes.

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6. Minimum sample number per site. A minimum of 13 samples are to be collected for each testing consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 sec and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each ambient area.

ii. At one of the ambient sites.

(Note: Do not leave the blank open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

7. Abatement area sampling.

a. Conduct final clearance sampling only after the primary containment barriers have been removed; the abatement area has been thoroughly dried; and, it has passed visual inspection tests by qualified personnel. (See Reference 1 of Unit III.L.)

b. Containment barriers over windows, doors, and air passageways must remain in place until the TEM clearance sampling and analysis is completed and results meet clearance test criteria. The final plastic barrier remains in place for the sampling period.

c. Select sampling sites in the abatement area on a random basis to provide unbiased and representative samples.

d. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust.

i. Equipment used in aggressive sampling such as a leaf blower and/ or fan should be properly cleaned and decontaminated before use.

ii. Air filtration units shall remain on during the air monitoring period.

iii. Prior to air monitoring, floors, ceiling and walls shall be swept with the exhaust of a minimum one (1) horsepower leaf blower.

iv. Stationary fans are placed in locations which will not interfere with air monitoring equipment. Fan air is directed toward the ceiling. One fan shall be used for each 10,000 ft \3\ of worksite.

v. Monitoring of an abatement work area with high-volume pumps and the use of circulating fans will require electrical power. Electrical outlets in the abatement area may be used if available. If no such outlets are available, the equipment must be supplied with electricity by the use of extension cords and strip plug units. All electrical power supply equipment of this type must be approved Underwriter Laboratory equipment that has not been modified. All wiring must be grounded. Ground fault interrupters should be used. Extreme care must be taken to clean up any residual water and ensure that electrical equipment does not become wet while operational.

vi. Low volume pumps may be carefully wrapped in 6-mil polyethylene to insulate the pump from the air. High volume pumps cannot be sealed in this manner since the heat of the motor may melt the plastic. The pump exhausts should be kept free.

vii. If recleaning is necessary, removal of this equipment from the work area must be handled with care. It is not possible to completely decontaminate the pump motor and parts since these areas cannot be wetted. To minimize any problems in this area, all equipment such as fans and pumps should be carefully wet wiped prior to removal from the abatement area. Wrapping and sealing low volume pumps in 6-mil polyethylene will provide easier decontamination of this equipment. Use of clean water and disposable wipes should be available for this purpose.

e. Pump flow rate equal to or greater than 1 L/min or less than 10 L/min may be used for 25 mm cassettes. The larger cassette diameters may have comparably increased flow.

f. Sample a volume of air sufficient to ensure the minimum quantitation limits. (See Table I of Unit III.B.5.j.)

8. Ambient sampling.

a. Position ambient samplers at locations representative of the air entering the abatement site. If makeup air entering the abatement site is drawn from another area of the building which is outside of the abatement area, place the pumps in the building, pumps should be placed out of doors located near the building and away from any obstructions that may influence wind patterns. If construction is in progress immediately outside the enclosure, it may be necessary to select another ambient site. Samples should be representative of any air entering the work site.

b. Locate the ambient samplers at least 3 ft apart and protect them from adverse weather conditions.

c. Sample same volume of air as samples taken inside the abatement site.

C. Sample Shipment

1. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

2. Select a rigid shipping container and pack the cassettes upright in a noncontaminating nonfibrous medium such as a bubble pack. The use of resealable polyethylene bags may help to prevent jostling of individual cassettes.

3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.

4. Include a shipping bill and a detailed listing of samples shipped, their descriptions

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and all identifying numbers or marks, sampling data, shipper's name, and contact information. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.

5. Hand-carry samples to the laboratory in an upright position if possible; otherwise choose that mode of transportation least likely to jar the samples in transit.

6. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain of custody and sample tracking procedures. This will also help the laboratory schedule timely analysis for the samples when they are received.

D. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne **asbestos** requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined, and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the text below.

1. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of **asbestos** which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm\2\ in an area of 0.057 mm\2\ (nominally 10 200-mesh grid openings) and a maximum of 53 s/mm\2\ for that same area for any single preparation is acceptable for this method.

2. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter--not the filter which will be used in sampling--before and after the sampling operation.

3. Record all calibration information with the data to be used on a

standard sampling form.

4. Ensure that the samples are stored in a secure and representative location.

5. Ensure that mechanical calibrations from the pump will be minimized to prevent transferral of vibration to the cassette.

6. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by installing a damping chamber if necessary.

7. Open a loaded cassette momentarily at one of the indoor sampling sites when sampling is initiated. This sample will serve as an indoor field blank.

8. Open a loaded cassette momentarily at one of the outdoor sampling sites when sampling is initiated. This sample will serve as an outdoor field blank.

9. Carry a sealed blank into the field with each sample series. Do not open this cassette in the field.

10. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

11. Ensure that the sampler is turned upright before interrupting the pump flow.

12. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

E. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Adhere to the following procedures to ensure both the continued chain-of-custody and the accountability of all samples passing through the laboratory:

a. Note the condition of the shipping package and data written on it upon receipt.

b. Retain all bills of lading or shipping slips to document the shipper and delivery time.

c. Examine the chain-of-custody seal, if any, and the package for its integrity.

d. If there has been a break in the seal or substantive damage to the package, the sample coordinator shall immediately notify the shipper and a responsible laboratory manager before any action is taken to unpack the shipment.

e. Packages with significant damage shall be accepted only by the responsible laboratory manager after discussions with the client.

3. Unwrap the shipment in a clean, uncluttered facility. The sample coordinator or his or her designee will record the contents, including a description of each item and all identifying numbers or marks. A

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Sample Receiving Form to document this information is attached for use when necessary. (See the following Figure 3.)
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Note: The person breaking the chain-of-custody seal and itemizing the contents assumes responsibility for the shipment and signs documents accordingly.

4. Assign a laboratory number and schedule an analysis sequence.

5. Manage all chain-of-custody samples within the laboratory such that their integrity can be ensured and documented.

F. Sample Preparation

1. Personnel not affiliated with the Abatement Contractor shall be used to prepare samples and conduct TEM analysis. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them to the clean sample preparation facility.

2. Perform sample preparation in a well-equipped clean facility.

Note: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA filtered. The cumulative analytical blank concentration must average less than 18 s/mm\2\ in an area of 0.057 s/mm\2\ (nominally 10 200-mesh grid openings) with no more than one single preparation to exceed 53 s/mm\2\ for that same area.

3. Preparation areas for air samples must be separated from preparation areas for bulk samples. Personnel must not prepare air samples if they have previously been preparing bulk samples without performing appropriate personal hygiene procedures, i.e., clothing change, showering, etc.

4. Preparation. Direct preparation techniques are required. The objective is to produce an intact carbon film containing the particulates from the filter surface which is sufficiently clear for TEM analysis. Currently recommended direct preparation procedures for polycarbonate (PC) and mixed cellulose ester (MCE) filters are described in Unit III.F.7. and 8. Sample preparation is a subject requiring additional research. Variation on those steps which do not substantively change the procedure, which improve filter clearing or which reduce contamination problems in a laboratory are permitted.

a. Use only TEM grids that have had grid opening areas measured according to directions in Unit III.J.

b. Remove the inlet and outlet plugs prior to opening the cassette to minimize any pressure differential that may be present.

c. Examples of techniques used to prepare polycarbonate filters are described in Unit III.F.7.

d. Examples of techniques used to prepare mixed cellulose ester filters are described in Unit III.F.8.

e. Prepare multiple grids for each sample.

f. Store the three grids to be measured in appropriately labeled grid holders or polyethylene capsules.

5. Equipment.

a. Clean area.

b. Tweezers. Fine-point tweezers for handling of filters and TEM grids.

c. Scalpel Holder and Curved No. 10 Surgical Blades.

d. Microscope slides.

e. Double-coated adhesive tape.

f. Gummed page reinforcements.

g. Micro-pipet with disposal tips 10 to 100 [mu]L variable volume.

h. Vacuum coating unit with facilities for evaporation of carbon. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surface by oil from the pumping system. The vacuum-coating unit can also be used for deposition of a thin film of gold.

i. Carbon rod electrodes. Spectrochemically pure carbon rods are required for use in the vacuum evaporator for carbon coating of filters.

j. Carbon rod sharpener. This is used to sharpen carbon rods to a neck. The use of necked carbon rods (or equivalent) allows the carbon to be applied to the filters with a minimum of heating.

k. Low-temperature plasma asher. This is used to etch the surface of collapsed mixed cellulose ester (MCE) filters. The asher should be supplied with oxygen, and should be modified as necessary to provide a throttle or bleed valve to control the speed of the vacuum to minimize disturbance of the filter. Some early models of ashers admit air too rapidly, which may disturb particulates on the surface of the filter during the etching step.

l. Glass petri dishes, 10 cm in diameter, 1 cm high. For prevention of excessive evaporation of solvent when these are in use, a good seal must be provided between the base and the lid. The seal can be improved by grinding the base and lid together with an abrasive grinding material.

m. Stainless steel mesh.

n. Lens tissue.

o. Copper 200-mesh TEM grids, 3 mm in diameter, or equivalent.

p. Gold 200-mesh TEM grids, 3 mm in diameter, or equivalent.

q. Condensation washer.

r. Carbon-coated, 200-mesh TEM grids, or equivalent.

s. Analytical balance, 0.1 mg sensitivity.

t. Filter paper, 9 cm in diameter.

u. Oven or slide warmer. Must be capable of maintaining a temperature of 65-70 deg.C.

v. Polyurethane foam, 6 mm thickness.

w. Gold wire for evaporation.

6. Reagents.

a. General. A supply of ultra-clean, fiber-free water must be available for washing of all components used in the analysis. Water

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that has been distilled in glass or filtered or deionized water is satisfactory for this purpose. Reagents must be fiber-free.

b. Polycarbonate preparation method--chloroform.

c. Mixed Cellulose Ester (MCE) preparation method--acetone or the Burdette procedure (Ref. 7 of Unit III.L.).

7. TEM specimen preparation from polycarbonate filters.

a. Specimen preparation laboratory. It is most important to ensure that contamination of TEM specimens by extraneous **asbestos** fibers is minimized during preparation.

b. Cleaning of sample cassettes. Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces.

c. Preparation of the carbon evaporator. If the polycarbonate filter has already been carbon-coated prior to receipt, the carbon coating step will be omitted, unless the analyst believes the carbon film is too thin. If there is a need to apply more carbon, the filter will be treated in the same way as an uncoated filter. Carbon coating must be performed with a high-vacuum coating unit. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application, and must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 to 12 cm from the surface of a microscope slide held in the rotating and tilting device.

d. Selection of filter area for carbon coating. Before preparation of the filters, a 75 mmx50 mm microscope slide is washed and dried. This slide is used to support strips of filter during the carbon evaporation. Two parallel strips of double-sided adhesive tape are applied along the length of the slide. Polycarbonate filters are easily stretched during handling, and cutting of areas for further preparation must be performed with great care. The filter and the MCE backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. The filter can be cut with a curved scalpel blade by rocking the blade from the point placed in contact with the filter. The process can be repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of polycarbonate filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide. The filter strip can be held with fine-point tweezers and supported underneath by the scalpel blade during placement on the microscope slide. The analyst can place several such strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and tweezers before handling a new sample. The filter strips should be identified by etching the glass slide or marking the slide using a marker insoluble in water and solvents. After the filter strip has been cut from each filter, the residual parts of the filter must be returned to the cassette and held in position by reassembly of the cassette. The cassette will then be archived for a period of 30 days or returned to the client upon request.

e. Carbon coating of filter strips. The glass slide holding the filter strips is placed on the rotation-tilting device, and the evaporator chamber is evacuated. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation is too rapid, the strips of polycarbonate filter will begin to curl, which will lead to cross-linking of the surface material and make it relatively insoluble in chloroform. An experienced analyst can judge the thickness of carbon film to be applied, and some test should be made first on unused filters. If the film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film will also lead to a TEM image that is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film should be as thin as possible and remain intact on most of the grid openings of the TEM specimen intact.

f. Preparation of the Jaffe washer. The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used. A washer consisting of a simple stainless steel bridge is recommended. Several pieces of lens tissue approximately 1.0 cmx0.5 cm are placed on the stainless steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. See References 8 and 10 of Unit III.L.

g. Placing of specimens into the Jaffe washer. The TEM grids are first placed on a piece of lens tissue so that individual grids can be picked up with tweezers. Using a curved scalpel blade, the analyst excises three 3 mm square pieces of the carbon-coated polycarbonate filter from the filter strip. The three squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. The piece of filter is placed on a TEM specimen grid with the shiny side of the TEM grid facing upwards, and the whole assembly is placed boldly onto the saturated lens tissue in the Jaffe washer. If carbon-coated grids are used, the filter should be

placed carbon-coated side down. The three excised squares of filters are placed on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight.

h. Condensation washing. It has been found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if the surface of the filter was overheated during the carbon evaporation. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuration of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a low result. Undissolved filter medium also compromises the ability to obtain ED patterns. Before they are counted, TEM grids must be examined critically to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids after the initial Jaffe washer treatment, with chloroform as the solvent, clears all residual filter medium in a period of approximately 1 hour. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of the condensation washer, and the washer is operated for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washer step may be unnecessary.

8. TEM specimen preparation from MCE filters.

a. This method of preparing TEM specimens from MCE filters is similar to that specified in NIOSH Method 7402. See References 7, 8, and 9 of Unit III.L.

b. Upon receipt at the analytical laboratory, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces before entering the clean sample preparation area.

c. Remove a section from any quadrant of the sample and blank filters.

d. Place the section on a clean microscope slide. Affix the filter section to the slide with a gummed paged reinforcement or other suitable means. Label the slide with a water and solvent-proof marking pen.

e. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 minutes for the sample filter to fuse and clear.

f. Plasma etching of the collapsed filter is required.

i. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. This is one area of the method that requires further evaluation. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for a particular asher and operating conditions will then be set such that a 1-2 [mu]m (10 percent) layer of collapsed surface will be removed.

ii. Place the slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.

g. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 mmx5 mm section of graphite rod onto the cleared filter. Remove the slide to a clean, dry, covered petri dish.

h. Prepare a second petri dish as a Jaffe washer with the wicking substrate prepared from filter or lens paper placed on top of a 6 mm thick disk of clean spongy polyurethane foam. Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent. The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

i. Place carbon-coated TEM grids face up on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish lid and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated. The level of acetone should be just high enough to saturate the filter paper without creating puddles.

j. Remove about a quarter section of the carbon-coated filter samples from the glass slides using a surgical knife and tweezers. Carefully place the section of the filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation.

G. TEM Method

1. Instrumentation.

a. Use an 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm

in diameter at crossover. The microscope shall be calibrated routinely (see Unit III.J.) for magnification and camera constant.

b. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory. This must be an Energy Dispersive X-ray Detector mounted on TEM column and associated hardware/software to collect, save, and read out spectral information. Calibration of Multi-Channel Analyzer shall be checked regularly for A1 at 1.48 KeV and Cu at 8.04 KeV, as well as the manufacturer's procedures.

i. Standard replica grating may be used to determine magnification (e.g., 2160 lines/mm).

ii. Gold standard may be used to determine camera constant.

c. Use a specimen holder with single tilt and/or double tilt capabilities.

2. Procedure.

a. Start a new Count Sheet for each sample to be analyzed. Record on count sheet: analyst's initials and date; lab sample number; client sample number microscope identification; magnification for analysis; number of predetermined grid openings to be analyzed; and grid identification. See the following Figure 4:

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b. Check that the microscope is properly aligned and calibrated according to the manufacturer's specifications and instructions.

c. Microscope settings: 80-120 kV, grid assessment 250-1000X, then 15,000-20,000X screen magnification for analysis.

d. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

e. Determine the suitability of the grid.

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i. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading shall not be analyzed.

ii. Examine the grid at low magnification (<1000X) to determine its suitability for detailed study at higher magnifications.

iii. Reject the grid if:

(1) Less than 50 percent of the grid openings covered by the replica are intact.

(2) It is doubled or folded.

(3) It is too dark because of incomplete dissolution of the filter.

iv. If the grid is rejected, load the next sample grid.

v. If the grid is acceptable, continue on to Step 6 if mapping is to be used; otherwise proceed to Step 7.

f. Grid Map (Optional).

i. Set the TEM to the low magnification mode.

ii. Use flat edge or finder grids for mapping.

iii. Index the grid openings (fields) to be counted by marking the acceptable fields for one-half (0.5) of the area needed for analysis on each of the two grids to be analyzed. These may be marked just before examining each grid opening (field), if desired.

iv. Draw in any details which will allow the grid to be properly oriented if it is reloaded into the microscope and a particular field is to be reliably identified.

g. Scan the grid.

i. Select a field to start the examination.

ii. Choose the appropriate magnification (15,000 to 20,000X screen magnification).

iii. Scan the grid as follows.

(1) At the selected magnification, make a series of parallel traverses across the field. On reaching the end of one traverse, move the image one window and reverse the traverse.

Note: A slight overlap should be used so as not to miss any part of the grid opening (field).

(2) Make parallel traverses until the entire grid opening (field) has been scanned.

h. Identify each structure for appearance and size.

i. Appearance and size: Any continuous grouping of particles in which an **asbestos** fiber within aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 [mu]m is detected shall be recorded on the count sheet. These will be designated **asbestos** structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. See the following Figure 5:

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[GRAPHIC] [TIFF OMITTED] TC01AP92.012

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[GRAPHIC] [TIFF OMITTED] TC01AP92.013

An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. Combinations such as a matrix and cluster, matrix and bundle, or bundle and cluster are categorized by the dominant fiber quality--cluster, bundle, and matrix, respectively. Separate categories will be maintained for fibers less than 5 [mu]m and for fibers greater than or equal to 5 [mu]m in length. Not required, but useful, may be to record the fiber length in 1 [mu]m intervals. (Identify each structure morphologically and analyze it as it enters the ``window''.)

(1) Fiber. A structure having a minimum length greater than 0.5 [mu]m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed, no intersections.

(2) Bundle. A structure composed of 3 or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

(3) Cluster. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings must have more than 2 intersections.

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(4) Matrix. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

(5) NSD. Record NSD when no structures are detected in the field.

(6) Intersection. Non-parallel touching or crossing of fibers, with the projection having an aspect ratio 5:1 or greater.

ii. Structure Measurement.

(1) Recognize the structure that is to be sized.

(2) Memorize its location in the ``window'' relative to the sides, inscribed square and to other particulates in the field so this exact location can be found again when scanning is resumed.

(3) Measure the structure using the scale on the screen.

(4) Record the length category and structure type classification on the count sheet after the field number and fiber number.

(5) Return the fiber to its original location in the window and scan the rest of the field for other fibers; if the direction of travel is not remembered, return to the right side of the field and begin the traverse again.

i. Visual identification of Electron Diffraction (ED) patterns is required for each **asbestos** structure counted which would cause the analysis to exceed the 70 s/mm\2\ concentration. (Generally this means the first four fibers identified as **asbestos** must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)

ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.

(1) Chrysotile: The chrysotile **asbestos** pattern has characteristic streaks on the layer lines other than the central line and some streaking also on the central line. There will be spots of normal sharpness on the central layer line and on alternate lines (2nd, 4th, etc.). The repeat distance between layer lines is 0.53 nm and the center doublet is at 0.73 nm. The pattern should display (002), (110), (130) diffraction maxima; distances and geometry should match a chrysotile pattern and be measured semiquantitatively.

(2) Amphibole Group [includes grunerite (amosite), crocidolite, anthophyllite, tremolite, and actinolite]: Amphibole **asbestos** fiber patterns show layer lines formed by very closely spaced dots, and the repeat distance between layer lines is also about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology.

iii. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns. In the event that examination of the pattern by the qualified individual indicates that the pattern had been misidentified visually, the client shall be contacted. If the pattern is a suspected chrysotile, take a photograph of the diffraction pattern at 0 degrees tilt. If the structure is suspected to be amphibole, the sample may have to be tilted to obtain a simple geometric array of spots.

j. Energy Dispersive X-Ray Analysis (EDXA).

i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm\2\ concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

ii. Can be used alone to confirm chrysotile after the 70 s/mm\2\ concentration has been exceeded.

iii. Can be used alone to confirm all nonasbestos.

iv. Compare spectrum profiles with profiles obtained from **asbestos** standards. The closest match identifies and categorizes the structure.

v. If the EDXA is used for confirmation, record the properly labeled spectrum on a computer disk, or if a hard copy, file with analysis data.

vi. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm\2\ concentration, their identities must be confirmed by EDXA or measurement of a zone axis diffraction pattern to establish that the particles are nonasbestos.

k. Stopping Rules.

i. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.

ii. After having counted 50 asbestiform structures in a minimum of 4 grid openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.

iii. For blank samples, the analysis is always continued until 10 grid openings have been analyzed.

iv. In all other samples the analysis shall be continued until an analytical sensitivity of 0.005 s/cm\3\ is reached.

l. Recording Rules. The count sheet should contain the following information:

i. Field (grid opening): List field number.

ii. Record ``NSD'' if no structures are detected.

iii. Structure information.

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(1) If fibers, bundles, clusters, and/or matrices are found, list them in consecutive numerical order, starting over with each field.

(2) Length. Record length category of **asbestos** fibers examined. Indicate if less than 5 [mu]m or greater than or equal to 5 [mu]m.

(3) Structure Type. Positive identification of **asbestos** fibers is required by the method. At least one diffraction pattern of each fiber type from every five samples must be recorded and compared with a standard diffraction pattern. For each **asbestos** fiber reported, both a morphological descriptor and an identification descriptor shall be specified on the count sheet.

(4) Fibers classified as chrysotile must be identified by diffraction and/or X-ray analysis and recorded on the count sheet. X-ray analysis alone can be used as sole identification only after 70s/mm\2\ have been exceeded for a particular sample.

(5) Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used as sole identification only after 70s/mm\2\ have been exceeded for a particular sample.)

(6) If a diffraction pattern was recorded on film, the micrograph number must be indicated on the count sheet.

(7) If an electron diffraction was attempted and an appropriate spectra is not observed, N should be recorded on the count sheet.

(8) If an X-ray analysis is attempted but not observed, N should be recorded on the count sheet.

(9) If an X-ray analysis spectrum is stored, the file and disk number must be recorded on the count sheet.

m. Classification Rules.

i. Fiber. A structure having a minimum length greater than or equal to 0.5 [mu]m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. Bundle. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. Cluster. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

iv. Matrix. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

v. NSD. Record NSD when no structures are detected in the field.

n. After all necessary analyses of a particle structure have been completed, return the goniometer stage to 0 degrees, and return the structure to its original location by recall of the original location.

o. Continue scanning until all the structures are identified, classified and sized in the field.

p. Select additional fields (grid openings) at low magnification; scan at a chosen magnification (15,000 to 20,000X screen magnification); and analyze until the stopping rule becomes applicable.

q. Carefully record all data as they are being collected, and check for accuracy.

r. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid hold. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

H. Sample Analytical Sequence

1. Carry out visual inspection of work site prior to air monitoring.

2. Collect a minimum of five air samples inside the work site and five samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

3. Analyze the abatement area samples according to this protocol. The analysis must meet the 0.005 s/cm\3\ analytical sensitivity.

4. Remaining steps in the analytical sequence are contained in Unit IV. of this Appendix.

I. Reporting

The following information must be reported to the client. See the following Table II:

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[GRAPHIC] [TIFF OMITTED] TC01AP92.014

1. Concentration in structures per square millimeter and structures per cubic centimeter.
2. Analytical sensitivity used for the analysis.
3. Number of **asbestos** structures.
4. Area analyzed.
5. Volume of air samples (which was initially provided by client).
6. Average grid size opening.
7. Number of grids analyzed.
8. Copy of the count sheet must be included with the report.

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9. Signature of laboratory official to indicate that the laboratory met specifications of the AHERA method.
10. Report form must contain official laboratory identification (e.g., letterhead).
11. Type of **asbestos**.

J. Calibration Methodology

Note: Appropriate implementation of the method requires a person knowledgeable in electron diffraction and mineral identification by ED and EDXA. Those inexperienced laboratories wishing to develop capabilities may acquire necessary knowledge through analysis of appropriate standards and by following detailed methods as described in References 8 and 10 of Unit III.L.

1. Equipment Calibration. In this method, calibration is required for the air-sampling equipment and the transmission electron microscope (TEM).

a. TEM Magnification. The magnification at the fluorescent screen of the TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica. A logbook must be maintained, and the dates of calibration depend on the past history of the particular microscope; no frequency is specified. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all speciments and the cross grating replica must be placed at the eucentric position.

b. Determination of the TEM magnification on the fluorescent screen.

i. Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

ii. Insert a diffraction grating replica (for example a grating containing 2,160 lines/mm) into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 degrees.

iii. Adjust microscope magnification to 10,000X or 20,000X. Measure the distance (mm) between two widely separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).

Note: The more spaces included in the measurement, the more accurate the final calculation. On most microscopes, however, the magnification is substantially constant only within the central 8-10 cm diameter region of the fluorescent screen.

iv. Calculate the true magnification (M) on the fluorescent screen:

$$M=XG/Y$$

where:

X=total distance (mm) between the designated grating lines;
G=calibration constant of the grating replica (lines/mm):
Y=number of grating replica spaces counted along X.

c. Calibration of the EDXA System. Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of **asbestos** can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for **asbestos** abatement work, and in this procedure the calibration is specified in terms of a ``fingerprint'' technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.

d. Calibration of Grid Openings.

i. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass slide and examining it under the PCM. Use a calibrated graticule to measure the average field diameter and use this number to calculate the field area for an average grid opening. Grids are to be randomly selected from batches up to 1,000.

Note: A grid opening is considered as one field.

ii. The mean grid opening area must be measured for the type of specimen grids in use. This can be accomplished on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

e. Determination of Camera Constant and ED Pattern Analysis.

i. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been

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sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film.

ii. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulates. Since the unknown d-spacings of most interest in **asbestos** analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter, D, of the rings times the interplanar spacing, d, of the ring being measured.

K. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne **asbestos** requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

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1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
2. Check all laboratory reagents and supplies for acceptable **asbestos** background levels.
3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks and special testing after cleaning or servicing the room.
4. Prepare multiple grids of each sample.
5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If this average is greater than 53 f/mm \2\ per 10 200-mesh grid openings, check the system for possible sources of contamination.
6. Check for recovery of **asbestos** from cellulose ester filters submitted to plasma asher.
7. Check for **asbestos** carryover in the plasma asher by including a blank alongside the positive control sample.

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8. Perform a systems check on the transmission electron microscope daily.
9. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III of Unit III.K.
10. Ensure qualified operator performance by evaluation of replicate counting, duplicate analysis, and standard sample comparisons as set forth in Table III of Unit III.K.
11. Validate all data entries.
12. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III.
13. Record an electron diffraction pattern of one **asbestos** structure from every five samples that contain **asbestos**. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions.

The outline of quality control procedures presented above is viewed as the minimum required to assure that quality data is produced for clearance testing of an **asbestos** abated area. Additional information may be gained by other control tests. Specifics on those control procedures and options available for environmental testing can be obtained by consulting References 6, 7, and 11 of Unit III.L.

L. References

For additional background information on this method the following references should be consulted.

1. ``Guidelines for Controlling **Asbestos**-Containing Materials in Buildings,'' EPA 560/5-85-024, June 1985.
2. ``Measuring Airborne **Asbestos** Following an Abatement Action,'' USEP/Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
3. Small, John and E. Steel. **Asbestos** Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
6. Method 2A: Direct Measurement of Gas Volume Through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.
7. Burdette, G.J. Health & Safety Exec., Research & Lab. Services Div., London, ``Proposed Analytical Method for Determination of **Asbestos** in Air.''
8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc. ``Standard Operating Procedure for Determination of Airborne **Asbestos** Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters.'' WERL SOP 87-1, March 5, 1987.
9. NIOSH. Method 7402 for **Asbestos** Fibers, December 11, 1986 Draft.
10. Yamate, G., S.C. Agarwall, R.D. Gibbons, IIT Research Institute, ``Methodology for the Measurement of Airborne **Asbestos** by Electron Microscopy.'' Draft report, USEPA Contract 68-02-3266, July 1984.
11. Guidance to the Preparation of Quality Assurance Project Plans. USEPA, Office of Pollution Prevention and Toxics, 1984.

IV. Mandatory Interpretation of Transmission Electron Microscopy Results to Determine Completion of Response Actions

A. Introduction

A response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne **asbestos** concentration inside the abatement area is no higher than concentrations at locations outside the abatement area. ``Outside'' means outside the abatement area, but not necessarily outside the building. EPA reasons that an **asbestos** removal contractor cannot be expected to clean an abatement area to an airborne **asbestos** concentration that is lower than the concentration of air entering the abatement area from outdoors or from other parts of the building. After the abatement area has passed a thorough visual inspection, and before the outer containment barrier is removed, a minimum of five air samples inside the abatement area and a minimum of five air samples outside the abatement area must be collected. Hence, the response action is determined to be completed when the average airborne **asbestos** concentration measured inside the abatement area is not statistically different from the average airborne **asbestos** concentration measured outside the abatement area.

The inside and outside concentrations are compared by the Z-test, a statistical test that takes into account the variability in the measurement process. A minimum of five samples inside the abatement area and five samples outside the abatement area are required to control the false negative error rate, i.e., the probability of declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

When volumes greater than or equal to 1,199 L for a 25 mm filter and 2,799 L for a 37 mm filter have been collected and the average number of **asbestos** structures on samples inside the abatement area is no greater than 70 s/mm \2\ of filter, the response action

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may be considered complete without comparing the inside samples to the outside samples. EPA is permitting this initial screening test to save analysis costs in situations where the airborne **asbestos** concentration is sufficiently low so that it cannot be distinguished from the filter contamination/background level (fibers deposited on the filter that are unrelated to the air being sampled). The screening test cannot be used when volumes of less than 1,199 L for 25 mm filter or 2,799 L for a 37 mm filter are collected because the ability to distinguish levels significantly different from filter background is reduced at low volumes.

The initial screening test is expressed in structures per square millimeter of filter because filter background levels come from sources other than the air being sampled and cannot be meaningfully expressed as a concentration per cubic centimeter of air. The value of 70 s/mm\2\ is based on the experience of the panel of microscopists who consider one structure in 10 grid openings (each grid opening with an area of 0.0057 mm\2\) to be comparable with contamination/background levels of blank filters. The decision is based, in part, on Poisson statistics which indicate that four structures must be counted on a filter before the fiber count is statistically distinguishable from the count for one structure. As more information on the performance of the method is collected, this criterion may be modified. Since different combinations of the number and size of grid openings are permitted under the TEM protocol, the criterion is expressed in structures per square millimeter of filter to be consistent across all combinations. Four structures per 10 grid openings corresponds to approximately 70 s/mm\2\.

B. Sample Collection and Analysis

1. A minimum of 13 samples is required: five samples collected inside the abatement area, five samples collected outside the abatement area, two field blanks, and one sealed blank.

2. Sampling and TEM analysis must be done according to either the mandatory or nonmandatory protocols in Appendix A. At least 0.057 mm\2\ of filter must be examined on blank filters.

C. Interpretation of Results

1. The response action shall be considered complete if either:

a. Each sample collected inside the abatement area consists of at least 1,199 L of air for a 25 mm filter, or 2,799 L of air for a 37 mm filter, and the arithmetic mean of their **asbestos** structure concentrations per square millimeter of filter is less than or equal to 70 s/mm\2\; or

b. The three blank samples have an arithmetic mean of the **asbestos** structure concentration on the blank filters that is less than or equal to 70 s/mm\2\ and the average airborne **asbestos** concentration measured inside the abatement area is not statistically higher than the average airborne **asbestos** concentration measured outside the abatement area as determined by the Z-test. The Z-test is carried out by calculating
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where Y<INF>I</INF> is the average of the natural logarithms of the inside samples and Y<INF>O</INF> is the average of the natural logarithms of the outside samples, n<INF>I</INF> is the number of inside samples and n<INF>O</INF> is the number of outside samples. The response action is considered complete if Z is less than or equal to 1.65.

Note: When no fibers are counted, the calculated detection limit for that analysis is inserted for the concentration.

2. If the abatement site does not satisfy either (1) or (2) of this Section C, the site must be recleaned and a new set of samples collected.

D. Sequence for Analyzing Samples

It is possible to determine completion of the response action without analyzing all samples. Also, at any point in the process, a decision may be made to terminate the analysis of existing samples, reclean the abatement site, and collect a new set of samples. The following sequence is outlined to minimize the number of analyses needed to reach a decision.

1. Analyze the inside samples.

2. If at least 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for each inside sample and the arithmetic mean concentration of structures per square millimeter of filter is less than or equal to 70 s/mm\2\, the response action is complete and no further analysis is needed.

3. If less than 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for any of the inside samples, or the arithmetic mean concentration of structures per square millimeter of filter is greater than 70 s/mm\2\, analyze the three blanks.

4. If the arithmetic mean concentration of structures per square millimeter on the blank filters is greater than 70 s/mm\2\, terminate

the analysis, identify and correct the source of blank contamination, and collect a new set of samples.

5. If the arithmetic mean concentration of structures per square millimeter on the blank filters is less than or equal to 70 s/mm\2\, analyze the outside samples and perform the Z-test.

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6. If the Z-statistic is less than or equal to 1.65, the response action is complete. If the Z-statistic is greater than 1.65, reclean the abatement site and collect a new set of samples.

[52 FR 41857, Oct. 30, 1987]

Appendix B to Subpart E of Part 763 [Reserved]

Appendix C to Subpart E of Part 763--**Asbestos** Model Accreditation Plan

I. **Asbestos** Model Accreditation Plan for States

The **Asbestos** Model Accreditation Plan (MAP) for States has eight components:

(A) Definitions
(B) Initial Training
(C) Examinations
(D) Continuing Education
(E) Qualifications
(F) Recordkeeping Requirements for Training Providers
(G) Deaccreditation
(H) Reciprocity

A. Definitions

For purposes of Appendix C:

1. ``Friable **asbestos**-containing material (ACM)'' means any material containing more than one percent **asbestos** which has been applied on ceilings, walls, structural members, piping, duct work, or any other part of a building, which when dry, may be crumbled, pulverized, or reduced to powder by hand pressure. The term includes non-friable **asbestos**-containing material after such previously non-friable material becomes damaged to the extent that when dry it may be crumbled, pulverized, or reduced to powder by hand pressure.

2. ``Friable **asbestos**-containing building material (ACBM)'' means any friable ACM that is in or on interior structural members or other parts of a school or public and commercial building.

3. ``Inspection'' means an activity undertaken in a school building, or a public and commercial building, to determine the presence or location, or to assess the condition of, friable or non-friable **asbestos**-containing building material (ACBM) or suspected ACBM, whether by visual or physical examination, or by collecting samples of such material. This term includes reinspections of friable and non-friable known or assumed ACBM which has been previously identified. The term does not include the following:

a. Periodic surveillance of the type described in 40 CFR 763.92(b) solely for the purpose of recording or reporting a change in the condition of known or assumed ACBM;

b. Inspections performed by employees or agents of Federal, State, or local government solely for the purpose of determining compliance with applicable statutes or regulations; or

c. visual inspections of the type described in 40 CFR 763.90(i) solely for the purpose of determining completion of response actions.

4. ``Major fiber release episode'' means any uncontrolled or

unintentional disturbance of ACBM, resulting in a visible emission, which involves the falling or dislodging of more than 3 square or linear feet of friable ACBM.

5. ``Minor fiber release episode'' means any uncontrolled or unintentional disturbance of ACBM, resulting in a visible emission, which involves the falling or dislodging of 3 square or linear feet or less of friable ACBM.

6. ``Public and commercial building'' means the interior space of any building which is not a school building, except that the term does not include any residential apartment building of fewer than 10 units or detached single-family homes. The term includes, but is not limited to: industrial and office buildings, residential apartment buildings and condominiums of 10 or more dwelling units, government-owned buildings, colleges, museums, airports, hospitals, churches, preschools, stores, warehouses and factories. Interior space includes exterior hallways connecting buildings, porticos, and mechanical systems used to condition interior space.

7. ``Response action'' means a method, including removal, encapsulation, enclosure, repair, and operation and maintenance, that protects human health and the environment from friable ACBM.

8. ``Small-scale, short-duration activities (SSSD)'' are tasks such as, but not limited to:

a. Removal of **asbestos**-containing insulation on pipes.

b. Removal of small quantities of **asbestos**-containing insulation on beams or above ceilings.

c. Replacement of an **asbestos**-containing gasket on a valve.

d. Installation or removal of a small section of drywall.

e. Installation of electrical conduits through or proximate to **asbestos**-containing materials.

SSSD can be further defined by the following considerations:

f. Removal of small quantities of ACM only if required in the performance of another maintenance activity not intended as **asbestos** abatement.

g. Removal of **asbestos**-containing thermal system insulation not to exceed amounts greater than those which can be contained in a single glove bag.

h. Minor repairs to damaged thermal system insulation which do not require removal.

i. Repairs to a piece of **asbestos**-containing wallboard.

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j. Repairs, involving encapsulation, enclosure, or removal, to small amounts of friable ACM only if required in the performance of emergency or routine maintenance activity and not intended solely as **asbestos** abatement. Such work may not exceed amounts greater than those which can be contained in a single prefabricated mini-enclosure. Such an enclosure shall conform spatially and geometrically to the localized work area, in order to perform its intended containment function.

B. Initial Training

Training requirements for purposes of accreditation are specified both in terms of required subjects of instruction and in terms of length of training. Each initial training course has a prescribed curriculum and number of days of training. One day of training equals 8 hours, including breaks and lunch. Course instruction must be provided by EPA or State-approved instructors. EPA or State instructor approval shall be based upon a review of the instructor's academic credentials and/or field experience in **asbestos** abatement.

Beyond the initial training requirements, individual States may wish to consider requiring additional days of training for purposes of supplementing hands-on activities or for reviewing relevant state regulations. States also may wish to consider the relative merits of a worker apprenticeship program. Further, they might consider more stringent minimum qualification standards for the approval of training instructors. EPA recommends that the enrollment in any given course be limited to 25 students so that adequate opportunities exist for individual hands-on experience.

States have the option to provide initial training directly or approve other entities to offer training. The following requirements are for the initial training of persons required to have accreditation under TSCA Title II.

Training requirements for each of the five accredited disciplines are outlined below. Persons in each discipline perform a different job function and distinct role. Inspectors identify and assess the condition of ACBM, or suspect ACBM. Management planners use data gathered by inspectors to assess the degree of hazard posed by ACBM in schools to determine the scope and timing of appropriate response actions needed for schools. Project designers determine how **asbestos** abatement work should be conducted. Lastly, workers and contractor/supervisors carry out and oversee abatement work. In addition, a recommended training curriculum is also presented for a sixth discipline, which is not federally-accredited, that of ``Project Monitor.'' Each accredited discipline and training curriculum is separate and distinct from the others. A person seeking accreditation in any of the five accredited MAP disciplines cannot attend two or more courses concurrently, but may attend such courses sequentially.

In several instances, initial training courses for a specific discipline (e.g., workers, inspectors) require hands-on training. For **asbestos** abatement contractor/supervisors and workers, hands-on training should include working with **asbestos**-substitute materials, fitting and using respirators, use of glovebags, donning protective clothing, and constructing a decontamination unit as well as other abatement work activities.

1. Workers

A person must be accredited as a worker to carry out any of the following activities with respect to friable ACBM in a school or public and commercial building: (1) A response action other than a SSSD activity, (2) a maintenance activity that disturbs friable ACBM other than a SSSD activity, or (3) a response action for a major fiber release episode. All persons seeking accreditation as **asbestos** abatement workers shall complete at least a 4-day training course as outlined below. The 4-day worker training course shall include lectures, demonstrations, at least 14 hours of hands-on training, individual respirator fit testing, course review, and an examination. Hands-on training must permit workers to have actual experience performing tasks associated with **asbestos** abatement. A person who is otherwise accredited as a contractor/supervisor may perform in the role of a worker without possessing separate accreditation as a worker.

Because of cultural diversity associated with the **asbestos** workforce, EPA recommends that States adopt specific standards for the approval of foreign language courses for abatement workers. EPA further recommends the use of audio-visual materials to complement lectures, where appropriate.

The training course shall adequately address the following topics:

(a) Physical characteristics of **asbestos**. Identification of **asbestos**, aerodynamic characteristics, typical uses, and physical appearance, and a summary of abatement control options.

(b) Potential health effects related to **asbestos** exposure. The nature of **asbestos**-related diseases; routes of exposure; dose-response relationships and the lack of a safe exposure level; the synergistic effect between cigarette smoking and **asbestos** exposure; the latency periods for **asbestos**-related diseases; a discussion of the relationship of **asbestos** exposure to asbestosis, lung cancer, mesothelioma, and cancers of other organs.

(c) Employee personal protective equipment. Classes and characteristics of respirator

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types; limitations of respirators; proper selection, inspection; donning, use, maintenance, and storage procedures for respirators; methods for field testing of the facepiece-to-face seal (positive and negative-pressure fit checks); qualitative and quantitative fit testing procedures; variability between field and laboratory protection factors that alter respiratory fit (e.g., facial hair); the components of a proper respiratory protection program; selection and use of personal protective clothing; use, storage, and handling of non-disposable clothing; and regulations covering personal protective equipment.

(d) State-of-the-art work practices. Proper work practices for **asbestos** abatement activities, including descriptions of proper construction; maintenance of barriers and decontamination enclosure systems; positioning of warning signs; lock-out of electrical and ventilation systems; proper working techniques for minimizing fiber release; use of wet methods; use of negative pressure exhaust ventilation equipment; use of high-efficiency particulate air (HEPA) vacuums; proper clean-up and disposal procedures; work practices for removal, encapsulation, enclosure, and repair of ACM; emergency procedures for sudden releases; potential exposure situations; transport and disposal procedures; and recommended and prohibited work practices.

(e) Personal hygiene. Entry and exit procedures for the work area; use of showers; avoidance of eating, drinking, smoking, and chewing (gum or tobacco) in the work area; and potential exposures, such as family exposure.

(f) Additional safety hazards. Hazards encountered during abatement activities and how to deal with them, including electrical hazards, heat stress, air contaminants other than **asbestos**, fire and explosion hazards, scaffold and ladder hazards, slips, trips, and falls, and confined spaces.

(g) Medical monitoring. OSHA and EPA Worker Protection Rule requirements for physical examinations, including a pulmonary function test, chest X-rays, and a medical history for each employee.

(h) Air monitoring. Procedures to determine airborne concentrations of **asbestos** fibers, focusing on how personal air sampling is performed and the reasons for it.

(i) Relevant Federal, State, and local regulatory requirements, procedures, and standards. With particular attention directed at relevant EPA, OSHA, and State regulations concerning **asbestos** abatement workers.

(j) Establishment of respiratory protection programs.

(k) Course review. A review of key aspects of the training course.

2. Contractor/Supervisors

A person must be accredited as a contractor/supervisor to supervise any of the following activities with respect to friable ACBM in a school or public and commercial building: (1) A response action other than a

SSSD activity, (2) a maintenance activity that disturbs friable ACBM other than a SSSD activity, or (3) a response action for a major fiber release episode. All persons seeking accreditation as **asbestos** abatement contractor/supervisors shall complete at least a 5-day training course as outlined below. The training course must include lectures, demonstrations, at least 14 hours of hands-on training, individual respirator fit testing, course review, and a written examination. Hands-on training must permit supervisors to have actual experience performing tasks associated with **asbestos** abatement.

EPA recommends the use of audiovisual materials to complement lectures, where appropriate.

Asbestos abatement supervisors include those persons who provide supervision and direction to workers performing response actions. Supervisors may include those individuals with the position title of foreman, working foreman, or leadman pursuant to collective bargaining agreements. At least one supervisor is required to be at the worksite at all times while response actions are being conducted. **Asbestos** workers must have access to accredited supervisors throughout the duration of the project.

The contractor/supervisor training course shall adequately address the following topics:

(a) The physical characteristics of **asbestos** and **asbestos**-containing materials. Identification of **asbestos**, aerodynamic characteristics, typical uses, physical appearance, a review of hazard assessment considerations, and a summary of abatement control options.

(b) Potential health effects related to **asbestos** exposure. The nature of **asbestos**-related diseases; routes of exposure; dose-response relationships and the lack of a safe exposure level; synergism between cigarette smoking and **asbestos** exposure; and latency period for diseases.

(c) Employee personal protective equipment. Classes and characteristics of respirator types; limitations of respirators; proper selection, inspection, donning, use, maintenance, and storage procedures for respirators; methods for field testing of the facepiece-to-face seal (positive and negative-pressure fit checks); qualitative and quantitative fit testing procedures; variability between field and laboratory protection factors that alter respiratory fit (e.g., facial hair); the components of a proper respiratory protection program; selection and use of personal protective clothing; and use, storage,

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and handling of non-disposable clothing; and regulations covering personal protective equipment.

(d) State-of-the-art work practices. Proper work practices for **asbestos** abatement activities, including descriptions of proper construction and maintenance of barriers and decontamination enclosure systems; positioning of warning signs; lock-out of electrical and ventilation systems; proper working techniques for minimizing fiber release; use of wet methods; use of negative pressure exhaust ventilation equipment; use of HEPA vacuums; and proper clean-up and disposal procedures. Work practices for removal, encapsulation, enclosure, and repair of ACM; emergency procedures for unplanned releases; potential exposure situations; transport and disposal procedures; and recommended and prohibited work practices. New abatement-related techniques and methodologies may be discussed.

(e) Personal hygiene. Entry and exit procedures for the work area; use of showers; and avoidance of eating, drinking, smoking, and chewing (gum or tobacco) in the work area. Potential exposures, such as family exposure, shall also be included.

(f) Additional safety hazards. Hazards encountered during abatement activities and how to deal with them, including electrical hazards, heat stress, air contaminants other than **asbestos**, fire and explosion hazards, scaffold and ladder hazards, slips, trips, and falls, and confined spaces.

(g) Medical monitoring. OSHA and EPA Worker Protection Rule requirements for physical examinations, including a pulmonary function test, chest X-rays and a medical history for each employee.

(h) Air monitoring. Procedures to determine airborne concentrations of **asbestos** fibers, including descriptions of aggressive air sampling, sampling equipment and methods, reasons for air monitoring, types of samples and interpretation of results.

EPA recommends that transmission electron microscopy (TEM) be used for analysis of final air clearance samples, and that sample analyses be performed by laboratories accredited by the National Institute of Standards and Technology's (NIST) National Voluntary Laboratory Accreditation Program (NVLAP).

(i) Relevant Federal, State, and local regulatory requirements, procedures, and standards, including:

(i) Requirements of TSCA Title II.

(ii) National Emission Standards for Hazardous Air Pollutants (40 CFR part 61), Subparts A (General Provisions) and M (National Emission Standard for **Asbestos**).

(iii) OSHA standards for permissible exposure to airborne concentrations of **asbestos** fibers and respiratory protection (29 CFR 1910.134).

(iv) OSHA **Asbestos** Construction Standard (29 CFR 1926.58). (v)EPA Worker Protection Rule, (40 CFR part 763, Subpart G).

(j) Respiratory Protection Programs and Medical Monitoring Programs.

(k) Insurance and liability issues. Contractor issues; worker's compensation coverage and exclusions; third-party liabilities and defenses; insurance coverage and exclusions.

(l) Recordkeeping for **asbestos** abatement projects. Records required by Federal, State, and local regulations; records recommended for legal and insurance purposes.

(m) Supervisory techniques for **asbestos** abatement activities. Supervisory practices to enforce and reinforce the required work practices and discourage unsafe work practices.

(n) Contract specifications. Discussions of key elements that are included in contract specifications.

(o) Course review. A review of key aspects of the training course.

3. Inspector

All persons who inspect for ACBM in schools or public and commercial buildings must be accredited. All persons seeking accreditation as an inspector shall complete at least a 3-day training course as outlined below. The course shall include lectures, demonstrations, 4 hours of hands-on training, individual respirator fit-testing, course review, and a written examination.

EPA recommends the use of audiovisual materials to complement lectures, where appropriate. Hands-on training should include conducting a simulated building walk-through inspection and respirator fit testing. The inspector training course shall adequately address the following topics:

(a) Background information on **asbestos**. Identification of **asbestos**, and examples and discussion of the uses and locations of **asbestos** in buildings; physical appearance of **asbestos**.

(b) Potential health effects related to **asbestos** exposure. The nature of **asbestos**-related diseases; routes of exposure; dose-response relationships and the lack of a safe exposure level; the synergistic effect between cigarette smoking and **asbestos** exposure; the latency periods for **asbestos**-related diseases; a discussion of the relationship of **asbestos** exposure to asbestosis, lung cancer, mesothelioma, and cancers of other organs.

(c) Functions/qualifications and role of inspectors. Discussions of prior experience and qualifications for inspectors and management planners; discussions of the functions of an accredited inspector as compared to those of an accredited management planner; discussion of inspection process including inventory of ACM and physical assessment.

(d) Legal liabilities and defenses. Responsibilities of the inspector and management

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planner; a discussion of comprehensive general liability policies, claims-made, and occurrence policies, environmental and pollution liability policy clauses; state liability insurance requirements; bonding and the relationship of insurance availability to bond availability.

(e) Understanding building systems. The interrelationship between building systems, including: an overview of common building physical plan layout; heat, ventilation, and air conditioning (HVAC) system types, physical organization, and where **asbestos** is found on HVAC components; building mechanical systems, their types and organization, and where to look for **asbestos** on such systems; inspecting electrical systems, including appropriate safety precautions; reading blueprints and as-built drawings.

(f) Public/employee/building occupant relations. Notifying employee organizations about the inspection; signs to warn building occupants; tact in dealing with occupants and the press; scheduling of inspections to minimize disruptions; and education of building occupants about actions being taken.

(g) Pre-inspection planning and review of previous inspection records. Scheduling the inspection and obtaining access; building record review; identification of probable homogeneous areas from blueprints or as-built drawings; consultation with maintenance or building personnel; review of previous inspection, sampling, and abatement records of a building; the role of the inspector in exclusions for previously performed inspections.

(h) Inspecting for friable and non-friable ACM and assessing the condition of friable ACM. Procedures to follow in conducting visual inspections for friable and non-friable ACM; types of building materials that may contain **asbestos**; touching materials to determine friability; open return air plenums and their importance in HVAC systems; assessing damage, significant damage, potential damage, and potential significant damage; amount of suspected ACM, both in total quantity and as a percentage of the total area; type of damage; accessibility; material's potential for disturbance; known or suspected causes of damage or significant damage; and deterioration as assessment factors.

(i) Bulk sampling/documentation of **asbestos**. Detailed discussion of the ``Simplified Sampling Scheme for Friable Surfacing Materials (EPA 560/5-85-030a October 1985)''; techniques to ensure sampling in a randomly distributed manner for other than friable surfacing materials; sampling of non-friable materials; techniques for bulk sampling; inspector's sampling and repair equipment; patching or repair of damage from sampling; discussion of polarized light microscopy; choosing an accredited laboratory to analyze bulk samples; quality control and quality assurance procedures. EPA's recommendation that all bulk samples collected from school or public and commercial buildings be analyzed by a laboratory accredited under the NVLAP administered by NIST.

(j) Inspector respiratory protection and personal protective equipment. Classes and characteristics of respirator types; limitations of respirators; proper selection, inspection; donning, use, maintenance, and storage procedures for respirators; methods for field testing of the facepiece-to-face seal (positive and negative-pressure fit checks); qualitative and quantitative fit testing procedures; variability between field and laboratory protection factors that alter respiratory fit (e.g., facial hair); the components of a proper respiratory protection program; selection and use of personal protective clothing; use, storage, and handling of non-disposable clothing.

(k) Recordkeeping and writing the inspection report. Labeling of samples and keying sample identification to sampling location; recommendations on sample labeling; detailing of ACM inventory; photographs of selected sampling areas and examples of ACM condition; information required for inclusion in the management plan required for school buildings under TSCA Title II, section 203 (i)(1). EPA recommends that States develop and require the use of standardized forms for recording the results of inspections in schools or public or commercial buildings, and that the use of these forms be incorporated into the curriculum of training conducted for accreditation.

(l) Regulatory review. The following topics should be covered: National Emission Standards for Hazardous Air Pollutants (NESHAP; 40 CFR part 61, Subparts A and M); EPA Worker Protection Rule (40 CFR part 763, Subpart G); OSHA **Asbestos** Construction Standard (29 CFR 1926.58); OSHA respirator requirements (29 CFR 1910.134); the **Asbestos**-Containing Materials in School Rule (40 CFR part 763, Subpart E; applicable State and local regulations, and differences between Federal and State requirements where they apply, and the effects, if any, on public and nonpublic schools or commercial or public buildings.

(m) Field trip. This includes a field exercise, including a walk-through inspection; on-site discussion about information gathering and the determination of sampling locations; on-site practice in physical assessment; classroom discussion of field exercise.

(n) Course review. A review of key aspects of the training course.

4. Management Planner

All persons who prepare management plans for schools must be accredited. All persons seeking accreditation as management planners shall complete a 3-day inspector training course as outlined above and a 2-day

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management planner training course. Possession of current and valid inspector accreditation shall be a prerequisite for admission to the management planner training course. The management planner course shall include lectures, demonstrations, course review, and a written examination.

EPA recommends the use of audiovisual materials to complement lectures, where appropriate.

TSCA Title II does not require accreditation for persons performing the management planner role in public and commercial buildings. Nevertheless, such persons may find this training and accreditation helpful in preparing them to design or administer **asbestos** operations and maintenance programs for public and commercial buildings.

The management planner training course shall adequately address the following topics:

(a) Course overview. The role and responsibilities of the management planner; operations and maintenance programs; setting work priorities; protection of building occupants.

(b) Evaluation/interpretation of survey results. Review of TSCA Title II requirements for inspection and management plans for school buildings as given in section 203(i)(1) of TSCA Title II; interpretation of field data and laboratory results; comparison of field inspector's data sheet with laboratory results and site survey.

(c) Hazard assessment. Amplification of the difference between physical assessment and hazard assessment; the role of the management planner in hazard assessment; explanation of significant damage, damage, potential damage, and potential significant damage; use of a description (or decision tree) code for assessment of ACM; assessment of friable ACM; relationship of accessibility, vibration sources, use of adjoining space, and air plenums and other factors to hazard assessment.

(d) Legal implications. Liability; insurance issues specific to planners; liabilities associated with interim control measures, in-house maintenance, repair, and removal; use of results from previously performed inspections.

(e) Evaluation and selection of control options. Overview of encapsulation, enclosure, interim operations and maintenance, and removal; advantages and disadvantages of each method; response actions described via a decision tree or other appropriate method; work practices for each response action; staging and prioritizing of work in both vacant and occupied buildings; the need for containment barriers and decontamination in response actions.

(f) Role of other professionals. Use of industrial hygienists, engineers, and architects in developing technical specifications for response actions; any requirements that may exist for architect sign-off of plans; team approach to design of high-quality job specifications.

(g) Developing an operations and maintenance (O&M) plan. Purpose of the plan; discussion of applicable EPA guidance documents; what actions should be taken by custodial staff; proper cleaning procedures; steam cleaning and HEPA vacuuming; reducing disturbance of ACM; scheduling O&M for off-hours; rescheduling or canceling renovation in areas with ACM; boiler room maintenance; disposal of ACM; in-house procedures for ACM--bridging and penetrating encapsulants; pipe fittings; metal sleeves; polyvinyl chloride (PVC), canvas, and wet wraps; muslin with straps, fiber mesh cloth; mineral wool, and insulating cement; discussion of employee protection programs and staff training; case study in developing an O&M plan (development, implementation process, and problems that have been experienced).

(h) Regulatory review. Focusing on the OSHA **Asbestos** Construction Standard found at 29 CFR 1926.58; the National Emission Standard for Hazardous Air Pollutants (NESHAP) found at 40 CFR part 61, Subparts A (General Provisions) and M (National Emission Standard for **Asbestos**); EPA Worker Protection Rule found at 40 CFR part 763, Subpart G; TSCA Title II; applicable State regulations.

(i) Recordkeeping for the management planner. Use of field inspector's data sheet along with laboratory results; on-going recordkeeping as a means to track **asbestos** disturbance; procedures for recordkeeping. EPA recommends that States require the use of standardized forms for purposes of management plans and incorporate the use of such forms into the initial training course for management planners.

(j) Assembling and submitting the management plan. Plan requirements for schools in TSCA Title II section 203(i)(1); the management plan as a planning tool.

(k) Financing abatement actions. Economic analysis and cost estimates; development of cost estimates; present costs of abatement versus future operation and maintenance costs; **Asbestos** School Hazard

Abatement Act grants and loans.

(l) Course review. A review of key aspects of the training course.

5. Project Designer

A person must be accredited as a project designer to design any of the following activities with respect to friable ACBM in a school or public and commercial building: (1) A response action other than a SSSD maintenance activity, (2) a maintenance activity that disturbs friable ACBM other than a SSSD maintenance activity, or (3) a response action for a major fiber release episode. All persons seeking accreditation as a project designer shall complete at least a minimum

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3-day training course as outlined below. The project designer course shall include lectures, demonstrations, a field trip, course review and a written examination.

EPA recommends the use of audiovisual materials to complement lectures, where appropriate.

The abatement project designer training course shall adequately address the following topics:

(a) Background information on **asbestos**. Identification of **asbestos**; examples and discussion of the uses and locations of **asbestos** in buildings; physical appearance of **asbestos**.

(b) Potential health effects related to **asbestos** exposure. Nature of **asbestos**-related diseases; routes of exposure; dose-response relationships and the lack of a safe exposure level; the synergistic effect between cigarette smoking and **asbestos** exposure; the latency period of **asbestos**-related diseases; a discussion of the relationship between **asbestos** exposure and asbestosis, lung cancer, mesothelioma, and cancers of other organs.

(c) Overview of abatement construction projects. Abatement as a portion of a renovation project; OSHA requirements for notification of other contractors on a multi-employer site (29 CFR 1926.58).

(d) Safety system design specifications. Design, construction, and maintenance of containment barriers and decontamination enclosure systems; positioning of warning signs; electrical and ventilation system lock-out; proper working techniques for minimizing fiber release; entry and exit procedures for the work area; use of wet methods; proper techniques for initial cleaning; use of negative-pressure exhaust ventilation equipment; use of HEPA vacuums; proper clean-up and disposal of **asbestos**; work practices as they apply to encapsulation, enclosure, and repair; use of glove bags and a demonstration of glove bag use.

(e) Field trip. A visit to an abatement site or other suitable building site, including on-site discussions of abatement design and building walk-through inspection. Include discussion of rationale for the concept of functional spaces during the walk-through.

(f) Employee personal protective equipment. Classes and characteristics of respirator types; limitations of respirators; proper selection, inspection; donning, use, maintenance, and storage procedures for respirators; methods for field testing of the facepiece-to-face seal (positive and negative-pressure fit checks); qualitative and quantitative fit testing procedures; variability between field and laboratory protection factors that alter respiratory fit (e.g., facial hair); the components of a proper respiratory protection program; selection and use of personal protective clothing; use, storage, and handling of non-disposable clothing.

(g) Additional safety hazards. Hazards encountered during abatement activities and how to deal with them, including electrical hazards, heat stress, air contaminants other than **asbestos**, fire, and explosion hazards.

(h) Fiber aerodynamics and control. Aerodynamic characteristics of **asbestos** fibers; importance of proper containment barriers; settling time for **asbestos** fibers; wet methods in abatement; aggressive air monitoring following abatement; aggressive air movement and negative-pressure exhaust ventilation as a clean-up method.

(i) Designing abatement solutions. Discussions of removal, enclosure, and encapsulation methods; **asbestos** waste disposal.

(j) Final clearance process. Discussion of the need for a written sampling rationale for aggressive final air clearance; requirements of a complete visual inspection; and the relationship of the visual inspection to final air clearance.

EPA recommends the use of TEM for analysis of final air clearance samples. These samples should be analyzed by laboratories accredited under the NIST NVLAP.

(k) Budgeting/cost estimating. Development of cost estimates; present costs of abatement versus future operation and maintenance costs; setting priorities for abatement jobs to reduce costs.

(l) Writing abatement specifications. Preparation of and need for a written project design; means and methods specifications versus performance specifications; design of abatement in occupied buildings; modification of guide specifications for a particular building; worker and building occupant health/medical considerations; replacement of ACM with non-**asbestos** substitutes.

(m) Preparing abatement drawings. Significance and need for drawings, use of as-built drawings as base drawings; use of inspection photographs and on-site reports; methods of preparing abatement drawings; diagramming containment barriers; relationship of drawings to design specifications; particular problems related to abatement drawings.

(n) Contract preparation and administration.

(o) Legal/liabilities/defenses. Insurance considerations; bonding; hold-harmless clauses; use of abatement contractor's liability insurance; claims made versus occurrence policies.

(p) Replacement. Replacement of **asbestos** with **asbestos**-free substitutes.

(q) Role of other consultants. Development of technical specification sections by industrial hygienists or engineers; the multi-disciplinary team approach to abatement design.

(r) Occupied buildings. Special design procedures required in occupied buildings; education of occupants; extra monitoring recommendations; staging of work to minimize occupant exposure; scheduling of renovation to minimize exposure.

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(s) Relevant Federal, State, and local regulatory requirements, procedures and standards, including, but not limited to:

(i) Requirements of TSCA Title II.

(ii) National Emission Standards for Hazardous Air Pollutants, (40 CFR part 61) subparts A (General Provisions) and M (National Emission Standard for **Asbestos**).

(iii) OSHA Respirator Standard found at 29 CFR 1910.134.

(iv) EPA Worker Protection Rule found at 40 CFR part 763, subpart G.

(v) OSHA **Asbestos** Construction Standard found at 29 CFR 1926.58.

(vi) OSHA Hazard Communication Standard found at 29 CFR 1926.59.

(t) Course review. A review of key aspects of the training course.

6. Project Monitor

EPA recommends that States adopt training and accreditation requirements for persons seeking to perform work as project monitors. Project monitors observe abatement activities performed by contractors and generally serve as a building owner's representative to ensure that abatement work is completed according to specification and in compliance with all relevant statutes and regulations. They may also perform the vital role of air monitoring for purposes of determining final clearance. EPA recommends that a State seeking to accredit individuals as project monitors consider adopting a minimum 5-day training course covering the topics outlined below. The course outlined below consists of lectures and demonstrations, at least 6 hours of hands-on training, course review, and a written examination. The hands-on training component might be satisfied by having the student simulate participation in or performance of any of the relevant job functions or activities (or by incorporation of the workshop component described in item ``n'' below of this unit).

EPA recommends that the project monitor training course adequately address the following topics:

(a) Roles and responsibilities of the project monitor. Definition and responsibilities of the project monitor, including regulatory/specification compliance monitoring, air monitoring, conducting visual inspections, and final clearance monitoring.

(b) Characteristics of **asbestos** and **asbestos**-containing materials. Typical uses of **asbestos**; physical appearance of **asbestos**; review of **asbestos** abatement and control techniques; presentation of the health effects of **asbestos** exposure, including routes of exposure, dose-response relationships, and latency periods for **asbestos**-related diseases.

(c) Federal **asbestos** regulations. Overview of pertinent EPA regulations, including: NESHAP, 40 CFR part 61, subparts A and M; AHERA, 40 CFR part 763, subpart E; and the EPA Worker Protection Rule, 40 CFR part 763, subpart G. Overview of pertinent OSHA regulations, including: Construction Industry Standard for **Asbestos**, 29 CFR 1926.58; Respirator Standard, 29 CFR 1910.134; and the Hazard Communication Standard, 29 CFR 1926.59. Applicable State and local **asbestos** regulations; regulatory interrelationships.

(d) Understanding building construction and building systems. Building construction basics, building physical plan layout; understanding building systems (HVAC, electrical, etc.); layout and organization, where **asbestos** is likely to be found on building systems; renovations and the effect of **asbestos** abatement on building systems.

(e) **Asbestos** abatement contracts, specifications, and drawings. Basic provisions of the contract; relationships between principle parties, establishing chain of command; types of specifications, including means and methods, performance, and proprietary and nonproprietary; reading and interpreting records and abatement drawings; discussion of change orders; common enforcement responsibilities and authority of project monitor.

(f) Response actions and abatement practices. Pre-work inspections; pre-work considerations, precleaning of the work area, removal of furniture, fixtures, and equipment; shutdown/modification of building systems; construction and maintenance of containment barriers, proper demarcation of work areas; work area entry/exit, hygiene practices; determining the effectiveness of air filtration equipment; techniques for minimizing fiber release, wet methods, continuous cleaning; abatement methods other than removal; abatement area clean-up procedures; waste transport and disposal procedures; contingency planning for emergency response.

(g) **Asbestos** abatement equipment. Typical equipment found on an abatement project; air filtration devices, vacuum systems, negative pressure differential monitoring; HEPA filtration units, theory of filtration, design/construction of HEPA filtration units, qualitative and quantitative performance of HEPA filtration units, sizing the ventilation requirements, location of HEPA filtration units, qualitative and quantitative tests of containment barrier integrity; best available technology.

(h) Personal protective equipment. Proper selection of respiratory protection; classes and characteristics of respirator types, limitations of respirators; proper use of other safety equipment, protective clothing selection, use, and proper handling, hard/bump hats, safety shoes; breathing air systems, high pressure v. low pressure, testing for Grade D air, determining proper backup air volumes.

(i) Air monitoring strategies. Sampling equipment, sampling pumps (low v. high volume), flow regulating devices (critical and

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limiting orifices), use of fibrous aerosol monitors on abatement projects; sampling media, types of filters, types of cassettes, filter orientation, storage and shipment of filters; calibration techniques, primary calibration standards, secondary calibration standards, temperature/pressure effects, frequency of calibration, recordkeeping and field work documentation, calculations; air sample analysis, techniques available and limitations of AHERA on their use, transmission electron microscopy (background to sample preparation and analysis, air sample conditions which prohibit analysis, EPA's recommended technique for analysis of final air clearance samples), phase contrast microscopy (background to sample preparation, and AHERA's limits on the use of phase contrast microscopy), what each technique measures; analytical methodologies, AHERA TEM protocol, NIOSH 7400, OSHA reference method (non clearance), EPA recommendation for clearance (TEM); sampling strategies for clearance monitoring, types of air samples (personal breathing zone v. fixed-station area) sampling location and objectives (pre-abatement, during abatement, and clearance monitoring), number of samples to be collected, minimum and maximum air volumes, clearance monitoring (post-visual-inspection) (number of samples required, selection of sampling locations, period of sampling, aggressive sampling, interpretations of sampling results, calculations), quality assurance; special sampling problems, crawl spaces, acceptable samples for laboratory analysis, sampling in occupied buildings (barrier monitoring).

(j) Safety and health issues other than **asbestos**. Confined-space entry, electrical hazards, fire and explosion concerns, ladders and scaffolding, heat stress, air contaminants other than **asbestos**, fall hazards, hazardous materials on abatement projects.

(k) Conducting visual inspections. Inspections during abatement, visual inspections using the ASTM E1368 document; conducting inspections for completeness of removal; discussion of ``how clean is clean?''

(l) Legal responsibilities and liabilities of project monitors. Specification enforcement capabilities; regulatory enforcement; licensing; powers delegated to project monitors through contract documents.

(m) Recordkeeping and report writing. Developing project logs/daily logs (what should be included, who sees them); final report preparation; recordkeeping under Federal regulations.

(n) Workshops (6 hours spread over 3 days). Contracts, specifications, and drawings: This workshop could consist of each participant being issued a set of contracts, specifications, and drawings and then being asked to answer questions and make recommendations to a project architect, engineer or to the building owner based on given conditions and these documents.

Air monitoring strategies/**asbestos** abatement equipment: This workshop could consist of simulated abatement sites for which sampling strategies would have to be developed (i.e., occupied buildings, industrial situations). Through demonstrations and exhibition, the project monitor may also be able to gain a better understanding of the function of various pieces of equipment used on abatement projects (air filtration units, water filtration units, negative pressure monitoring devices, sampling pump calibration devices, etc.).

Conducting visual inspections: This workshop could consist, ideally, of an interactive video in which a participant is ``taken through'' a work area and asked to make notes of what is seen. A series of questions will be asked which are designed to stimulate a person's recall of the area. This workshop could consist of a series of two or three videos with different site conditions and different degrees of cleanliness.

C. Examinations

1. Each State shall administer a closed book examination or designate other entities such as State-approved providers of training courses to administer the closed-book examination to persons seeking accreditation who have completed an initial training course. Demonstration testing may also be included as part of the examination. A person seeking initial accreditation in a specific discipline must pass the examination for that discipline in order to receive accreditation. For example, a person seeking accreditation as an abatement project designer must pass the State's examination for abatement project designer.

States may develop their own examinations, have providers of training courses develop examinations, or use standardized examinations developed for purposes of accreditation under TSCA Title II. In addition, States may supplement standardized examinations with questions about State regulations. States may obtain commercially developed standardized examinations, develop standardized examinations independently, or do so in cooperation with other States, or with commercial or non-profit providers on a regional or national basis. EPA recommends the use of standardized, scientifically-validated testing instruments, which may be beneficial in terms of both promoting competency and in fostering accreditation reciprocity between States.

Each examination shall adequately cover the topics included in the training course for that discipline. Each person who completes a

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training course, passes the required examination, and fulfills whatever other requirements the State imposes must receive an accreditation certificate in a specific discipline. Whether a State directly issues accreditation certificates, or authorizes training providers to issue accreditation certificates, each certificate issued to an accredited person must contain the following minimum information:

a. A unique certificate number
b. Name of accredited person
c. Discipline of the training course completed.
d. Dates of the training course.
e. Date of the examination.
f. An expiration date of 1 year after the date upon which the person successfully completed the course and examination.
g. The name, address, and telephone number of the training provider that issued the certificate.

h. A statement that the person receiving the certificate has completed the requisite training for **asbestos** accreditation under TSCA Title II.

States or training providers who reaccredit persons based upon completion of required refresher training must also provide accreditation certificates with all of the above information, except the examination date may be omitted if a State does not require a refresher examination for reaccreditation.

Where a State licenses accredited persons but has authorized training providers to issue accreditation certificates, the State may issue licenses in the form of photo-identification cards. Where this applies, EPA recommends that the State licenses should include all of the same information required for the accreditation certificates. A State may also choose to issue photo-identification cards in addition to the required accreditation certificates.

Accredited persons must have their initial and current accreditation certificates at the location where they are conducting work.

2. The following are the requirements for examination in each discipline:

a. Worker:
i. 50 multiple-choice questions
ii. Passing score: 70 percent correct
b. Contractor/Supervisor:
i. 100 multiple-choice questions
ii. Passing score: 70 percent correct
c. Inspector:
i. 50 Multiple-choice questions
ii. Passing score: 70 percent correct
d. Management Planner:
i. 50 Multiple-choice questions
ii. Passing score: 70 percent correct
e. Project Designer:
i. 100 multiple-choice questions
ii. Passing score: 70 percent correct

D. Continuing Education

For all disciplines, a State's accreditation program shall include annual refresher training as a requirement for reaccreditation as indicated below:

1. Workers: One full day of refresher training.
2. Contractor/Supervisors: One full day of refresher training.
3. Inspectors: One half-day of refresher training.
4. Management Planners: One half-day of inspector refresher training and one half-day of refresher training for management planners.
5. Project Designers: One full day of refresher training.

The refresher courses shall be specific to each discipline. Refresher courses shall be conducted as separate and distinct courses and not combined with any other training during the period of the refresher course. For each discipline, the refresher course shall review and discuss changes in Federal, State, and local regulations, developments in state-of-the-art procedures, and a review of key aspects of the initial training course as determined by the State. After completing the annual refresher course, persons shall have their accreditation extended for an additional year from the date of the refresher course. A State may consider requiring persons to pass reaccreditation examinations at specific intervals (for example, every 3 years).

EPA recommends that States formally establish a 12-month grace period to enable formerly accredited persons with expired certificates to complete refresher training and have their accreditation status reinstated without having to re-take the initial training course.

E. Qualifications

In addition to requiring training and an examination, a State may require candidates for accreditation to meet other qualification and/or experience standards that the State considers appropriate for some or all disciplines. States may choose to consider requiring qualifications similar to the examples outlined below for inspectors, management planners and project designers. States may modify these examples as appropriate. In addition, States may want to include some requirements based on experience in performing a task directly as a part of a job or in an apprenticeship role. They may also wish to consider additional criteria for the approval of training course instructors beyond those prescribed by EPA.

1. Inspectors: Qualifications - possess a high school diploma. States may want to require an Associate's Degree in specific fields (e.g., environmental or physical sciences).

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2. Management Planners: Qualifications - Registered architect, engineer, or certified industrial hygienist or related scientific field.
3. Project Designers: Qualifications - registered architect, engineer, or certified industrial hygienist.
4. **Asbestos** Training Course Instructor: Qualifications - academic credentials and/or field experience in **asbestos** abatement.

EPA recommends that States prescribe minimum qualification standards for training instructors employed by training providers.

F. Recordkeeping Requirements for Training Providers

All approved providers of accredited **asbestos** training courses must comply with the following minimum recordkeeping requirements.

1. Training course materials. A training provider must retain copies of all instructional materials used in the delivery of the classroom training such as student manuals, instructor notebooks and handouts.
2. Instructor qualifications. A training provider must retain copies of all instructors' resumes, and the documents approving each instructor issued by either EPA or a State. Instructors must be approved by either EPA or a State before teaching courses for accreditation purposes. A training provider must notify EPA or the State, as appropriate, in advance whenever it changes course instructors. Records must accurately identify the instructors that taught each particular course for each date that a course is offered.
3. Examinations. A training provider must document that each person who receives an accreditation certificate for an initial training course has achieved a passing score on the examination. These records must clearly indicate the date upon which the exam was administered, the training course and discipline for which the exam was given, the name of the person who proctored the exam, a copy of the exam, and the name and test score of each person taking the exam. The topic and dates of the training course must correspond to those listed on that person's accreditation certificate. States may choose to apply these same requirements to examinations for refresher training courses.
4. Accreditation certificates. The training providers or States, whichever issues the accreditation certificate, shall maintain records that document the names of all persons who have been awarded certificates, their certificate numbers, the disciplines for which

accreditation was conferred, training and expiration dates, and the training location. The training provider or State shall maintain the records in a manner that allows verification by telephone of the required information.

5. Verification of certificate information. EPA recommends that training providers of refresher training courses confirm that their students possess valid accreditation before granting course admission. EPA further recommends that training providers offering the initial management planner training course verify that students have met the prerequisite of possessing valid inspector accreditation at the time of course admission.

6. Records retention and access. (a) The training provider shall maintain all required records for a minimum of 3 years. The training provider, however, may find it advantageous to retain these records for a longer period of time.

(b) The training provider must allow reasonable access to all of the records required by the MAP, and to any other records which may be required by States for the approval of **asbestos** training providers or the accreditation of **asbestos** training courses, to both EPA and to State Agencies, on request. EPA encourages training providers to make this information equally accessible to the general public.

(c) If a training provider ceases to conduct training, the training provider shall notify the approving government body (EPA or the State) and give it the opportunity to take possession of that providers **asbestos** training records.

G. Deaccreditation

1. States must establish criteria and procedures for deaccrediting persons accredited as workers, contractor/supervisors, inspectors, management planners, and project designers. States must follow their own administrative procedures in pursuing deaccreditation actions. At a minimum, the criteria shall include:

(a) Performing work requiring accreditation at a job site without being in physical possession of initial and current accreditation certificates;

(b) Permitting the duplication or use of one's own accreditation certificate by another;

(c) Performing work for which accreditation has not been received; or

(d) Obtaining accreditation from a training provider that does not have approval to offer training for the particular discipline from either EPA or from a State that has a contractor accreditation plan at least as stringent as the EPA MAP.

EPA may directly pursue deaccreditation actions without reliance on State deaccreditation or enforcement authority or actions. In addition to the above-listed situations, the Administrator may suspend or revoke the accreditation of persons who have been subject to a final order imposing a civil penalty or convicted under section 16 of TSCA, 15 U.S.C. 2615 or 2647, for violations of 40 CFR part 763, or section 113 of the Clean

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Air Act, 42 U.S.C. 7413, for violations of 40 CFR part 61, subpart M.

2. Any person who performs **asbestos** work requiring accreditation under section 206(a) of TSCA, 15 U.S.C. 2646(a), without such accreditation is in violation of TSCA. The following persons are not accredited for purposes of section 206(a) of TSCA:

(a) Any person who obtains accreditation through fraudulent representation of training or examination documents;

(b) Any person who obtains training documentation through fraudulent means;

(c) Any person who gains admission to and completes refresher training through fraudulent representation of initial or previous refresher training documentation; or

(d) Any person who obtains accreditation through fraudulent representation of accreditation requirements such as education, training, professional registration, or experience.

H. Reciprocity

EPA recommends that each State establish reciprocal arrangements with other States that have established accreditation programs that meet or exceed the requirements of the MAP. Such arrangements might address cooperation in licensing determinations, the review and approval of training programs and/or instructors, candidate testing and exam administration, curriculum development, policy formulation, compliance monitoring, and the exchange of information and data. The benefits to be derived from these arrangements include a potential cost-savings from the reduction of duplicative activity and the attainment of a more professional accredited workforce as States are able to refine and improve the effectiveness of their programs based upon the experience and methods of other States.

II. EPA Approval Process for State Accreditation Programs

A. States may seek approval for a single discipline or all disciplines as specified in the MAP. For example, a State that currently only requires worker accreditation may receive EPA approval for that discipline alone. EPA encourages States that currently do not have accreditation requirements for all disciplines required under section 206(b)(2) of TSCA, 15 U.S.C. 2646(b)(2), to seek EPA approval for those disciplines the State does accredit. As States establish accreditation requirements for the remaining disciplines, the requested information outlined below should be submitted to EPA as soon as possible. Any State that had an accreditation program approved by EPA under an earlier version of the MAP may follow the same procedures to obtain EPA approval of their accreditation program under this MAP.

B. Partial approval of a State Program for the accreditation of one or more disciplines does not mean that the State is in full compliance with TSCA where the deadline for that State to have adopted a State Plan no less stringent than the MAP has already passed. State Programs which are at least as stringent as the MAP for one or more of the accredited disciplines may, however, accredit persons in those disciplines only.

C. States seeking EPA approval or reapproval of accreditation programs shall submit the following information to the Regional **Asbestos** Coordinator at their EPA Regional office:

1. A copy of the legislation establishing or upgrading the State's accreditation program (if applicable).

2. A copy of the State's accreditation regulations or revised regulations.

3. A letter to the Regional **Asbestos** Coordinator that clearly indicates how the State meets the program requirements of this MAP. Addresses for each of the Regional **Asbestos** Coordinators are shown below:

EPA, Region I, (ATC-111) **Asbestos** Coordinator, JFK Federal Bldg., Boston, MA 02203-2211, (617) 565-3836.

EPA, Region II, (MS-500), **Asbestos** Coordinator, 2890 Woodbridge Ave., Edison, NJ 08837-3679, (908) 321-6671.

EPA, Region III, (3AT-33), **Asbestos** Coordinator, 841 Chestnut Bldg., Philadelphia, PA 19107, (215) 597-3160.
EPA, Region IV, **Asbestos** Coordinator, 345 Courtland St., N.E., Atlanta, GA 30365, (404) 347-5014.
EPA, Region V, (SP-14J), **Asbestos** Coordinator, 77 W. Jackson Blvd., Chicago, IL 60604-3590, (312) 886-6003.
EPA, Region VI, (6T-PT), **Asbestos** Coordinator, 1445 Ross Ave. Dallas, TX 75202-2744, (214) 655-7244.
EPA, Region VII, (ARTX/ASBS), **Asbestos** Coordinator, 726 Minnesota Ave., Kansas City, KS 66101, (913) 551-7020.
EPA, Region VIII, (8AT-TS), **Asbestos** Coordinator, 1 Denver Place, Suite 500 999 - 18th St., Denver, CO 80202-2405, (303) 293-1442.
EPA, Region IX, (A-4-4), **Asbestos** Coordinator, 75 Hawthorne St., San Francisco, CA 94105, (415) 744-1128.
EPA, Region X, (AT-083), **Asbestos** Coordinator, 1200 Sixth Ave., Seattle, WA 98101, (206) 553-4762.

EPA maintains a listing of all those States that have applied for and received EPA approval for having accreditation requirements that are at least as stringent as the MAP for one or more disciplines. Any training courses approved by an EPA-approved State Program are considered to be EPA-approved for purposes of accreditation.

III. Approval of Training Courses

Individuals or groups wishing to sponsor training courses for disciplines required to

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be accredited under section 206(b)(1)(A) of TSCA, 15 U.S.C. 2646(b)(1)(A), may apply for approval from States that have accreditation program requirements that are at least as stringent as this MAP. For a course to receive approval, it must meet the requirements for the course as outlined in this MAP, and any other requirements imposed by the State from which approval is being sought. Courses that have been approved by a State with an accreditation program at least as stringent as this MAP are approved under section 206(a) of TSCA, 15 U.S.C. 2646(a), for that particular State, and also for any other State that does not have an accreditation program as stringent as this MAP.

A. Initial Training Course Approval

A training provider must submit the following minimum information to a State as part of its application for the approval of each training course:

1. The course provider's name, address, and telephone number.
2. A list of any other States that currently approve the training course.
3. The course curriculum.
4. A letter from the provider of the training course that clearly indicates how the course meets the MAP requirements for:
    a. Length of training in days.
    b. Amount and type of hands-on training.
    c. Examination (length, format, and passing score).
    d. Topics covered in the course.
5. A copy of all course materials (student manuals, instructor notebooks, handouts, etc.).
6. A detailed statement about the development of the examination used in the course.

7. Names and qualifications of all course instructors. Instructors must have academic and/or field experience in **asbestos** abatement.

8. A description of and an example of the numbered certificates issued to students who attend the course and pass the examination.

B. Refresher Training Course Approval

The following minimum information is required for approval of refresher training courses by States:

1. The length of training in half-days or days.
2. The topics covered in the course.
3. A copy of all course materials (student manuals, instructor notebooks, handouts, etc.).
4. The names and qualifications of all course instructors. Instructors must have academic and/or field experience in **asbestos** abatement.
5. A description of and an example of the numbered certificates issued to students who complete the refresher course and pass the examination, if required.

C. Withdrawal of Training Course Approval

States must establish criteria and procedures for suspending or withdrawing approval from accredited training programs. States should follow their own administrative procedures in pursuing actions for suspension or withdrawal of approval of training programs. At a minimum, the criteria shall include:

(1) Misrepresentation of the extent of a training course's approval by a State or EPA;

(2) Failure to submit required information or notifications in a timely manner;

(3) Failure to maintain requisite records;

(4) Falsification of accreditation records, instructor qualifications, or other accreditation information; or

(5) Failure to adhere to the training standards and requirements of the EPA MAP or State Accreditation Program, as appropriate.

In addition to the criteria listed above, EPA may also suspend or withdraw a training course's approval where an approved training course instructor, or other person with supervisory authority over the delivery of training has been found in violation of other **asbestos** regulations administered by EPA. An administrative or judicial finding of violation, or execution of a consent agreement and order under 40 CFR 22.18, constitutes evidence of a failure to comply with relevant statutes or regulations. States may wish to adopt this criterion modified to include their own **asbestos** statutes or regulations. EPA may also suspend or withdraw approval of training programs where a training provider has submitted false information as a part of the self-certification required under Unit V.B. of the revised MAP.

Training course providers shall permit representatives of EPA or the State which approved their training courses to attend, evaluate, and monitor any training course without charge. EPA or State compliance inspection staff are not required to give advance notice of their inspections. EPA may suspend or withdraw State or EPA approval of a training course based upon the criteria specified in this Unit III.C.

IV. EPA Procedures for Suspension or Revocation of Accreditation or Training Course Approval.

A. If the Administrator decides to suspend or revoke the accreditation of any person or suspend or withdraw the approval of a training course, the Administrator will notify the affected entity of the following:

1. The grounds upon which the suspension, revocation, or withdrawal is based.

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2. The time period during which the suspension, revocation, or withdrawal is effective, whether permanent or otherwise.

3. The conditions, if any, under which the affected entity may receive accreditation or approval in the future.

4. Any additional conditions which the Administrator may impose.

5. The opportunity to request a hearing prior to final Agency action to suspend or revoke accreditation or suspend or withdraw approval.

B. If a hearing is requested by the accredited person or training course provider pursuant to the preceding paragraph, the Administrator will:

1. Notify the affected entity of those assertions of law and fact upon which the action to suspend, revoke, or withdraw is based.

2. Provide the affected entity an opportunity to offer written statements of facts, explanations, comments, and arguments relevant to the proposed action.

3. Provide the affected entity such other procedural opportunities as the Administrator may deem appropriate to ensure a fair and impartial hearing.

4. Appoint an EPA attorney as Presiding Officer to conduct the hearing. No person shall serve as Presiding Officer if he or she has had any prior connection with the specific case.

C. The Presiding Officer appointed pursuant to the preceding paragraph shall:

1. Conduct a fair, orderly, and impartial hearing, without unnecessary delay.

2. Consider all relevant evidence, explanation, comment, and argument submitted pursuant to the preceding paragraph.

3. Promptly notify the affected entity of his or her decision and order. Such an order is a final Agency action.

D. If the Administrator determines that the public health, interest, or welfare warrants immediate action to suspend the accreditation of any person or the approval of any training course provider, the Administrator will:

1. Notify the affected entity of the grounds upon which the emergency suspension is based;

2. Notify the affected entity of the time period during which the emergency suspension is effective.

3. Notify the affected entity of the Administrator's intent to suspend or revoke accreditation or suspend or withdraw training course approval, as appropriate, in accordance with Unit IV.A. above. If such suspension, revocation, or withdrawal notice has not previously been issued, it will be issued at the same time the emergency suspension notice is issued.

E. Any notice, decision, or order issued by the Administrator under this section, and any documents filed by an accredited person or approved training course provider in a hearing under this section, shall be available to the public except as otherwise provided by section 14 of TSCA or by 40 CFR part 2. Any such hearing at which oral testimony is presented shall be open to the public, except that the Presiding Officer may exclude the public to the extent necessary to allow presentation of information which may be entitled to confidential treatment under section 14 of TSCA or 40 CFR part 2.

V. Implementation Schedule

The various requirements of this MAP become effective in accordance with the following schedules:

A. Requirements applicable to State Programs

1. Each State shall adopt an accreditation plan that is at least as stringent as this MAP within 180 days after the commencement of the first regular session of the legislature of the State that is convened on or after April 4, 1994.

2. If a State has adopted an accreditation plan at least as stringent as this MAP as of April 4, 1994, the State may continue to:

a. Conduct TSCA training pursuant to this MAP.

b. Approve training course providers to conduct training and to issue accreditation that satisfies the requirements for TSCA accreditation under this MAP.

c. Issue accreditation that satisfies the requirements for TSCA accreditation under this MAP.

3. A State that had complied with an earlier version of the MAP, but has not adopted an accreditation plan at least as stringent as this MAP by April 4, 1994, may:

a. Conduct TSCA training which remains in compliance with the requirements of Unit V.B. of this MAP. After such training has been self-certified in accordance with Unit V.B. of this MAP, the State may issue accreditation that satisfies the requirement for TSCA accreditation under this MAP.

b. Sustain its approval for any training course providers to conduct training and issue TSCA accreditation that the State had approved before April 4, 1994, and that remain in compliance with Unit V.B. of this MAP.

c. Issue accreditation pursuant to an earlier version of the MAP that provisionally satisfies the requirement for TSCA accreditation until October 4, 1994.

Such a State may not approve new TSCA training course providers to conduct training or to issue TSCA accreditation that satisfies the requirements of this MAP until the State adopts an accreditation plan that is at least as stringent as this MAP.

4. A State that had complied with an earlier version of the MAP, but fails to adopt a

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plan as stringent as this MAP by the deadline established in Unit V.A.1., is subject to the following after that deadline date:

a. The State loses any status it may have held as an EPA-approved State for accreditation purposes under section 206 of TSCA, 15 U.S.C. 2646.

b. All training course providers approved by the State lose State approval to conduct training and issue accreditation that satisfies the requirements for TSCA accreditation under this MAP.

c. The State may not:

i. Conduct training for accreditation purposes under section 206 of TSCA, 15 U.S.C. 2646.

ii. Approve training course providers to conduct training or issue accreditation that satisfies the requirements for TSCA accreditation; or

iii. Issue accreditation that satisfies the requirement for TSCA accreditation.

EPA will extend EPA-approval to any training course provider that loses State approval because the State does not comply with the deadline, so long as the provider is in compliance with Unit V.B. of this MAP, and the provider is approved by a State that had complied with an earlier version of the MAP as of the day before the State loses its EPA approval.

5. A State that does not have an accreditation program that satisfies the requirements for TSCA accreditation under either an earlier version of the MAP or this MAP, may not:

a. Conduct training for accreditation purposes under section 206 of TSCA, 15 U.S.C. 2646;

b. Approve training course providers to conduct training or issue accreditation that satisfies the requirements for TSCA accreditation; or

c. Issue accreditation that satisfies the requirement for TSCA accreditation.

B. Requirements applicable to Training Courses and Providers

As of October 4, 1994, an approved training provider must certify to EPA and to any State that has approved the provider for TSCA accreditation, that each of the provider's training courses complies with the requirements of this MAP. The written submission must document in specific detail the changes made to each training course in order to comply with the requirements of this MAP and clearly state that the provider is also in compliance with all other requirements of this MAP, including the new recordkeeping and certificate provisions. Each submission must include the following statement signed by an authorized representative of the training provider: ``Under civil and criminal penalties of law for the making or submission of false or fraudulent statements or representations (18 U.S.C. 1001 and 15 U.S.C. 2615), I certify that the training described in this submission complies with all applicable requirements of Title II of TSCA, 40 CFR part 763, Appendix C to Subpart E, as revised, and any other applicable Federal, state, or local requirements.'' A consolidated self-certification submission from each training provider that addresses all of its approved training courses is permissible and encouraged.

The self-certification must be sent via registered mail, to EPA Headquarters at the following address: Attn. Self-Certification Program, Field Programs Branch, Chemical Management Division (7404), Office of Pollution Prevention and Toxics, Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460. A duplicate copy of the complete submission must also be sent to any States from which approval had been obtained.

The timely receipt of a complete self-certification by EPA and all approving States shall have the effect of extending approval under this MAP to the training courses offered by the submitting provider. If a self-certification is not received by the approving government bodies on or before the due date, the affected training course is not approved under this MAP. Such training providers must then reapply for approval of these training courses pursuant to the procedures outlined in Unit III.

C. Requirements applicable to Accredited Persons.

Persons accredited by a State with an accreditation program no less stringent than an earlier version of the MAP or by an EPA-approved training provider as of April 3, 1994, are accredited in accordance with the requirements of this MAP, and are not required to retake initial training. They must continue to comply with the requirements for annual refresher training in Unit I.D. of the revised MAP.

D. Requirements applicable to Non-Accredited Persons.

In order to perform work requiring accreditation under TSCA Title II, persons who are not accredited by a State with an accreditation program no less stringent than an earlier version of the MAP or by an EPA-approved training provider as of April 3, 1994, must comply with the upgraded training requirements of this MAP by no later than October 4, 1994. Non-accredited persons may obtain initial accreditation on a provisional basis by successfully completing any of the training programs approved under an earlier version of the MAP, and thereby perform work during the first 6 months after this MAP takes effect. However, by October 4, 1994, these persons must have successfully completed an upgraded training program

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that fully complies with the requirements of this MAP in order to continue to perform work requiring accreditation under section 206 of TSCA, 15 U.S.C. 2646.

[59 FR 5251, Feb. 3, 1994, as amended at 60 FR 31922, June 19, 1995]

Appendix D to Subpart E of Part 763--Transport and Disposal of **Asbestos** Waste

For the purposes of this appendix, transport is defined as all activities from receipt of the containerized **asbestos** waste at the generation site until it has been unloaded at the disposal site. Current EPA regulations state that there must be no visible emissions to the outside air during waste transport. However, recognizing the potential hazards and subsequent liabilities associated with exposure, the following additional precautions are recommended.

Recordkeeping. Before accepting wastes, a transporter should determine if the waste is properly wetted and containerized. The transporter should then require a chain-of-custody form signed by the generator. A chain-of-custody form may include the name and address of the generator, the name and address of the pickup site, the estimated quantity of **asbestos** waste, types of containers used, and the destination of the waste. The chain-of-custody form should then be signed over to a disposal site operator to transfer responsibility for the **asbestos** waste. A copy of the form signed by the disposal site operator should be maintained by the transporter as evidence of receipt at the disposal site.

Waste handling. A transporter should ensure that the **asbestos** waste is properly contained in leak-tight containers with appropriate labels, and that the outside surfaces of the containers are not contaminated with **asbestos** debris adhering to the containers. If there is reason to believe that the condition of the **asbestos** waste may allow significant fiber release, the transporter should not accept the waste. Improper containerization of wastes is a violation of the NESHAPs regulation and should be reported to the appropriate EPA Regional **Asbestos** NESHAPs contact below:

Region I

Asbestos NESHAPs Contact, Air Management Division, USEPA, Region I, JFK Federal Building, Boston, MA 02203, (617) 223-3266.

Region II

Asbestos NESHAPs Contact, Air & Waste Management Division, USEPA, Region II, 26 Federal Plaza, New York, NY 10007, (212) 264-6770.

Region III

Asbestos NESHAPs Contact, Air Management Division, USEPA, Region III, 841 Chestnut Street, Philadelphia, PA 19107, (215) 597-9325.

Region IV

Asbestos NESHAPs Contact, Air, Pesticide & Toxic Management, USEPA, Region IV, 345 Courtland Street, NE., Atlanta, GA 30365, (404) 347-4298.

Region V

Asbestos NESHAPs Contact, Air Management Division, USEPA, Region V, 77 West Jackson Boulevard, Chicago, IL 60604, (312) 353-6793.

Region VI

Asbestos NESHAPs Contact, Air & Waste Management Division, USEPA, Region VI, 1445 Ross Avenue, Dallas, TX 75202, (214) 655-7229.

Region VII

Asbestos NESHAPs Contact, Air & Waste Management Division, USEPA, Region VII, 726 Minnesota Avenue, Kansas City, KS 66101, (913) 236-2896.

Region VIII

Asbestos NESHAPs Contact, Air & Waste Management Division, USEPA, Region VIII, 999 18th Street, Suite 500, Denver, CO 80202, (303) 293-1814.

Region IX

Asbestos NESHAPs Contact, Air Management Division, USEPA, Region IX, 215 Fremont Street, San Francisco, CA 94105, (415) 974-7633.

Region X

Asbestos NESHAPs Contact, Air & Toxics Management Division, USEPA, Region X, 1200 Sixth Avenue, Seattle, WA 98101, (206) 442-2724.

Once the transporter is satisfied with the condition of the **asbestos** waste and agrees to handle it, the containers should be loaded into the transport vehicle in a careful manner to prevent breaking of the containers. Similarly, at the disposal site, the **asbestos** waste containers should be transferred carefully to avoid fiber release.

Waste transport. Although there are no regulatory specifications regarding the transport vehicle, it is recommended that vehicles used for transport of containerized **asbestos** waste have an enclosed carrying compartment or utilize a canvas covering sufficient to contain the transported waste, prevent

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damage to containers, and prevent fiber release. Transport of large quantities of **asbestos** waste is commonly conducted in a 20-cubic-yard ``roll off'' box, which should also be covered. Vehicles that use compactors to reduce waste volume should not be used because these will cause the waste containers to rupture. Vacuum trucks used to transport waste slurry must be inspected to ensure that water is not leaking from the truck.

Disposal involves the isolation of **asbestos** waste material in order to prevent fiber release to air or water. Landfilling is recommended as an environmentally sound isolation method because **asbestos** fibers are virtually immobile in soil. Other disposal techniques such as incineration or chemical treatment are not feasible due to the unique properties of **asbestos**. EPA has established **asbestos** disposal requirements for active and inactive disposal sites under NESHAPs (40 CFR Part 61, subpart M) and specifies general requirements for solid waste disposal under RCRA (40 CFR Part 257). Advance EPA notification of the intended disposal site is required by NESHAPs.

Selecting a disposal facility. An acceptable disposal facility for **asbestos** wastes must adhere to EPA's requirements of no visible emissions to the air during disposal, or minimizing emissions by covering the waste within 24 hours. The minimum required cover is 6 inches of nonasbestos material, normally soil, or a dust-suppressing chemical. In addition to these Federal requirements, many state or local government agencies require more stringent handling procedures. These agencies usually supply a list of ``approved'' or licensed **asbestos** disposal sites upon request. Solid waste control agencies are listed in local telephone directories under state, county, or city headings. A list of state solid waste agencies may be obtained by calling the RCRA hotline: 1-800-424-9346 (382-3000 in Washington, DC). Some landfill owners or operators place special requirements on **asbestos** waste, such as placing all bagged waste into 55-gallon metal drums. Therefore, **asbestos** removal contractors should contact the intended landfill before arriving with the waste.

Receiving **asbestos** waste. A landfill approved for receipt of **asbestos** waste should require notification by the waste hauler that the load contains **asbestos**. The landfill operator should inspect the loads to verify that **asbestos** waste is properly contained in leak-tight containers and labeled appropriately. The appropriate EPA Regional **Asbestos** NESHAPs Contact should be notified if the landfill operator believes that the **asbestos** waste is in a condition that may cause significant fiber release during disposal. In situations when the wastes are not properly containerized, the landfill operator should thoroughly soak the **asbestos** with a water spray prior to unloading, rinse out the truck, and immediately cover the wastes with nonasbestos material prior to compacting the waste in the landfill.

Waste deposition and covering. Recognizing the health dangers associated with **asbestos** exposure, the following procedures are recommended to augment current federal requirements:

<bullet> Designate a separate area for **asbestos** waste disposal. Provide a record for future landowners that **asbestos** waste has been buried there and that it would be hazardous to attempt to excavate that area. (Future regulations may require property deeds to identify the location of any **asbestos** wastes and warn against excavation.)

<bullet> Prepare a separate trench to receive **asbestos** wastes. The size of the trench will depend upon the quantity and frequency of **asbestos** waste delivered to the disposal site. The trenching technique allows application of soil cover without disturbing the **asbestos** waste containers. The trench should be ramped to allow the transport vehicle to back into it, and the trench should be as narrow as possible to reduce the amount of cover required. If possible, the trench should be aligned perpendicular to prevailing winds.

<bullet> Place the **asbestos** waste containers into the trench carefully to avoid breaking them. Be particularly careful with plastic bags because when they break under pressure **asbestos** particles can be emitted.

<bullet> Completely cover the containerized waste within 24 hours with a minimum of 6 inches of nonasbestos material. Improperly containerized waste is a violation of the NESHAPs and EPA should be notified.

However, if improperly containerized waste is received at the disposal site, it should be covered immediately after unloading. Only after the wastes, including properly containerized wastes, are completely covered, can the wastes be compacted or other heavy equipment run over it. During compacting, avoid exposing wastes to the air or tracking **asbestos** material away from the trench.

<bullet> For final closure of an area containing **asbestos** waste, cover with at least an additional 30 inches of compacted nonasbestos material to provide a 36-inch final cover. To control erosion of the final cover, it should be properly graded and vegetated. In areas of the United States where excessive soil erosion may occur or the frost line exceeds 3 feet, additional final cover is recommended. In desert areas where vegetation would be difficult to maintain, 3-6 inches of well graded crushed rock is recommended for placement on top of the final cover.

Controlling public access. Under the current NESHAPs regulation, EPA does not require that a landfill used for **asbestos** disposal use warning signs or fencing if it meets the requirement to cover **asbestos** wastes. However, under RCRA, EPA requires that access

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be controlled to prevent exposure of the public to potential health and safety hazards at the disposal site. Therefore, for liability protection of operators of landfills that handle **asbestos**, fencing and warning signs are recommended to control public access when natural barriers do not exist. Access to a landfill should be limited to one or two entrances with gates that can be locked when left unattended. Fencing should be installed around the perimeter of the disposal site in a manner adequate to deter access by the general public. Chain-link fencing, 6-ft high and topped with a barbed wire guard, should be used. More specific fencing requirements may be specified by local regulations. Warning signs should be displayed at all entrances and at intervals of 330 feet or less along the property line of the landfill or perimeter of the sections where **asbestos** waste is deposited. The sign should read as follows:

ASBESTOS WASTE DISPOSAL SITE
BREATHING **ASBESTOS** DUST MAY CAUSE LUNG DISEASE AND CANCER

Recordkeeping. For protection from liability, and considering possible future requirements for notification on disposal site deeds, a landfill owner should maintain documentation of the specific location and quantity of the buried **asbestos** wastes. In addition, the estimated depth of the waste below the surface should be recorded whenever a landfill section is closed. As mentioned previously, such information should be recorded in the land deed or other record along with a notice warning against excavation of the area.

[52 FR 41897, Oct. 30, 1987, as amended at 62 FR 1834, Jan. 14, 1997]

Appendix E to Subpart E of Part 763--Interim Method of the Determination of **Asbestos** in Bulk Insulation Samples

Section 1, Polarized Light Microscopy

1.1 Principle and Applicability

Bulk samples of building materials taken for **asbestos** identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.\1\ \2\ A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting \2\ and the interpretation of point count data \3\ is available.

This method is applicable to all bulk samples of friable insulation materials submitted for identification and quantitation of **asbestos** components.

1.2 Range

The point counting method may be used for analysis of samples containing from 0 to 100 percent **asbestos**. The upper detection limit is 100 percent. The lower detection limit is less than 1 percent.

1.3 Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the **asbestos** mineral content. Spray-on binder materials may coat fibers and affect color or obscure optical characteristics to the extent of masking fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Procedures that may be used for the removal of interferences are presented in Section 1.7.2.2.

1.4 Precision and Accuracy

Adequate data for measuring the accuracy and precision of the method for samples with various matrices are not currently available. Data obtained for samples containing a single **asbestos** type in a simple matrix are available in the EPA report Bulk Sample Analysis for **Asbestos** Content: Evaluation of the Tentative Method.\4\

1.5 Apparatus

1.5.1 Sample Analysis

A low-power binocular microscope, preferably stereoscopic, is used to examine the bulk insulation sample as received.
<bullet> Microscope: binocular, 10-45X (approximate).
<bullet> Light Source: incandescent or fluorescent.
<bullet> Forceps, Dissecting Needles, and Probes
<bullet> Glassine Paper or Clean Glass Plate

Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360 deg. graduated rotating stage, substage condenser, lamp, and lamp iris.
<bullet> Polarized Light Microscope: described above.

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<bullet> Objective Lenses: 10X, 20X, and 40X or near equivalent.
<bullet> Dispersion Staining Objective Lens (optional)
<bullet> Ocular Lens: 10X minimum.
<bullet> Eyepiece Reticle: cross hair or 25 point Chalkley Point Array.
<bullet> Compensator Plate: 550 millimicron retardation.

1.5.2 Sample Preparation

Sample preparation apparatus requirements will depend upon the type of insulation sample under consideration. Various physical and/or chemical means may be employed for an adequate sample assessment.
<bullet> Ventilated Hood or negative pressure glove box.
<bullet> Microscope Slides
<bullet> Coverslips
<bullet> Mortar and Pestle: agate or porcelain. (optional)
<bullet> Wylie Mill (optional)
<bullet> Beakers and Assorted Glassware (optional)
<bullet> Certrifuge (optional)
<bullet> Filtration apparatus (optional)
<bullet> Low temperature asher (optional)

1.6 Reagents

1.6.1 Sample Preparation

<bullet> Distilled Water (optional)
<bullet> Dilute CH<INF>3</INF>COOH: ACS reagent grade (optional)
<bullet> Dilute HCl: ACS reagent grade (optional)
<bullet> Sodium metaphosphate (NaPO<INF>3</INF>)<INF>6</INF> (optional)

1.6.2 Analytical Reagents

Refractive Index Liquids: 1.490-1.570, 1.590-1.720 in increments of 0.002 or 0.004.
<bullet> Refractive Index Liquids for Dispersion Staining: high-dispersion series, 1.550, 1.605, 1.630 (optional).
<bullet> UICC **Asbestos** Reference Sample Set: Available from: UICC MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan CF6 1XW, UK, and commercial distributors.
<bullet> Tremolite-**asbestos** (source to be determined)
<bullet> Actinolite-**asbestos** (source to be determined)

1.7 Procedures

Note: Exposure to airborne **asbestos** fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

1.7.1 Sampling

Samples for analysis of **asbestos** content shall be taken in the manner prescribed in Reference 5 and information on design of sampling and analysis programs may be found in Reference 6. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

1.7.2 Analysis

1.7.2.1 Gross Examination

Bulk samples of building materials taken for the identification and quantitation of **asbestos** are first examined for homogeneity at low magnification with the aid of a stereomicroscope. The core sample may be examined in its container or carefully removed from the container onto a glassine transfer paper or clean glass plate. If possible, note is made of the top and bottom orientation. When discrete strata are identified, each is treated as a separate material so that fibers are first identified and quantified in that layer only, and then the results for each layer are combined to yield an estimate of **asbestos** content for the whole sample.

1.7.2.2 Sample Preparation

Bulk materials submitted for **asbestos** analysis involve a wide variety of matrix materials. Representative subsamples may not be readily obtainable by simple means in heterogeneous materials, and various steps may be required to alleviate the difficulties encountered. In most cases, however, the best preparation is made by using forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope.

Alternatively, attempts may be made to homogenize the sample or eliminate interferences before further characterization. The selection of appropriate procedures is dependent upon the samples encountered and personal preference. The following are presented as possible sample preparation steps.

A mortar and pestle can sometimes be used in the size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced in a Wylie mill. Apparatus should be clean and extreme care exercised to avoid cross-contamination of samples. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. These procedures are not recommended for samples that contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments with aspect ratios greater than 3:1. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

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Acid treatment may occasionally be required to eliminate interferences. Calcium carbonate, gypsum, and bassanite (plaster) are frequently present in sprayed or trowelled insulations. These materials may be removed by treatment with warm dilute acetic acid. Warm dilute hydrochloric acid may also be used to remove the above materials. If acid treatment is required, wash the sample at least twice with distilled water, being careful not to lose the particulates during decanting steps. Centrifugation or filtration of the suspension will prevent significant fiber loss. The pore size of the filter should be 0.45 micron or less. Caution: prolonged acid contact with the sample may alter the optical characteristics of chrysotile fibers and should be avoided.

Coatings and binding materials adhering to fiber surfaces may also be removed by treatment with sodium metaphosphate.\7\ Add 10 mL of 10g/L sodium metaphosphate solution to a small (0.1 to 0.5 mL) sample of bulk material in a 15-mL glass centrifuge tube. For approximately 15 seconds each, stir the mixture on a vortex mixer, place in an ultrasonic bath and then shake by hand. Repeat the series. Collect the dispersed solids by centrifugation at 1000 rpm for 5 minutes. Wash the sample three times by suspending in 10 mL distilled water and recentrifuging. After washing, resuspend the pellet in 5 mL distilled water, place a drop of the suspension on a microscope slide, and dry the slide at 110 deg.C.

In samples with a large portion of cellulosic or other organic fibers, it may be useful to ash part of the sample and view the residue. Ashing should be performed in a low temperature asher. Ashing may also be performed in a muffle furnace at temperatures of 500 deg.C or lower. Temperatures of 550 deg.C or higher will cause dehydroxylation of the **asbestos** minerals, resulting in changes of the refractive index and other key parameters. If a muffle furnace is to be used, the furnace thermostat should be checked and calibrated to ensure that samples will not be heated at temperatures greater than 550 deg.C.

Ashing and acid treatment of samples should not be used as standard procedures. In order to monitor possible changes in fiber characteristics, the material should be viewed microscopically before and after any sample preparation procedure. Use of these procedures on samples to be used for quantitation requires a correction for percent weight loss.

1.7.2.3 Fiber Identification

Positive identification of **asbestos** requires the determination of the following optical properties.

<bullet> Morphology
<bullet> Color and pleochroism
<bullet> Refractive indices
<bullet> Birefringence
<bullet> Extinction characteristics
<bullet> Sign of elongation

Table 1-1 lists the above properties for commercial **asbestos** fibers. Figure 1-1 presents a flow diagram of the examination procedure. Natural variations in the conditions under which deposits of asbestiform minerals are formed will occasionally produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Alternatively, dispersion staining may be used. Inexperienced operators may find that the dispersion staining technique is more easily learned, and should consult Reference 9 for guidance. Central stop dispersion staining colors are presented in Table 1-2. Available high-dispersion (HD) liquids should be used.

Table 1-1--Optical Properties of Asbestoc Fibers

Mineral	Morphology, color \a\	Refrac- tive indices \b\		Birefring- ence
		[alpha]	[gamma]	

Chrysotile (asbestiform serpentine).	Wavy fibers. Fiber bundles have splayed ends and ``kinks''. Aspect ratio typically <ls-thn-eq>10:1. Colorless \3\, nonpleochroic.	1.493-1.560	1.517-1.562\f\ (normally 1.556).	.008
Amosite (asbestiform grunerite).	Straight, rigid fibers. Aspect ratio typically <ls-thn-eq>10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635-1.696	1.655-1.729 \f\ (normally 1.696-1.710.	.020-.033
Crocidolite (asbestiform Riebeckite).	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654-1.701	1.668-1.717\3e\ (normally close to 1.700).	.014-.016

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Anthophyllite-**asbestos**.	Straight fibers and acicular cleavage fragments.\d\ Some composite fibers. Aspect ratio <10:1. Colorless to light brown.	1.596-1.652	1.615-1.676 \f\.	.019-.024
Tremolite-actinolite-**asbestos**.	Normally present as acicular or prismatic cleavage fragments.\d\ Single crystals predominate, aspect ratio <10:1. Colorless to pale green.	1.599-1.668	1.622-1.688 \f\.	.023-.020

\a\ From reference 5; colors cited are seen by observation with plane polarized ligh
\b\ From references 5 and 8.
\c\ Fibers subjected to heating may be brownish.
\d\ Fibers defined as having aspect ratio <ls-thn-eq>3:1.
\e\ to fiber length.
\f\ [verbar]To fiber length.

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[GRAPHIC] [TIFF OMITTED] TC01AP92.017

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Table 1-2--Central Stop Dispersion Staining Colors \a\

Mineral	RI Liquid	[eta]
Chrysotile...........................	1.550 \HD\............	Blue................
Amosite..............................	1.680.................	Blue-magenta to pale blue.
	1.550\HD\.............	Yellow to white.....
Crocidolite \b\......................	1.700.................	Red magenta.........
	1.550\HD\.............	Yellow to white.....
Anthophyllite........................	1.605\HD\.............	Blue................
Tremolite............................	1.605\HD c\...........	Pale blue...........
Actinolite...........................	1.605\HD\.............	Gold-magenta to blue
	1.630\HD c\...........	Magenta.............

\a\ From reference 9.
\b\ Blue absorption color.
\c\ Oblique extinction view.

1.7.2.4 Quantitation of **Asbestos** Content

Asbestos quantitation is performed by a point-counting procedure or an equivalent estimation method. An ocular reticle (cross-hair or point array) is used to visually superimpose a point or points on the microscope field of view. Record the number of points positioned directly above each kind of particle or fiber of interest. Score only points directly over **asbestos** fibers or nonasbestos matrix material. Do not score empty points for the closest particle. If an **asbestos** fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories. Point counting provides a determination of the area percent **asbestos**. Reliable conversion of area percent to percent of dry weight is not currently feasible unless the specific gravities and relative volumes of the materials are known.

For the purpose of this method, ``**asbestos** fibers'' are defined as having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 1-1.

A total of 400 points superimposed on either **asbestos** fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forcep samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either
<bullet> A cross-hair reticle and mechanical stage; or
<bullet> A reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X or at the lowest magnification of the polarized light microscope that can effectively distinguish the sample components.

Confirmation of the quantitation result by a second analyst on some percentage of analyzed samples should be used as standard quality control procedure.

The percent **asbestos** is calculated as follows:

% **asbestos**=(a/n) 100%

where

a=number of **asbestos** counts,
n=number of nonempty points counted (400).

If a=0, report ``No **asbestos** detected.'' If 0< a[lE]3, report ``<1% **asbestos**''.

The value reported should be rounded to the nearest percent.

1.8 References

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Section 2, X-Ray Powder Diffraction

2.1 Principle and Applicability

The principle of X-ray powder diffraction (XRD) analysis is well established.\1 2\ Any solid, crystalline material will diffract an impingent beam of parallel, monochromatic X-rays whenever Bragg's Law,

[lambda] = 2d sin [thetas],

is satisfied for a particular set of planes in the crystal lattice, where

[lambda] = the X-ray wavelength, A;
d = the interplanar spacing of the set of reflecting lattice planes, A; and

[thetas] = the angle of incidence between the X-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident X-ray beam, a diffraction pattern can be generated that, in most cases, will be uniquely characteristic of both the chemical composition and structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in **asbestos** analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1). However, when used in conjunction with optical methods such as polarized light microscopy (PLM), XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For qualitative analysis by XRD methods, samples are initially scanned over limited diagnostic peak regions for the serpentine ([sim]7.4 A) and amphibole (8.2-8.5 A) minerals (Table 2-2). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of **asbestos** (<ls-thn-eq>5-10 percent). Detection of minor or trace amounts of **asbestos** may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals are submitted to a full (5 deg.-60 deg. 2[thetas]; 1 deg. 2[thetas]/min) qualitative XRD scan, and their diffraction patterns are compared with standard reference powder diffraction patterns \3\ to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Table 2-1--The **Asbestos** Minerals and Their Nonasbestiform Analogs

Asbestiform	Nonasbestiform
SERPENTINE	
Chrysotile	Antigorite, lizardite
AMPHIBOLE	
Anthophyllite **asbestos**	Anthophyllite
Cummingtonite-grunerite **asbestos** (``Amosite'')	Cummingtonite-grunerite
Crocidolite	Riebeckite
Tremolite **asbestos**	Tremolite
Actinolite **asbestos**	Actinolite

Table 2-2--Principal Lattice Spacings of Asbestiform Minera

Minerals	Principal d-spacings (A) and relative intensities		J
Chrysotile...............................	7.37<INF>100</INF>	3.65<INF>70</	
	7.36<INF>100</INF>	3.66<INF>80</	
	7.10<INF>100</INF>	2.33<INF>80</	
``Amosite''..............................	8.33<INF>100</INF>	3.06<INF>70</	
	8.22<INF>100</INF>	3.060<INF>85<	
Anthophyllite............................	3.05<INF>100</INF>	3.24<INF>60</	
	3.06<INF>100</INF>	8.33<INF>70</	
Anthophyllite............................	2.72<INF>100</INF>	2.54<INF>100<	
Crocidolite..............................	8.35<INF>100</INF>	3.10<INF>55</	

Tremolite................................	8.38<INF>100</INF> 2.706<INF>100</INF> 3.13<INF>100</INF>	3.12<INF>100< 3.14<INF>95</ 2.706<INF>60<

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^a This information is intended as a guide, only. Complete powder diffract source, should be referred to, to ensure comparability of sample and reference mat Additional precision XRD data on amosite, crocidolite, tremolite, and chrysotile a Bureaus of Mines.\4\

^b Fibrosity questionable.

Accurate quantitative analysis of **asbestos** in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis is selected for quantitation of each asbestiform mineral. A ``thin-layer'' method of analysis \5 6\ is recommended in which, subsequent to comminution of the bulk material to [sim]10 [mu]m by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of

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asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative ``thick-layer'' or bulk methods,\7 8\ may be used for semiquantitative analysis.

This XRD method is applicable as a confirmatory method for identification and quantitation of **asbestos** in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.2 Range and Sensitivity

The range of the method has not been determined.

The sensitivity of the method has not been determined. It will be variable and dependent upon many factors, including matrix effects (absoprtion and interferences), diagnostic reflections selected, and their relative intensities.

2.3 Limitations

2.3.1 Interferences

Since the fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,\9\ the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally occurring materials the commonly associated **asbestos**-related mineral interferences can usually be anticipated. However, for fabricated materials the nature of the interferences may vary greatly (Table 2-3) and present more serious problems in identification and quantitation.\10\ Potential interferences are summarized in Table 2-4 and include the following:

<bullet> Chlorite has major peaks at 7.19 A and 3.58 A That interfere with both the primary (7.36 A) and secondary (3.66 A) peaks for chrysotile. Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.
<bullet> Halloysite has a peak at 3.63 A that interferes with the secondary (3.66 A) peak for chrysotile.
<bullet> Kaolinite has a major peak at 7.15 A that may interfere with the primary peak of chrysotile at 7.36 A when present at concentrations of <ls-thn-eq>10 percent. However, the secondary chrysotile peak at 3.66 A may be used for quantitation.
<bullet> Gypsum has a major peak at 7.5 A that overlaps the 7.36 A peak of chrysotile when present as a major sample constituent. This may be removed by careful washing with distilled water, or be heating to 300 deg.C to convert gypsum to plaster of paris.
<bullet> Cellulose has a broad peak that partially overlaps the secondary (3.66 A) chrysotile peak.\8\
<bullet> Overlap of major diagnostic peaks of the amphibole **asbestos** minerals, amosite, anthophyllite, crocidolite, and tremolite, at approximately 8.3 A and 3.1 A causes mutual interference when these minerals occur in the presence of one another. In some instances, adquate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the X-ray port.

Table 2-3--Common Constituents in Insulation and Wall Materials

A. Insulation materials

Chrysotile
``Amosite''
Crocidolite
*Rock wool
*Slag wool
*Fiber glass
Gypsum ($CaSO_4 \cdot 2H_2O$)
Vermiculite (micas)
*Perlite
Clays (kaolin)
*Wood pulp
*Paper fibers (talc, clay, carbonate fillers)
Calcium silicates (synthetic)
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in ``amosite'')
Magnesite
*Diatomaceous earth

B. Spray finishes or paints

Bassanite
Carbonate minerals (calcite, dolomite, vaterite)
Talc
Tremolite
Anthophyllite
Serpentine (including chrysotile)
Amosite
Crocidolite
*Mineral wool
*Rock wool
*Slag wool
*Fiber glass

Clays (kaolin)
Micas
Chlorite
Gypsum (CaSO<INF>4</INF> [middot] 2H<INF>2</INF>O)
Quartz
*Organic binders and thickeners
Hyrdomagnesite
Wollastonite
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in ``amosite'')

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*Amorphous materials----contribute only to overall scattered radiation and increased background radiation.

Table 2-4--Interferences in XRD Analysis Asbestiform Minerals

Asbestiform mineral	Primary diagnostic peaks (approximate d-spacings, in A)	Interference
Serpentine		
Chrysotile	7.4	Nonasbestiform serpentines (antigorite, lizardite) Chlorite Kaolinite Gypsum
	3.7	Chlorite Halloysite Cellulose
Amphibole		
``Amosite'' Anthophyllite <3-ln [>||<ct-integr<sq-root><integral><3-ln |><3-ln Crocidolite Tremolite	3.1	Nonasbestiform amphiboles (cummingtonite-grunerite, anthophyllite, riebeckite, tremolite) Mutual interferences Carbonates Talc
	8.3	Mutual interferences

<bullet> Carbonates may also interfere with quantitative analysis of the amphibole **asbestos** minerals, amosite, anthophyllite, crocidolite, and tremolite. Calcium carbonate (CaCO<INF>3</INF>) has a peak at 3.035 A that overlaps major amphibole peaks at approximately 3.1 A when present in concentrations of <ls-thn-eq>5 percent. Removal of carbonates with a dilute acid wash is possible; however, if present, chrysotile may be partially dissolved by this treatment.\11\
<bullet> A major talc peak at 3.12 A interferes with the primary tremolite peak at this same position and with secondary peaks of crocidolite (3.10 A), amosite (3.06 A), and anthophyllite (3.05 A). In

the presence of talc, the major diagnostic peak at approximately 8.3 A should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interferences is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the **asbestos** minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, with the result being that their diffraction patterns are chracterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of the orthorhombic anthophyllite separated by less than 0.2 A.\12\

2.3.2 Matrix Effects

If a copper X-ray source is used, the presence of iron at high concentrations in a sample will result in significant X-ray fluorescence, leading to loss of peak intensity along with increased background intensity and an overall decrease in sensitivity. This situation may be corrected by choosing an X-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a diffracted beam monochromator will reduce background fluorescent raditation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently ``thin'' samples for analysis.\5 13 14\ However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard \7 8\ or filter substrate (Ag) peak.\5 6\

2.3.3 Particle Size Dependence

Because the intensity of diffracted X-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size range for quantitative analysis of **asbestos** by XRD has been reported to be 1 to 10 [mu] m.\15\ Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in ``thick layer'' samples.\7 8 15\ However, for ``thin'' samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of **asbestos** minerals, actually increasing the overall sensitivity of the method.\12 14\ (Further investigation into preferred orientation effects in both thin layer and bulk samples is required.)

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2.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for **asbestos** analysis is clearly recognized. NIOSH has recently directed a major research effort toward the preparation and characterization of analytical reference materials, including **asbestos** standards; ^{16 17} however, these are not available in large quantities for routine analysis.

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.\18\ have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be underestimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4 Precision and Accuracy

Precision of the method has not been determined.
Accuracy of the method has not been determined.

2.5 Apparatus

2.5.1 Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

<bullet> Mortar and Pestle: Agate or porcelain.
<bullet> Razor Blades
<bullet> Sample Mill: SPEX, Inc., freezer mill or equivalent.
<bullet> Bulk Sample Holders
<bullet> Silver Membrane Filters: 25-mm diameter, 0.45-[mu] m pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006.
<bullet> Microscope Slides
<bullet> Vacuum Filtration Apparatus: Gelman No. 1107 or equivalent, and side-arm vacuum flask.
<bullet> Microbalance
<bullet> Ultrasonic Bath or Probe: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent.
<bullet> Volumetric Flasks: 1-L volume.
<bullet> Assorted Pipettes
<bullet> Pipette Bulb
<bullet> Nonserrated Forceps
<bullet> Polyethylene Wash Bottle
<bullet> Pyrex Beakers: 50-mL volume.
<bullet> Desiccator
<bullet> Filter Storage Cassettes
<bullet> Magnetic Stirring Plate and Bars
<bullet> Porcelain Crucibles
<bullet> Muffle Furnace or Low Temperature Asher

2.5.2 Sample Analysis

Sample analysis requirements include an X-ray diffraction unit, equipped with:

- Constant Potential Generator; Voltage and mA Stabilizers
- Automated Diffractometer with Step-Scanning Mode
- Copper Target X-Ray Tube: High intensity, fine focus, preferably.
- X-Ray Pulse Height Selector
- X-Ray Detector (with high voltage power supply): Scintillation or proportional counter.
- Focusing Graphite Crystal Monochromator; or Nickel Filter (if copper source is used, and iron fluorescence is not a serious problem).
- Data Output Accessories:
    - Strip Chart Recorder
    - Decade Scaler/Timer
    - Digital Printer
- Sample Spinner (optional).
- Instrument Calibration Reference Specimen: [alpha]-quartz reference crystal (Arkansas quartz standard, <greek-i>180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

2.6 Reagents

2.6.1 Standard Reference Materials

The reference materials listed below are intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- Chrysotile: UICC Canadian, or NIEHS Plastibest. (UICC reference materials available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK).
- Crocidolite: UICC
- Amosite: UICC
- Anthophyllite: UICC
- Tremolite **Asbestos**: Wards Natural Science Establishment, Rochester, N.Y.; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 90064 (washed with dilute HCl to remove small amount of calcite impurity); India tremolite, Rajasthan State, India.
- Actinolite **Asbestos**

2.6.2 Adhesive

Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

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2.6.3 Surfactant

1 percent aerosol OT aqueous solution or equivalent.

2.6.4 Isopropanol

ACS Reagent Grade.

2.7 Procedure

2.7.1 Sampling

Samples for analysis of **asbestos** content shall be collected as specified in EPA Guidance Document <greek-i>C0090, **Asbestos**-Containing Materials in School Buildings.\10\

2.7.2 Analysis

All samples must be analyzed initially for **asbestos** content by PLM. XRD should be used as an auxiliary method when a second, independent analysis is requested.

Note: **Asbestos** is a toxic substance. All handling of dry materials should be performed in an operating fume hood.

2.7.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) The condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative, quantitative, thin layer or bulk).

Bulk materials are usually received as inhomogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from **asbestos**-containing materials is particularly difficult because the fibrous nature of the **asbestos** minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.7.2.2 and 2.7.2.3.

Note: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.

2.7.2.1.1 Milling-- Mechanical milling of **asbestos** materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.^{19,&thnsp[gE]22} Therefore, all milling times should be kept to a minimum.

For qualitative analysis, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (<ls-thn-eq>2-3 mm) should be comminuted to [sim]100 [mu]m. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other suitable wetting agent, to minimize exposures.

For accurate, reproducible quantitative analysis, the particle size of both sample and standard materials should be reduced to [sim]10 [mu]m (see Section 2.3.3). Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill, or equivalent) for a maximum time of 10 min. is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice. \5\ Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to <10 [mu]m. \8, 16\

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.7.2.1.2 Low temperature ashing-- For materials shown by PLM to contain large amounts of gypsum, cellulosic, or other organic materials, it may be desirable to ash the samples prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550 deg.C and and 650 deg.C, with subsequent transformation to forsterite,\23, 24\ ashing temperatures should be kept below 500 deg.C. Use of a low temperature asher is recommended. In all cases, calibration of the oven is essential to ensure that a maximum ashing temperature of 500 deg.C is not exceeded.

2.7.2.1.3 Acid leaching--Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.3.1), it may be necessary to remove these interferents by a simple acid leaching procedure prior to analysis (see Section 1.7.2.2).

2.7.2.2 Qualitative Analysis

2.7.2.2.1 Initial screening of bulk material-- Qualitative analysis should be performed on a representative, homogeneous portion of the sample with a minimum of sample treatment.

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.7.2.1.1.) to a final particle size sufficiently small ([sim]100 [mu]m) to allow adequate packing into the sample holder.
2. Pack the sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the

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milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. (Note: Use of a back-packing method \25\ of bulk sample preparation may reduce preferred orientation effects.)

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine ([sim]67.4 A) and amphibole (8.2-8.5 A) minerals (see Table 2-2). The X-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1 deg. 2<INF>[thetas]</INF>/min is recommended for adequate resolution. Use of a sample spinner is recommended.
4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5 deg.-60 deg. 2<INF>[thetas]</INF>; 1 deg.2<INF>[thetas]</INF>/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.
5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data \3\ or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-2; common constituents of bulk insulation and wall materials are listed in Table 2-3.

2.7.2.2.2 Detection of minor or trace constituents-- Routine screening of bulk materials by XRD may fail to detect small concentrations (<5 percent) of **asbestos**. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 [mu]m range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances where confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by

XRD (see Section 2.7.2.2.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.7.2.3) and step-scan over appropriate 2<INF>[thetas]</INF> ranges of selected diagnostic peaks (Table 2-2). Accurate transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.7.2.3 Quantitative Analysis

The proposed method for quantitation of **asbestos** in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air. \5\ A thick-layer or bulk method involving pelletizing the sample may be used for semiquantitative analysis; \7,8\ however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative **asbestos** analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.7.2.1.1.
2. Dry at 100 deg.C for 2 hr; cool in a desiccator.
3. Weigh accurately to the nearest 0.01 mg.
4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.7.2.1.2 and 2.7.2.1.3. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss; L.
5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask with approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.
6. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the sample material.
7. Dilute to volume with isopropanol.
8. Place flask on a magnetic stirring plate. Stir.
9. Place a silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the **asbestos** suspension and immediately withdraw an aliquot from the center of the suspension so that total sample weight, W<INF>T</INF>, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)
10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.
11. For each **asbestos** mineral to be quantitated select a reflection (or reflections) that has been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-2).
12. Analyze the selected diagnostic reflection(s) by step scanning

in increments of

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0.02 deg. 2<INF>[thetas]</INF> for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I<INF>a</INF>, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I<INF>Ag</INF>, of the filter 2.36 A silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I<INF>A.7g</INF>. Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 A peak of an [alpha]-quartz reference crystal). After each unknown is scanned, determine the net count, I<INF>r</INFI, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I<INF>r.7:</INF>
[GRAPHIC] [TIFF OMITTED] TC01AP92.018

2.8 Calibration

2.8.1 Preparation of Calibration Standards

1. Mill and size standard **asbestos** materials according to the procedure outlined in Section 2.7.2.1.1. Equivalent, standardized matrix reduction and sizing techniques should be used for both standard and sample materials.

2. Dry at 100 deg.C for 2 hr; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Quantitatively transfer each to a 1-L volumetric flask with approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the **asbestos** material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions and the following procedure.

Mount a silver membrane filter on the filtration apparatus. Place a few milliliters of isopropanol in the reservoir. Vigorously hand shake the **asbestos** suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.8.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.7.2.3, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, I<INF>s.7td</INF>, as outlined in Section 2.7.2.3, step 14.

2.9 Calculations

For each **asbestos** reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing I2<INF>s.7td</INF> on w. Poor reproducibility (<plus-minus>15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight line equation.

Determine the slope, m, of the calibration curve in counts/ microgram. The intercept, b, of the line with the I<INF>s.7td</INF> axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, I<INF>Ag</INF>, for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak, I<INF>A.7g</INF>, of the sample filter, calculate the transmittance, T, for each sample as follows:\26\ \27\

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[GRAPHIC] [TIFF OMITTED] TC01AP92.019

Determine the correction factor, f(T), for each sample according to the formula:

```
                          -R (ln T)
          f (T)           --------
               =
                            l-T<SUP>R</SUP>
```

where

```
                     sin [Theta]<INF>Ag</INF>
           R =          --------
                      sin [Theta]<INF>a</INF>
```

<INF>[thetas]Ag</INF>=angular position of the measured silver peak (from Bragg's Law), and
<INF>[thetas]a</INF>=angular position of the diagnostic **asbestos** peak.

Calculate the weight, W<INF>a</INF>, in micrograms, of the **asbestos** material analyzed for in each sample, using the appropriate calibration data and absorption corrections:
[GRAPHIC] [TIFF OMITTED] TC01AP92.020

Calculate the percent composition, P<INF>a</INF>, of each **asbestos** mineral analyzed for in the parent material, from the total sample weight, W<INF>T</INF>, on the filter:

W<INF>a</INF>(1-.01L)
P<INF>a</INF> <INF>=</INF> ---------- x 100
W<INF>T</INF>

where

P<INF>a</INF>=percent **asbestos** mineral in parent material;
W<INF>a</INF>=mass of **asbestos** mineral on filter, in [mu]g;
W<INF>T</INF>=total sample weight on filter, in [mu]g;
L=percent weight loss of parent material on ashing and/or acid treatment (see Section 2.7.2.3).

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[47 FR 23369, May 27, 1982; 47 FR 38535, Sept. 1, 1982; Redesignated at 60 FR 31922, June 19, 1995]

Subpart F [Reserved]

Subpart G--**Asbestos** Worker Protection

Source: 65 FR 69216, Nov. 15, 2000, unless otherwise noted.

Sec. 763.120 What is the purpose of this subpart?

This subpart protects certain State and local government employees who are not protected by the **Asbestos** Standards of the Occupational Safety and Health Administration (OSHA). This subpart applies the OSHA **Asbestos** Standards in 29 CFR 1910.1001 and 29 CFR 1926.1101 to these employees.

Sec. 763.121 Does this subpart apply to me?

If you are a State or local government employer and you are not subject to a State **asbestos** standard that OSHA has approved under section 18 of the Occupational Safety and Health Act or a State **asbestos** plan that EPA has exempted from the requirements of this subpart under Sec. 763.123, you must follow the requirements of this subpart to protect your employees from occupational exposure to **asbestos**.

Sec. 763.122 What does this subpart require me to do?

If you are a State or local government employer whose employees perform:

(a) Construction activities identified in 29 CFR 1926.1101(a), you must:

(1) Comply with the OSHA standards in 29 CFR 1926.1101.

(2) Submit notifications required for alternative control methods to the Director, National Program Chemicals Division (7404), Office of Pollution Prevention and Toxics, Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460.

(b) Custodial activities not associated with the construction activities identified in 29 CFR 1926.1101(a), you must comply with the OSHA standards in 29 CFR 1910.1001.

(c) Repair, cleaning, or replacement of **asbestos**-containing clutch plates and brake pads, shoes, and linings, or removal of **asbestos**-containing residue from brake drums or clutch housings, you must comply with the OSHA standards in 29 CFR 1910.1001.

Sec. 763.123 May a State implement its own **asbestos** worker protection plan?

This section describes the process under which a State may be exempted from the requirements of this subpart.

(a) States seeking an exemption. If your State wishes to implement its own **asbestos** worker protection plan, rather than complying with the requirements of this subpart, your State must apply

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for and receive an exemption from EPA.

(1) What must my State do to apply for an exemption? To apply for an exemption from the requirements of this subpart, your State must send to the Director of EPA's Office of Pollution Prevention and Toxics (OPPT) a copy of its **asbestos** worker protection regulations and a detailed explanation of how your State's **asbestos** worker protection plan meets the requirements of TSCA section 18 (15 U.S.C. 2617).

(2) What action will EPA take on my State's application for an exemption? EPA will review your State's application and make a preliminary determination whether your State's **asbestos** worker protection plan meets the requirements of TSCA section 18.

(i) If EPA's preliminary determination is that your State's plan does meet the requirements of TSCA section 18, EPA will initiate a rulemaking, including an opportunity for public comment, to exempt your State from the requirements of this subpart. After considering any comments, EPA will issue a final rule granting or denying the exemption.

(ii) If EPA's preliminary determination is that the State plan does not meet the requirements of TSCA section 18, EPA will notify your State in writing and will give your State a reasonable opportunity to respond to that determination.

(iii) If EPA does not grant your State an exemption, then the State and local government employers in your State are subject to the requirements of this subpart.

(b) States that have been granted an exemption. If EPA has exempted your State from the requirements of this subpart, your State must update its **asbestos** worker protection regulations as necessary to implement changes to meet the requirements of this subpart, and must apply to EPA for an amendment to its exemption.

(1) What must my State do to apply for an amendment to its exemption? To apply for an amendment to its exemption, your State must send to the Director of OPPT a copy of its updated **asbestos** worker protection regulations and a detailed explanation of how your State's updated **asbestos** worker protection plan meets the requirements of TSCA section 18. Your State must submit its application for an amendment within 6 months of the effective date of any changes to the requirements of this subpart, or within a reasonable time agreed upon by your State and OPPT.

(2) What action will EPA take on my State's application for an amendment? EPA will review your State's application for an amendment and make a preliminary determination whether your State's updated **asbestos** worker protection plan meets the requirements of TSCA section 18.

(i) If EPA determines that the updated State plan does meet the requirements of TSCA section 18, EPA will issue your State an amended exemption.

(ii) If EPA determines that the updated State plan does not meet the requirements of TSCA section 18, EPA will notify your State in writing and will give your State a reasonable opportunity to respond to that determination.

(iii) If EPA does not grant your State an amended exemption, or if your State does not submit a timely request for amended exemption, then the State and local government employers in your State are subject to the requirements of this subpart.

Subpart H [Reserved]

Subpart I--Prohibition of the Manufacture, Importation, Processing, and Distribution in Commerce of Certain **Asbestos**-Containing Products; Labeling Requirements

Source: 54 FR 29507, July 12, 1989, unless otherwise noted.

Sec. 763.160 Scope.

This subpart prohibits the manufacture, importation, processing, and distribution in commerce of the **asbestos**-containing products identified and at the dates indicated in Secs. 763.165, 763.167, and 763.169. This subpart requires that products subject to this rule's bans, but not yet subject to a ban on distribution in commerce, be labeled. This

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subpart also includes general exemptions and procedures for requesting exemptions from the provisions of this subpart.

Sec. 763.163 Definitions.

For purposes of this subpart:

Act means the Toxic Substances Control Act, 15 U.S.C. 2601 et seq.

Agency means the United States Environmental Protection Agency.

Asbestos means the asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite-grunerite); tremolite; anthophyllite; and actinolite.

Asbestos-containing product means any product to which **asbestos** is deliberately added in any concentration or which contains more than 1.0 percent **asbestos** by weight or area.

Chemical substance, has the same meaning as in section 3 of the Act.

Commerce has the same meaning as in section 3 of the Act.

Commercial paper means an **asbestos**-containing product which is made of paper intended for use as general insulation paper or muffler paper. Major applications of commercial papers are insulation against fire, heat transfer, and corrosion in circumstances that require a thin, but durable, barrier.

Corrugated paper means an **asbestos**-containing product made of corrugated paper, which is often cemented to a flat backing, may be laminated with foils or other materials, and has a corrugated surface. Major applications of **asbestos** corrugated paper include: thermal insulation for pipe coverings; block insulation; panel insulation in elevators; insulation in appliances; and insulation in low-pressure steam, hot water, and process lines.

Customs territory of the United States means the 50 States, Puerto Rico, and the District of Columbia.

Distribute in commerce has the same meaning as in section 3 of the Act, but the term does not include actions taken with respect to an **asbestos**-containing product (to sell, resale, deliver, or hold) in connection with the end use of the product by persons who are users (persons who use the product for its intended purpose after it is manufactured or processed). The term also does not include distribution by manufacturers, importers, and processors, and other persons solely for purposes of disposal of an **asbestos**-containing product.

Flooring felt means an **asbestos**-containing product which is made of paper felt intended for use as an underlayer for floor coverings, or to be bonded to the underside of vinyl sheet flooring.

Import means to bring into the customs territory of the United States, except for: (1) Shipment through the customs territory of the United States for export without any use, processing, or disposal within the customs territory of the United States; or (2) entering the customs territory of the United States as a component of a product during normal personal or business activities involving use of the product.

Importer means anyone who imports a chemical substance, including a chemical substance as part of a mixture or article, into the customs territory of the United States. Importer includes the person primarily liable for the payment of any duties on the merchandise or an authorized agent acting on his or her behalf. The term includes as appropriate:

(1) The consignee.

(2) The importer of record.

(3) The actual owner if an actual owner's declaration and superseding bond has been filed in accordance with 19 CFR 141.20.

(4) The transferee, if the right to withdraw merchandise in a bonded warehouse has been transferred in accordance with subpart C of 19 CFR part 144.

Manufacture means to produce or manufacture in the United States.

Manufacturer means a person who produces or manufactures in the United States.

New uses of **asbestos** means commercial uses of **asbestos** not identified in Sec. 763.165 the manufacture, importation or processing of which would be initiated for the first time after August 25, 1989.

Person means any natural person, firm, company, corporation, joint-venture, partnership, sole proprietorship, association, or any other business entity; any State or political subdivision thereof, or any municipality; any interstate body and any department,

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agency, or instrumentality of the Federal Government.

Process has the same meaning as in section 3 of the Act.

Processor has the same meaning as in section 3 of the Act.

Rollboard means an **asbestos**-containing product made of paper that is produced in a continuous sheet, is flexible, and is rolled to achieve a desired thickness. **Asbestos** rollboard consists of two sheets of **asbestos** paper laminated together. Major applications of this product include: office partitioning; garage paneling; linings for stoves and electric switch boxes; and fire-proofing agent for security boxes, safes, and files.

Specialty paper means an **asbestos**-containing product that is made of paper intended for use as filters for beverages or other fluids or as paper fill for cooling towers. Cooling tower fill consists of **asbestos** paper that is used as a cooling agent for liquids from industrial processes and air conditioning systems.

State has the same meaning as in section 3 of the Act.

Stock-on-hand means the products which are in the possession, direction, or control of a person and are intended for distribution in commerce.

United States has the same meaning as in section 3 of the Act.

[59 FR 33208, June 28, 1994]

Sec. 763.165 Manufacture and importation prohibitions.

(a) After August 27, 1990, no person shall manufacture or import the following **asbestos**-containing products, either for use in the United States or for export: flooring felt and new uses of **asbestos**.

(b) After August 26, 1996, no person shall manufacture or import the following **asbestos**-containing products, either for use in the United States or for export: commercial paper, corrugated paper, rollboard, and specialty paper.

(c) The import prohibitions of this subpart do not prohibit:

(1) The import into the customs territory of the United States of products imported solely for shipment outside the customs territory of the United States, unless further repackaging or processing of the product is performed in the United States; or

(2) Activities involving purchases or acquisitions of small quantities of products made outside the customs territory of the United States for personal use in the United States.

[59 FR 33209, June 28, 1994]

Sec. 763.167 Processing prohibitions.

(a) After August 27, 1990, no person shall process for any use, either in the United States or for export, any of the **asbestos**-containing products listed at Sec. 763.165(a).

(b) After August 26, 1996, no person shall process for any use, either in the United States or for export, any of the **asbestos**-containing products listed at Sec. 763.165(b).

[59 FR 33209, June 28, 1994]

Sec. 763.169 Distribution in commerce prohibitions.

(a) After August 25, 1992, no person shall distribute in commerce, either for use in the United States or for export, any of the **asbestos**-containing products listed at Sec. 763.165(a).

(b) After August 25, 1997, no person shall distribute in commerce, either for use in the United States or for export, any of the **asbestos**-containing products listed at Sec. 763.165(b).

(c) A manufacturer, importer, processor, or any other person who is subject to a ban on distribution in commerce in paragraph (a) or (b) of this section must, within 6 months of the effective date of the ban of a specific **asbestos**-containing product from distribution in commerce, dispose of all their remaining stock-on-hand of that product, by means that are in compliance with applicable local, State, and Federal restrictions which are current at that time.

[59 FR 33209, June 28, 1994]

Sec. 763.171 Labeling requirements.

(a) After August 27, 1990, manufacturers, importers, and processors of all **asbestos**-containing products that are identified in Sec. 763.165(a) shall label the products as specified in this subpart at the time of manufacture, import, or processing. This requirement includes labeling all manufacturers', importers',

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and processors' stock-on-hand as of August 27, 1990.

(b) After August 25, 1995, manufacturers, importers, and processors of all **asbestos**-containing products that are identified in Sec. 763.165(b), shall label the products as specified in this subpart at the time of manufacture, import, or processing. This requirement includes labeling all manufacturers', importers', and processors' stock-on-hand as of August 25, 1995.

(c) The label shall be placed directly on the visible exterior of the wrappings and packaging in which the product is placed for sale, shipment, or storage. If the product has more than one layer of external wrapping or packaging, the label must be attached to the innermost layer adjacent to the product. If the innermost layer of product wrapping or packaging does not have a visible exterior surface larger than 5 square inches, either a tag meeting the requirements of paragraph (d) of this section must be securely attached to the product's innermost layer of product wrapping or packaging, or a label must be attached to the next outer layer of product packaging or wrapping. Any products that are distributed in commerce to someone other than the end user, shipped, or stored without packaging or wrapping must be labeled or tagged directly on a visible exterior surface of the product as described in paragraph (d) of this section.

(d)(1) Labels must be either printed directly on product packaging or in the form of a sticker or tag made of plastic, paper, metal, or other durable substances. Labels must be attached in such a manner that they cannot be removed without defacing or destroying them. Product labels shall appear as in paragraph (d)(2) of this section and consist of block letters and numerals of color that contrasts with the background of the label or tag. Labels shall be sufficiently durable to equal or exceed the life, including storage and disposal, of the product packaging or wrapping. The size of the label or tag must be at least

15.25 cm (6 inches) on each side. If the product packaging is too small to accommodate a label of this size, the label may be reduced in size proportionately to the size of the product packaging or wrapping down to a minimum 2.5 cm (1 inch) on each side if the product wrapping or packaging has a visible exterior surface larger than 5 square inches.

(2) Products subject to this subpart shall be labeled in English as follows:

NOTICE

This product contains **ASBESTOS**. The U.S. Environmental Protection Agency has banned the distribution in U.S. commerce of this product under section 6 of the Toxic Substances Control Act (15 U.S.C. 2605) as of (insert effective date of ban on distribution in commerce). Distribution of this product in commerce after this date and intentionally removing or tampering with this label are violations of Federal law.

(e) No one may intentionally remove, deface, cover, or otherwise obscure or tamper with a label or sticker that has been applied in compliance with this section, except when the product is used or disposed of.

[59 FR 33209, June 28, 1994]

Sec. 763.173 Exemptions.

(a) Persons who are subject to the prohibitions imposed by Secs. 763.165, 763.167, or 763.169 may file an application for an exemption. Persons whose exemption applications are approved by the Agency may manufacture, import, process, or distribute in commerce the banned product as specified in the Agency's approval of the application. No applicant for an exemption may continue the banned activity that is the subject of an exemption application after the effective date of the ban unless the Agency has granted the exemption or the applicant receives an extension under paragraph (b)(4) or (5) of this section.

(b) Application filing dates. (1) Applications for products affected by the prohibitions under Secs. 763.165(a) and 763.167(a) may be submitted at any time and will be either granted or denied by EPA as soon as is feasible.

(2) Applications for products affected by the ban under Sec. 763.169(a) may be submitted at any time and will be either granted or denied by EPA as soon as is feasible.

(3) Applications for products affected by the ban under Secs. 763.165(b) and 763.167(b) may not be submitted prior

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to February 27, 1995. Complete applications received after that date, but before August 25, 1995, will be either granted or denied by the Agency prior to the effective date of the ban for the product. Applications received after August 25, 1995, will be either granted or denied by EPA as soon as is feasible.

(4) Applications for products affected by the ban under Sec. 763.169(b) may not be submitted prior to February 26, 1996. Complete applications received after that date, but before August 26, 1996, will be either granted or denied by the Agency prior to the effective date of the ban for the product. Applications received after August 26, 1996, will be either granted or denied by EPA as soon as is feasible.

(5) The Agency will consider an application for an exemption from a

ban under Sec. 763.169 for a product at the same time the applicant submits an application for an exemption from a ban under Sec. 763.165 or Sec. 763.167 for that product. EPA will grant an exemption at that time from a ban under Sec. 763.169 if the Agency determines it appropriate to do so.

(6) If the Agency denies an application less than 30 days before the effective date of a ban for a product, the applicant can continue the activity for 30 days after receipt of the denial from the Agency.

(7) If the Agency fails to meet the deadlines stated in paragraphs (b)(3) and (b)(4) of this section for granting or denying a complete application in instances in which the deadline is before the effective date of the ban to which the application applies, the applicant will be granted an extension of 1 year from the Agency's deadline date. During this extension period the applicant may continue the activity that is the subject of the exemption application. The Agency will either grant or deny the application during the extension period. The extension period will terminate either on the date the Agency grants the application or 30 days after the applicant receives the Agency's denial of the application. However, no extension will be granted if the Agency is scheduled to grant or deny an application at some date after the effective date of the ban, pursuant to the deadlines stated in paragraphs (b)(3) and (b)(4) of this section.

(c) Where to file. All applications must be submitted to the following location: TSCA Docket Receipts Office (7407), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Rm E-G99, 1200 Pennsylvania Ave., NW., Washington, DC 20460, ATTENTION: **Asbestos** Exemption. For information regarding the submission of exemptions containing information claimed as confidential business information (CBI), see Sec. 763.179.

(d) Content of application and criteria for decisionmaking.

(1) Content of application. Each application must contain the following:

(i) Name, address, and telephone number of the applicant.

(ii) Description of the manufacturing, import, processing, and/or distribution in commerce activity for which an exemption is requested, including a description of the **asbestos**-containing product to be manufactured, imported, processed, or distributed in commerce.

(iii) Identification of locations at which the exempted activity would take place.

(iv) Length of time requested for exemption (maximum length of an exemption is 4 years).

(v) Estimated amount of **asbestos** to be used in the activity that is the subject of the exemption application.

(vi) Data demonstrating the exposure level over the life cycle of the product that is the subject of the application.

(vii) Data concerning:

(A) The extent to which non-**asbestos** substitutes for the product that is the subject of the application fall significantly short in performance under necessary product standards or requirements, including laws or ordinances mandating product safety standards.

(B) The costs of non-**asbestos** substitutes relative to the costs of the **asbestos**-containing product and, in the case in which the product is a component of another product, the effect on the cost of the end use product of using the substitute component.

(C) The extent to which the product or use serves a high-valued use.

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(viii) Evidence of demonstrable good faith attempts by the applicant to develop and use a non-**asbestos** substance or product which may be substituted for the **asbestos**-containing product or the **asbestos** in the product or use that is the subject to the application.

(ix) Evidence, in addition to that provided in the other information required with the application, showing that the continued manufacture, importation, processing, distribution in commerce, and use, as applicable, of the product will not present an unreasonable risk of injury to human health.

(2) Criteria for decision (existing products). After considering all the information provided by an applicant under paragraphs (d)(1) and (e) of this section, and any other information available to EPA, EPA will grant an exemption from the prohibitions in Secs. 763.165, 763.167, or 763.169 for an applicant's **asbestos**-containing product only if EPA determines both of the following:

(i) The applicant has made good faith attempts to develop and use a non-**asbestos** substance or product which may be substituted for the **asbestos**-containing product or the **asbestos** in the product or use, and those attempts have failed to produce a substitute or a substitute that results in a product that can be economically produced.

(ii) Continued manufacturing, processing, distribution in commerce, and use, as applicable, of the product will not present an unreasonable risk of injury to human health.

(3) Criteria for decision (new products). Requests to develop and use an **asbestos** substance or product will be treated as a petition pursuant to section 21 of TSCA.

(e) The Agency reserves the right to request further information from an exemption applicant if necessary to complete the Agency's evaluation of an application.

(f) Upon receipt of a complete application, the Agency will issue a notice in the Federal Register announcing its receipt and invite public comments on the merits of the application.

(g) If the application does not include all of the information required in paragraph (d) of this section, the Agency will return it to the applicant as incomplete and any resubmission of the application will be considered a new application for purposes of the availability of any extension period. If the application is substantially inadequate to allow the Agency to make a reasoned judgment on any of the information required in paragraph (d) of this section and the Agency chooses to request additional information from the applicant, the Agency may also determine that an extension period provided for in paragraph (b)(5) of this section is unavailable to the applicant.

(h) When denying an application, the Agency will notify the applicant by registered mail of its decision and rationale. Whenever possible, the Agency will send this letter prior to the appropriate ban. This letter will be considered a final Agency action for purposes of judicial review. A notice announcing the Agency's denial of the application will be published in the Federal Register.

(i) If the Agency proposes to approve an exemption, it will issue a notice in the Federal Register announcing this intent and invite public comments. If, after considering any timely comments received, the Agency approves an exemption, its decision will be published in the Federal Register. This notice will be considered a final Agency action for purposes of judicial review.

(j) The length of an exemption period will be specified by the agency when it approves the exemption. To extend an exemption period beyond the period stipulated by EPA, applicants must submit a new application to the Agency, following the application procedures described in this section. Applications may not be submitted prior to 15 months before the expiration of the exemption period, unless stated otherwise in the notice granting the exemption. Applications received between 15 months and 1 year before the end of the exemption period will be either granted or denied by the Agency before the end of the exemption period. Applications received after the date 1 year prior to the end of the exemption period will be either granted or denied by the Agency as soon as is feasible. Applicants may not continue the activity that is the subject of the renewal application after

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the date of the end of the exemption period.

[54 FR 29507, July 12, 1989; 54 FR 37531, Sept. 11, 1989, as amended at 54 FR 46898, Nov. 8, 1989; 59 FR 33210, June 28, 1994]

Sec. 763.175 Enforcement.

(a) Failure to comply with any provision of this subpart is a violation of section 15 of the Act (15 U.S.C. 2614).

(b) Failure or refusal to establish and maintain records, or to permit access to or copying of records as required by section 11 of the Act (15 U.S.C. 2610) is a violation of section 15 of the Act (15 U.S.C. 2614).

(c) Failure or refusal to permit entry or inspection as required by section 11 of the Act (15 U.S.C. 2610) is a violation of section 15 of the Act (15 U.S.C. 2614).

(d) Violators may be subject to the civil and criminal penalties in section 16 of the Act (15 U.S.C. 2615) for each violation.

(e) The Agency may seek to enjoin the manufacture, import, processing, or distribution in commerce of **asbestos**-containing products in violation of this subpart, or act to seize any **asbestos**-containing products manufactured, imported, processed, or distributed in commerce in violation of this subpart, or take any other actions under the authority of section 7 or 17 of the Act (15 U.S.C. 2606 or 2616) that are appropriate.

Sec. 763.176 Inspections.

The Agency will conduct inspections under section 11 of the Act (15 U.S.C. 2610) to ensure compliance with this subpart.

Sec. 763.178 Recordkeeping.

(a) Inventory. (1) Each person who is subject to the prohibitions imposed by Secs. 763.165 and 763.167 must perform an inventory of the stock-on-hand of each banned product as of the effective date of the ban for that product for the applicable activity.

(2) The inventory shall be in writing and shall include the type of product, the number of product units currently in the stock-on-hand of the person performing the inventory, and the location of the stock.

(3) Results of the inventory for a banned product must be maintained by the person for 3 years after the effective date of the Sec. 763.165 or Sec. 763.167 ban on the product.

(b) Records. (1) Each person whose activities are subject to the bans imposed by Secs. 763.165, 763.167, and 763.169 for a product must, between the effective date of the Sec. 763.165 or Sec. 763.167 ban on the product and the Sec. 763.169 ban on the product, keep records of all commercial transactions regarding the product, including the dates of purchases and sales and the quantities purchased or sold. These records must be maintained for 3 years after the effective date of the Sec. 763.169 ban for the product.

(2) Each person who is subject to the requirements of Sec. 763.171

must, for each product required to be labeled, maintain a copy of the label used in compliance with Sec. 763.171. These records must be maintained for 3 years after the effective date of the ban on distribution in commerce for the product for which the Sec. 763.171 requirements apply.

[54 FR 29507, July 12, 1989, as amended by 54 FR 46898, Nov. 8, 1989; 58 FR 34205, June 23, 1993]

Sec. 763.179 Confidential business information claims.

(a) Applicants for exemptions under Sec. 763.173 may assert a Confidential Business Information (CBI) claim for information in an exemption application or supplement submitted to the Agency under this subpart only if the claim is asserted in accordance with this section, and release of the information would reveal trade secrets or confidential commercial or financial information, as provided in section 14(a) of the Act. Information covered by a CBI claim will be treated in accordance with the procedures set forth in 40 CFR part 2, subpart B. The Agency will place all information not claimed as CBI in the manner described in this section in a public file without further notice to the applicant.

(b) Applicants may assert CBI claims only at the time they submit a completed exemption application and only in the specified manner. If no such claim accompanies the information when it is received by the Agency, the information may be made available to the public without further notice to the applicant. Submitters that claim

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information as business confidential must do so by writing the word ``Confidential'' at the top of the page on which the information appears and by underlining, circling, or placing brackets ([ ]) around the information claimed CBI.

(c) Applicants who assert a CBI claim for submitted information must provide the Agency with two copies of their exemption application. The first copy must be complete and contain all information being claimed as CBI. The second copy must contain only information not claimed as CBI. The Agency will place the second copy of the submission in a public file. Failure to furnish a second copy of the submission when information is claimed as CBI in the first copy will be considered a presumptive waiver of the claim of confidentiality. The Agency will notify the applicant by certified mail that a finding of a presumptive waiver of the claim of confidentiality has been made. The applicant has 30 days from the date of receipt of notification to submit the required second copy. Failure to submit the second copy will cause the Agency to place the first copy in a public file.

(d) Applicants must substantiate all claims of CBI at the time the applicant asserts the claim, i.e., when the exemption application or supplement is submitted, by responding to the questions in paragraph (e) of this section. Failure to provide substantiation of a claim at the time the applicant submits the application will result in a waiver of the CBI claim, and the information may be disclosed to the public without further notice to the applicant.

(e) Applicants who assert any CBI claims must substantiate all claims by providing detailed responses to the following:

(1) Is this information subject to a patent or patent application in the United States or elsewhere? If so, why is confidentiality necessary?

(2) For what period do you assert a claim of confidentiality? If the claim is to extend until a certain event or point in time, please

indicate that event or time period. Explain why such information should remain confidential until such point.

(3) Has the information that you are claiming as confidential been disclosed to persons outside of your company? Will it be disclosed to such persons in the future? If so, what restrictions, if any, apply to use or further disclosure of the information?

(4) Briefly describe measures taken by your company to guard against undesired disclosure of the information you are claiming as confidential to others.

(5) Does the information claimed as confidential appear or is it referred to in advertising or promotional materials for the product or the resulting end product, safety data sheets or other similar materials for the product or the resulting end product, professional or trade publications, or any other media available to the public or to your competitors? If you answered yes, indicate where the information appears.

(6) If the Agency disclosed the information you are claiming as confidential to the public, how difficult would it be for the competitor to enter the market for your product? Consider in your answer such constraints as capital and marketing cost, specialized technical expertise, or unusual processes.

(7) Has the Agency, another Federal agency, or a Federal court made any confidentiality determination regarding this information? If so, provide copies of such determinations.

(8) How would your company's competitive position be harmed if the Agency disclosed this information? Why should such harm be considered substantial? Describe the causal relationship between the disclosure and harm.

(9) In light of section 14(b) of TSCA, if you have claimed information from a health and safety study as confidential, do you assert that disclosure of this information would disclose a process used in the manufacturing or processing of a product or information unrelated to the effects of **asbestos** on human health and the environment? If your answer is yes, explain.

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Section Nine:
Exhibits

Exhibit #8
Air Laboratory Audit Checklist

DATE: ☐ **NVLAP LAB CODE:** ☐

NIST HANDBOOK 150-13 CHECKLIST

AIRBORNE ASBESTOS ANALYSIS

Instructions to the Assessor: This checklist addresses specific accreditation criteria prescribed in NIST Handbook 150-13, *Airborne Asbestos Analysis* (2006 edition). The numbering of this checklist generally follows the numbering of NIST Handbook 150-13 and contains requirements additional to those in the handbook.

- All items on this checklist shall be addressed.
- Place an “X” beside each item that represents a nonconformity (formerly called "deficiency"); or
  Place a “C” beside each item on which you are making a comment; or
  Place an "OK" beside each item that you observed or verified at the laboratory.
- Record the item number and your nonconformity explanation and/or comments on the comment sheet(s) at the end of this checklist.

3 Accreditation process

3.3 Proficiency testing

The laboratory shall participate in mandatory airborne asbestos proficiency testing when available, which includes (but is not limited to) the following:

_____ 3.3.1 the laboratory shall have written procedures for handling, analysis, and use of NIST proficiency testing materials;

_____ 3.3.2 the laboratory shall keep and use proficiency testing materials as in-house instructional and reference materials, unless otherwise directed;

_____ 3.3.3 all analysts (full- and part-time) shall participate in all proficiency testing rounds with analytical results filed in their personnel folders;

***NOTE**: All analysts need not participate in proficiency testing prior to returning the results to NVLAP, but all analysts shall participate without prior knowledge of the testing results at a later date.*

_____ 3.3.4 each analyst shall separately analyze, record and report test results;

_____ 3.3.5 a single result shall be reported back to NVLAP by the laboratory unless otherwise specified in the testing instructions;

_____ 3.3.6 procedures and calculations (if any) shall be documented as to how a single result was determined;

_____ 3.3.7 test results shall be used for interanalyst comparisons;

_____ 3.3.8 corrective actions shall be taken for interanalyst comparisons;

_____ 3.3.9 plans shall be developed and implemented for resolving problems and shall be documented;

_____ 3.3.10 test results, when applicable, shall be used in determining accuracy and precision for each analyst.

4 Management requirements for accreditation

4.1 Organization

_____ 4.1.1 The laboratory's quality assurance analyses shall represent at least 10 % of the total number of TEM asbestos analyses performed.

NOTE: *The value of 10 % is a minimum value that applies to a laboratory that has: 1) trained analysts, 2) its laboratory calibrations, contamination checks and other quality systems components statistically characterized and in a state-of-control, and 3) a high frequency of analyses. For laboratories not fitting these criteria, the quality assurance analyses must be a higher percentage of the total number of TEM asbestos analyses performed.*

_____ 4.1.2 Quality assurance analyses shall be performed regularly 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. A disproportionate number of analyses shall not be performed prior to internal or external audits. Quality assurance analyses shall not be postponed during periods of heavy workloads.

4.1.3 The laboratory shall summarize all of the quality assurance activities each month including:

_____ a) the total number of samples (or grid openings) analyzed;

_____ b) the total number of samples (or grid openings) that have been QC'd;

_____ c) contamination checks, problems, and corrective measures;

_____ d) the total number of interlaboratory and intermicroscope analyses (if applicable);

_____ e) calibrations;

_____ f) identification of any sample custody errors, such as mixing up samples, losing samples, etc.;

_____ g) nonconformity corrections.

4.1.4 The laboratory shall have the latest versions of the following documents available for reference:

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

_____ b) NIST Handbook 150-13, *NVLAP Airborne Asbestos Analysis;*

_____ c) the 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 AEM method for the determination of completion of response actions;

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

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

_____ f) AEM manufacturer's operation manual;

_____ g) multichannel analyzer manufacturer's operation manual.

4.1.5 The laboratory shall have references available and shall be proficient on the following topics; however, **the exact reference is not required:**

_____ a) **for verified asbestos analysis**, see

- E.B. Steel and J.A. Small, *Accuracy of Transmission Electron Microscopy for the Analysis of Asbestos in Ambient Environments*, Analytical Chemistry, Vol. 57, Issue 1, 1985, pp. 209-213;

- S. Turner and E.B. Steel, *Analysis of Transmission Electron Microscopy Analysis of Asbestos on Filters: Interlaboratory Study,* Analytical Chemistry, Vol. 63, Issue 9, 1991, pp. 868-872;

- S. Turner and E.B. Steel, NISTIR 5351, *Airborne Asbestos Method: Transmission Electron Microscopy – Version 2.0*, 1994;

_____ b) **for spot size measurement**, see

- D.B. Williams, *Practical Analytical Electron Microscopy in Materials Science*, Philips Electronics Instruments, Inc., Mahway, New Jersey, 1984, pp. 34-35 (for TEM or STEM mode);

- David B. Williams and C. Barry Carter, *Transmission Electron Microscopy: A Textbook for Materials Science*, 4 vol. set Plenum Press, New York, 1996 (Paperback);

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

- J.I. Goldstein, et al, *Scanning Electron Microscopy and X-ray Microanalysis*, Plenum Press, New York, 1981, p. 48 (for STEM mode);

_____ c) **for k-factor measurement**, see

- D.C. Joy, A.D. Romig, J.I. Goldstein, *Introduction to Analytical Electron Microscopy*, Plenum Press, New York, 1986; or
- D.B. Williams, *Practical Analytical Electron Microscopy in Materials Science*, Philips Electronics Instruments, Inc., Mahway, New Jersey, 1984;

_____ d) **for quality assurance**, see

J.K. Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, Michigan, 1987;

_____ e) **for statistical analysis**, see

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

_____ f) **for control charts**, see

Manual on Presentation of Data and Control Chart Analysis, Seventh Edition, ASTM, Philadelphia, 2002;

_____ g) reference data on the crystallography and chemical composition of minerals that analytically interfere with the regulated asbestos minerals.

4.2 Management system

_____ 4.2.1 If a laboratory is accredited as a sub-facility, it shall be technically dependent on the main facility (i.e., technical management and supervision shall be provided by the main facility).

_____ 4.2.2 Quality assurance activities of the sub-facility shall be directed by the main facility.

4.2.3 The nature, scope, and frequency of on-site quality assurance reviews by the main facility quality manager shall be:

_____ a) clearly defined in the quality manual;

_____ b) appropriate for the nature and scope of work performed by the sub-facility.

_____ 4.2.4 All permanent quality assurance and personnel records shall be retained at the sub-facility and copies provided to the main facility.

DATE: ☐ **NVLAP LAB CODE:** ☐

_____ 4.2.5 Quality assurance data from each sub-facility shall be compared each month to both the main facility's data and to data from other sub-facilities. Records of such comparisons shall be retained in quality assurance records, along with actions taken to evaluate and resolve differences.

_____ 4.2.6 Analysts at sub-facilities shall participate in NVLAP proficiency testing and records shall be maintained of individual results.

4.3 Document control

(See NIST Handbook 150 Checklist)

4.4 Review of requests, tenders and contracts

(See NIST Handbook 150 Checklist)

4.5 Subcontracting of tests and calibrations

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

NOTE: *A laboratory that subcontracts proficiency testing to another laboratory will be immediately suspended for not participating in the test round and risks revocation of its accreditation.*

4.6 Purchasing services and supplies

(See NIST Handbook 150 Checklist)

4.7 Service to the customer

(See NIST Handbook 150 Checklist)

4.8 Complaints

(See NIST Handbook 150 Checklist)

4.9 Control of nonconforming testing and/or calibration work

(See NIST Handbook 150 Checklist)

4.10 Improvement

(See NIST Handbook 150 Checklist)

4.11 Corrective action

(See NIST Handbook 150 Checklist)

4.12 Preventive action

(See NIST Handbook 150 Checklist)

4.13 Control of records

_____ 4.13.1 All records shall be retained for a minimum of three years and shall be stored in a logical fashion allowing easy retrieval.

_____ 4.13.2 The laboratory shall have documentation, either electronic backup or "paper" hard copy, to ensure survival of original data if computers are used for data retention.

4.13.3 The management system documentation shall contain standardized methods (forms) for recording the following:

_____ a) log-in of samples;

_____ b) criteria for acceptance or rejection;

_____ c) evaluation of quality of prepared grids;

_____ d) AEM sample analysis data.

4.13.4 The laboratory shall have records relating to:

_____ a) sample custody;

_____ b) contamination monitoring.

4.13.5 Records related to contamination shall 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;

_____ f) a summary of blank results in control chart or similar format.

4.13.6 All records related to quality assurance testing of staff and laboratory shall be retained including results of:

_____ a) analyses of reference materials;

_____ b) analysis of NIST proficiency testing materials;

______ c) verified analyses;

______ d) interlaboratory analyses;

______ e) intermicroscope analyses (if the laboratory uses more than one AEM for asbestos analysis);

______ f) repeat preparation and analysis of same filter by same analyst and by different analysts;

______ g) identification of mineral types;

______ h) evaluation of filter preparations.

4.13.7 Records related to the AEM analysis of a filter shall include:

______ a) general information;

______ operator (analyst must sign and date analysis sheet),

______ sample identification,

______ client identification,

______ date;

______ b) instrument used (if more than one available);

______ c) operating parameters of the instrument used including:

______ magnification,

______ accelerating voltage,

______ other, as needed to ensure alignment and calibration compliance;

______ d) filter- and grid-related information:

______ filter sampling data sheet as received with sample,

______ filter type,

______ area of grid squares analyzed,

______ number of grids prepared and their grid storage location,

______ evaluation of prepared grids and grid openings,

______ orientation of grid in AEM,

______ grids and grid squares analyzed;

_____ e) original data records include (for AHERA analysis):

_____ structure type (fiber, bundle, cluster, matrix),

_____ the number of fibers that are ≥ 5 micrometers and < 5 micrometers,

_____ the number of fibers that are ≥ 5 micrometers,

_____ classification of structures as chrysotile, amphibole (as grunerite (amosite), riebeckite (crocidolite), anthophyllite, actinolite, or tremolite), or nonasbestos,

_____ at a minimum, one recorded diffraction pattern for each species and one file copy of a spectrum,

_____ at a minimum, measurement results of electron diffraction for each structure (usually the first 4) identified as chrysotile, that caused the concentration of regulated asbestos minerals on the filter to reach or exceed 70 structures/mm^2,

_____ documentation of positive electron diffraction ***or*** EDXA for each chrysotile asbestos structure subsequent to the asbestos structure that caused the concentration on the filter to reach or exceed 70 structures/mm^2, and documentation of positive EDXA or measured zone axis diffraction pattern, for each amphibole structure subsequent to the asbestos structure that caused the concentration of asbestos on the filter to reach or exceed 70 structures/mm^2,

_____ at a minimum, documentation or measurement of results of EDXA and/or measurement of a zone axis electron diffraction pattern for each structure on the non-asbestos class that corresponds to a concentration of over 70 structures/mm^2,

_____ micrograph numbers or appropriate identification for the required one electron diffraction pattern for every five samples that contain asbestos and for any other patterns taken,

_____ interpretation of electron diffraction pattern to verify the identity of asbestos types,

_____ criteria used to classify particles as non-asbestos, that is the property or properties that differentiate it from regulated asbestos minerals;

NOTE: *For structures whose qualitative chemical composition is distinct from regulated asbestos minerals, 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 distinguishing features of the diffraction pattern is required.*

Measurement results shall include sufficient quantitative data (e.g., x-ray intensities or diffraction maxima d-spacing) to identify regulated asbestos minerals positively, 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.

_____ f) information related to report to client:

_____ concentration of asbestos in structures/mm^2 on filter and structures/cm^3 in sampled air,

_____ number of asbestos structures counted,

_____ types of asbestos.

_____ area analyzed,

_____ volume of air sampled.

_____ 4.13.8 A cumulative record of results from precision and accuracy testing shall be maintained and summarized at least monthly.

4.14 Internal audits

(See NIST Handbook 150 Checklist)

4.15 Management reviews

(See NIST Handbook 150 Checklist)

5 Technical requirements for accreditation

5.2 Personnel

_____ 5.2.1 Staff members shall be aware of both the extent and the limitation of their area of responsibility.

_____ 5.2.2 The laboratory shall have a written description of its training program that includes training with standards and blind testing, and NVLAP documentation, to determine competency and criteria for successful completion.

_____ 5.2.3 Analysts and technical supervisors shall participate in an appropriate form of continuing education, such as formal coursework, in-house education, and scientific or technical meetings, and have access to journals that describe advances in the field of electron microscopy and/or asbestos analysis.

______ 5.2.4 The technical supervisor(s) shall be qualified to conduct AEM studies, apply AEM to crystalline materials and shall be proficient in the field of asbestos analysis including procedures for sample handling, preparation, analysis, storage, disposal, and contamination monitoring.

5.2.5 AEM analysts shall be trained and be proficient in:

______ a) AEM use, calibration, alignment, electron micrography (or functional equivalent);

______ b) EDXA, x-ray collection and interpretation including the formation of EDX patterns, recognition of artifacts and abnormal features in spectra resulting from detector problems, contamination or detector-sample geometry;

______ c) selected area electron diffraction measurement and interpretation including the formation of SAED patterns, determination of d-spacings, and Miller indices of individual diffraction spots and the corresponding zone axis;

d) asbestos counting methods including:

______ counting rules for simple and complex structures;

______ grid and grid square selection (nonadjacent, random);

______ x-y stage translation and parallel traverses;

______ stage positioning and repositioning;

e) asbestos identification including:

______ morphology criteria;

______ crystallographic criteria through selected area electron diffraction analysis;

______ chemical composition criteria through EDXA;

______ f) differentiation between regulated asbestos minerals and other minerals that resemble the regulated asbestos minerals;

______ g) determination of the concentration of fibers on a filter sample;

______ h) verified asbestos analysis;

______ i) recognition of acceptable and unacceptable sample preparations;

______ j) recognition of sample and instrumental artifacts;

______ k) AHERA clearance protocols.

5.2.6 The accuracy of asbestos identification and counting of each AEM analyst shall be evaluated by:

_____ a) analyses of reference materials prepared in-house and purchased;

_____ b) analyses of NIST proficiency testing samples; and

_____ c) verified analyses.

NOTE: *Additional quality assurance information may be gained by determining the precision of AEM analysts by repeat analysis of grid squares.*

5.2.7 Verified asbestos analyses shall be performed routinely by the laboratory with sufficient frequency and on sufficient types of samples to determine each operator’s initial and continuing performance that the following conditions are satisfied:

_____ a) samples having approximately 6–40 structures/grid opening shall be used to achieve statistically significant information on new analysts;

_____ b) after initial training, a variety of asbestos loadings, including routine AHERA samples, shall be used to validate analysts’ results. Samples shall include loadings seen in typical AHERA samples up to 6–40 structures/grid opening. At least 20 % of verified analyses shall be performed on samples with 6-40 structures/grid opening loadings and at least 5 grid openings with 6-40 structures/grid opening shall be counted annually;

_____ c) filter blanks, unless known to be contaminated, shall not be used for verified counting;

_____ d) during training, all counts used in reports shall be verified until verified status is attained;

_____ e) after verified status is attained, the frequency of verified analysis shall be at least 1 per 100 grid opening analyses.

NOTE: *Labs may find it advantageous to use as many as four operators on one analysis to characterize initial operator performance, if an analyst with verified status is not available. An analyst that obtains verified status shall have an average accuracy ≤ 80 % of true positives, ≤ 20% false negatives, and ≤ 10 % false positives on both standard and field samples.*

_____ 5.2.8 The laboratory shall have a person responsible for tracking and storing samples (a laboratory sample coordinator).

_____ 5.2.9 The laboratory shall be organized so that staff members are not subject to undue pressure or inducement that might influence their judgment or results of their work. The laboratory is able to demonstrate that the sample work required for each analysis is consistent with accurate and precise analytical measurements.

5.3 Accommodation and environmental conditions

5.3.1 The following facilities shall be available:

_____ a) clean room or clean areas for sample preparation and handling separate from bulk asbestos;

_____ b) electron microscopy facility;

_____ c) room or area for filter and grid storage separate from bulk asbestos.

5.3.2 The following shall be available in the clean room or clean area:

_____ a) class 100 (or cleaner) HEPA-filtered air under positive pressure, which shall be maintained except for maintenance;

_____ b) exhaust hood for safe use of filter dissolution reagents.

5.3.3 Safe working conditions shall be maintained, including:

_____ a) safe handling of asbestos;

_____ b) safe handling and storage of filter-dissolving reagents such as chloroform, dimethyl formamide, acetone, acetic acid, etc.

5.3.4 Management system documentation shall contain:

_____ a) procedures for the prevention, monitoring, and control of contamination of filters and grids;

_____ b) procedure (or flow chart) for the systematic checking for possible sources of contamination if contamination is detected. This includes checking all areas, instrumentation and materials used in the preparation and analysis of air filter samples.

5.3.5 To minimize the possibility of contamination of samples, the following shall be performed:

_____ a) all personnel shall be instructed in contamination prevention;

_____ b) personnel, instrumentation and materials used for the preparation of bulk materials that potentially contain asbestos shall be kept separate from areas used for air filter preparation, handling or analysis;

_____ c) all reagents shall be checked for asbestos contamination prior to use in sample preparation, or purchased from previously tested sources in previously tested grades.

NOTE: *Personnel who have worked with bulk samples should not subsequently be allowed to work with air filter samples on the same day. It is acceptable, however, for personnel to work on bulk samples after having worked with air samples.*

NOTE: *The next four items concern the use of blank materials for contamination monitoring. Definitions of filter lot, sealed, field and laboratory blanks are given in 1.5 of NIST Handbook 150-13. Other blanks should be used as needed to determine and correct sources of contamination, including blanks for AEM specimen holders, evaporators, Jaffe wick, low temperature asher, laboratory air samples, etc.*

_____ 5.3.6 A laboratory blank filter shall be present in the clean room or clean area during sample preparation and after cleaning or servicing of the clean room or clean area. The laboratory blanks shall be obtained from a filter lot that has been shown not to be contaminated.

5.3.7 The maximum allowed contamination levels for filter lot, sealed, field and laboratory blanks shall be:

_____ a) for polycarbonate filters — a cumulative average level of 18 structures per mm^2;

_____ b) for mixed cellulose ester — a cumulative average of < 5; single < 15.

_____ c) a single preparation level of 53 structures per mm^2.

5.3.8 Preparation of nominally blank filters shall be done:

_____ a) on a minimum of one laboratory blank per sample set or 10 % of samples (whichever is greater);

_____ b) on a laboratory blank after cleaning or servicing the clean room or clean area;

_____ c) on the field and sealed blanks with each series of samples (if these blanks are not identified and known to the laboratory, all filter samples are prepared with the series).

NOTE: *The laboratory shall properly record and archive prepared grids (even if not analyzed).*

5.3.9 Analysis of nominally blank filters shall be done:

_____ a) polycarbonate filters — on a minimum of one laboratory blank per 25 filter analyses;

_____ b) mixed cellulose ester — on a minimum of one laboratory blank per 100 filter analyses;

_____ c) on the laboratory blank when the average count for the full set of filters exceeds 70 structures/mm^2;

_____ d) on the field and sealed blank when the full indoor/outdoor analysis is performed.

NOTE: *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.*

The laboratory blank analyses may be counted towards the required 10 % quality assurance analyses. The field and sealed blank analyses; however, may not be counted towards this requirement.

_____ 5.3.10 When contamination above acceptable levels is found, analyses for AHERA clearance shall be discontinued until the cause is found and corrected or until data shows the contamination problem no longer exists.

5.4 Test and calibration methods and method validation

(See NIST Handbook 150 Checklist)

5.5 Equipment

5.5.1 The following sample preparation equipment or equivalent shall be available in the clean room or area:

_____ a) condensation washer and/or Jaffe wick with the appropriate reagents and supplies;

_____ b) filter preparation materials (e.g., scalpel, microscope slides, tweezers, etc.);

_____ c) indexed 200-mesh TEM grids—also referred to as finder grids (only grids with unique identifiable grid openings may be used; grids with only a symmetrical central marking do ***not*** qualify as finder grids);

_____ d) other materials as needed.

5.5.2 The laboratory shall have a low temperature plasma asher which:

_____ a) is supplied with oxygen;

_____ b) allows for control of speed of evaluation and venting to minimize disturbance of particles on filter surface;

_____ c) is not used for bulk samples (asbestos or other).

5.5.3 The laboratory shall have:

_____ a) a carbon evaporator which attains a vacuum of 13 Pa (10^{-4} torr) or lower and has controlled venting to atmospheric pressure;

______ b) spectrochemically pure carbon rods;

______ c) a carbon rod sharpener;

______ d) gold or aluminum wire for evaporation (or have sputter coater with gold target).

5.5.4 The laboratory shall have an electron microscope, which has the following under routine asbestos analysis conditions:

______ a) capability of operation at a voltage between 80 keV-120 keV;

______ b) capability of producing an electron diffraction pattern of a single fibril of all regulated asbestos minerals;

______ c) capability of displaying and resolving hollow tube of chrysotile;

______ d) capability of precise fiber length (at 0.5 µm) 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 perpendicular lines);

______ 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) an imaging system for recording brightfield images and electron diffraction patterns on electron micrographs or on other suitable media.

NOTE: *It is strongly recommended that the laboratory possess a holder capable of obtaining zone axis diffraction patterns (either a double-tilt or rotation-tilt holder).*

5.5.5 The laboratory shall be 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 1000 x to 100 000 x in brightfield imaging mode;

_____ e) a range of diffraction camera lengths that enable accurate diffraction pattern measurement (approximately 20 cm to 80 cm).

_____ 5.5.6 An EDXA system shall be interfaced to all electron microscopes used for asbestos analysis.

5.5.7 The EDXA system (detector and multichannel analyzer), under routine analysis conditions, shall meet the following specifications:

_____ a) 175 eV or better resolution at Mn Kα peak;

_____ b) proven statistically significant detection of Na Kα peak in standard crocidolite or equivalent;

_____ c) capable of obtaining statistically significant Mg and Si peaks from a single fibril of chrysotile;

_____ d) consistent relative sensitivity factors over large areas of the specimen grid (see 5.6.1.12).

NOTE: *(1) A low background holder may be necessary to meet these requirements, (2) for item 5.5.7b, 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 Na is measured above the Cu L-line background.*

5.5.8 The multichannel analyzer shall have the following:

_____ a) software capable of obtaining background corrected peak intensities or integrals for Na, Mg, Al, Si, Ca, Fe and other elements as needed;

_____ b) capability of accumulation and display of an x-ray spectrum (minimum 0.7 keV-10 keV);

_____ c) capability of making a hard copy of an x-ray spectrum.

5.6 Measurement traceability

5.6.1 General

NOTE: *Control charts shall be constructed to show calibration values vs. time, the magnitude of their variation, and the allowable limits of variation. The magnitude of variation specified for many calibrations in this program is defined as 2 s (s is the estimated standard deviation of a set of measurements). Initially, many (15-30) calibrations should be performed in a few months' time to establish a baseline for variation in the measurements. If the variation is within specified limits and the accuracy is acceptable, the frequency of the calibration can be reduced. In general, the majority of calibrations should have been done within 3 months prior to analyses performed for clients.*

All calibrations shall 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.) and with the microscope aligned. Tilting the viewing screen and specimen grid during fiber measurement, or the viewing screen during diffraction measurement is not recommended. Laboratories using tilts must demonstrate the required measurement accuracy and precision for all fiber and diffraction maxima orientations.*

_____ 5.6.1.1 The laboratory shall have specific procedures in its management system documentation for the development and use of control charts, including the algorithms for calculating warning and control limits.

_____ 5.6.1.2 The laboratory shall use control charts to summarize all calibration data.

5.6.1.3 The magnification of the electron microscope shall be calibrated:

_____ a) using an optical diffraction grating replica (the variation in the calibration measurements, i.e., two times the standard error (2 *s*) is < 5 % of the mean calibration value);

_____ b) for magnifications commonly used for asbestos analysis and for any other magnification used for measurement (e.g., the magnification used to measure grid square size);

_____ c) on all measurements systems applied in the laboratory for asbestos analysis such as the phosphor viewing screen, film, monitor and/or image analysis system.

5.6.1.4 The accuracy and precision of measurements at 0.5 μm shall be determined by:

_____ a) calibration of the measuring system(s) (on screen, film, monitor, and/or image analysis system) at 0.5 μm;

_____ b) repeat analysis by the same and different analysts of asbestos fibers approximately 0.5 μm in length. (This data may be derived in part from verified analysis data for fibers close to 0.5 micrometers in length.)

5.6.1.5 The diffraction camera constant shall be calibrated:

_____ a) using an evaporated gold or aluminum film (the variation in the calibration measurements (2 *s*) is < 5 % of the mean calibration value);

_____ b) for the camera lengths commonly used for asbestos analysis;

_____ c) under the conditions used for asbestos analysis;

_____ d) on all measurement systems including the TEM screen, film, monitor, image analysis system and/or any other system as applied in the laboratory for asbestos analysis.

NOTE: *A minimum of three measurements at 45° angles is required on the innermost ring. These measurements will allow detection of deviations of a ring from a circle. Measurements of at least two of the outer rings must be made to monitor for radial distortions and to ensure that an error in measurement of the inner ring did not occur. If significant distortion is found, more measurements are needed for better characterization.*

_____ 5.6.1.6 The beam dose shall be calibrated so that beam damage to chrysotile is minimized—specifically so that an electron diffraction pattern from a single fibril ≥ 1 μm in length from a NIST SRM chrysotile sample is stable in the electron beam for at least 15 seconds.

_____ 5.6.1.7 The laboratory shall have recorded the setting of the electron microscope (condenser aperture, spot size, etc.) that allows for the stability of chrysotile as specified in item 5.6.1.6 above. This setting shall be used as the standard operation procedure for routine analyses of possible chrysotile structures. For microscopes that cannot scan at electron fluxes which limit the damage to chrysotile, a procedure how to limit damage (e.g., immediately defocus the condenser) is required.

5.6.1.8 The spot size of the electron beam used for well resolved x-ray microanalysis shall be determined and the:

_____ a) the average spot size for a properly stigmated beam is ≤ 250 nm;

_____ b) the variation in diameter measurements (2 *s*) is < 25 % of the mean value.

5.6.1.9 The EDXA system shall be shown through calibration data to have:

_____ a) a resolution (full-width, half-maximum) for Mn Kα that is < 175 eV;

_____ b) a value for the sum of the resolution and the variation (2 *s*) that is < 180 eV for ≤ 5 measurements taken on a given day.

_____ 5.6.1.10 The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

5.6.1.11 The relative sensitivity (k-factors) factors relative to Si for elements found in asbestos (Na, Mg, Al, Si, Ca, Fe) shall be determined so that:

_____ a) the k-factors are determined to a precision (2 *s*) within 10 % relative to the mean value obtained for Mg, Al, Si, Fe, and within 20 % relative to the mean value obtained for Na;

_____ b) the k-factor relative to Si for Na is between 1.0 and 4.0, for Mg and Fe is between 1.0 and 2.0, and for Al and Ca is between 1.0 and 1.75;

_____ c) the k-factor for Mg relative to Fe on SRM 2063(a) or other standard traceable to NIST is 1.5 or less.

NOTE: *SRM 2063 or SRM 2063a can be used for the determination of k-factors for Mg, Si, Ca and Fe. The laboratory must obtain its own chemically characterized materials for determining the Na and Al k-factors. Examples include albite for Na k-factor determination and biotite or albite for Al k-factor determination. Na k-factors are sensitive to electron beam does (current and time). It is suggested that small particles (≤ 0.1 µm in size) be used for Na k-factor determination to minimize the effect of Na migration.*

_____ 5.6.1.12 The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas avoided in asbestos analysis.

NOTE: *X-rays can be absorbed due to the relative position of the area of interest, the grid bars, specimen holder and x-ray detector and give an abnormal spectra (for an example of an abnormal spectra see S. Turned, E.B. Steel, S.S. Doorn, and S.B. Burris, “Proficiency Tests for the NIST Airborne Asbestos Program – 1991,” NISTIR 5432). The laboratory should use a standard material (SRM 2063 is recommended) to map out the spectra obtained over the grid area and to thereby determine the regions that should be avoided in routine analysis.*

_____ 5.6.1.13 The low temperature asher shall be calibrated by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (mce) filters.

NOTE: *The AHERA method specifies that a mixed-cellulose-ester filter is to be ashed by 10 %. However, if ashing by this amount generates a texture in the replica that affects structure counting, it is permissible to etch by less than this amount.*

5.6.1.14 The determination of the quality of sample preparations shall be calibrated or the laboratory shall have the following documentation available:

_____ a) images and samples showing good preparations and examples of the types of problems that occur in poor preparations (readily available to analysts);

_____ b) a record of repeat evaluations of images and samples by the same and different analysts. (This data may be derived in part from sample preparation evaluations done in the course of verified analysis.)

_____ 5.6.1.15 The magnification of the grid opening measurements system shall be calibrated using an appropriate standard. The variation in the calibration measurements (2 *s*) is < 5 % of the mean calibration value.

_____ 5.6.1.16 Trained AEM analysts shall have an average accuracy ≥ 80 % of true positives, ≤ 20 % false negatives, and ≤ 10 % false positives (these data are reported on a structures per grid square basis).

_____ 5.6.1.17 The laboratory and AEM analysts shall obtain mean analytical results on SRM 1876b so that trimmed mean values fall within 80 % of the lower limit and 110 % of the upper limit of the 95 % confidence limits as published on the certificate (these limits are derived from the allowable false positives and false negatives given in the previous item). The SRM shall be analyzed a minimum of once a year by each AEM analyst.

_____ 5.6.1.18 The laboratory shall have documentation demonstrating that AEM analysts correctly classify at least 90 % of both bundles and single fibrils of asbestos structures ≥ 1 µm in length in known standard materials traceable to NIST (such as the bulk asbestos SRM 1866).

_____ 5.6.1.19 Interlaboratory analyses shall be performed to detect laboratory bias. The frequency of interlaboratory verified analyses shall correspond to a minimum of 1 of 200 grid square analyses for clients.

_____ 5.6.1.20 If more than one AEM is used for asbestos analysis, intermicroscope analyses shall be performed to detect instrument bias.

_____ 5.6.1.21 The sampling precision shall be determined by repeat preparation and analysis of the same filter by the same and different analysts.

_____ 5.6.1.22 Required calibrations shall be performed correctly and on a frequent enough basis to ensure accurate results.

5.6.2 Calibration

_____ 5.6.2.1 The laboratory shall use 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, and any NIST or U.S. Environmental Protection Agency clarifications, modifications, or updates to the TEM method for the analysis of asbestos.

_____ 5.6.2.2 Management system documentation shall detail the AEM method as it is applied in the laboratory. (A simple copy of the AHERA method is not sufficient). If departures are made from the method, the laboratory shall have written procedures detailing how the analyses are conducted.

5.6.2.3 The laboratory shall have written procedures for:

_____ a) preparation of mixed-cellulose-ester filters, including techniques for collapsing, etching, carbon coating and dissolution of filters;

_____ b) preparation of polycarbonate filters, including techniques for carbon coating and dissolution of filters;

_____ c) determination of the number of grid squares and grid area to be analyzed per sample.

5.6.2.4 Laboratory personnel shall:

_____ a) prepare and store at least three grids per filter;

_____ b) analyze approximately half of the predetermined sample area to be analyzed on one grid and the remaining half on a second grid preparation.

5.6.2.5 The laboratory shall have written procedures for the evaluation of the quality of prepared grids. The criteria for acceptance include:

_____ a) the percentage of grid openings covered by the replica section (coherent or noncoherent) is greater than approximately 50 %;

b) the percentage of grid openings covered by the replica section that:

_____ are intact is greater than approximately 50 %;

_____ have undissolved filter that is less than approximately 50 %;

_____ have overlapping or folded replica that is less than approximately 50 %.

_____ c) 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.

_____ 5.6.2.6 The laboratory shall have written procedures for the determination of the area of grid squares.

NOTE: *The AHERA method requires that either 1) the area of the grid square analyzed be determined, or 2) the average area of grid squares in a grid lot of 1000 be determined by measurement of 20 grid squares on each of 20 grids. If premeasured grids are purchased, the laboratory should confirm the measurements as a quality assurance procedure. Initially, many grid squares should be remeasured. If the values are in agreement with those given with the grids, then the number of remeasurements can be reduced to approximately 5 % of those required by the AHERA method. Premeasured grids must have a report of analysis, which gives the mean grid opening area, the number of grids and openings measured, the standard deviation of the opening area, and the method of analysis.*

5.6.2.7 The laboratory shall have written procedures for operation of the AEM for asbestos analysis including:

_____ a) method for alignment of the electron microscope so that the electron beam travels down the optic center of the column (this includes alignment of the electron gun, apertures, and tilt as described in the manufacturer’s and laboratory’s operating manual);

b) standard operating conditions of the AEM

_____ i) voltage (between 80 keV-120 keV)

_____ ii) microscope magnification (15 000 x –20 000 x for analysis).

_____ 5.6.2.8 There shall be documentation to show that the quality of alignment of the electron microscope is checked daily or prior to each use for analyses and calibrations. The alignment is checked by, at minimum, changing the magnification, spot size, and image focus and by checking the stigmation of the electron beam. The AEM analyst aligns the electron microscope if the instrument does not meet the laboratory's alignment criteria as stated in the quality manual. (Note: Alternatively, the AEM can be aligned daily or prior to each use).

5.6.2.9 The laboratory shall have written procedures for examining a grid square and for counting and analyzing particles (a detailed description is necessary—a copy of the EPA method is not sufficient) including:

_____ a) method for recording grid orientation in the microscope;

_____ b) particle loading acceptance criteria (> 10 % by area particulate loading or uneven particle loading is rejected, see 5.6.2.5);

_____ c) unique grid and grid square labeling system (indexed grids);

_____ d) 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 the other translator is moved for analysis, then the stage is returned to the original traverse position before continuation of the traverse.*

_____ e) recording rules;

_____ f) structure counting rules;

_____ g) determination of whether a sample set passes or fails AHERA clearance if required by the client.

5.6.2.10 The laboratory shall have management system documentation which contains criteria for:

_____ a) identification of electron diffraction patterns of regulated asbestos minerals and of nonasbestos minerals, including those that closely resemble regulated asbestos minerals;

_____ b) identification of EDXA spectra of regulated asbestos minerals and of nonasbestos patterns, including those that closely resemble regulated asbestos minerals;

_____ c) differentiating asbestos minerals from at least the following phases; the pyroxenes, hornblende, wollastonite, halloysite, palygorskite, sepiolite, antigorite, lizardite, talc, and vermiculite. The minimum criteria for differentiation must be presented.

_____ 5.6.2.11 AEM analysts shall record sufficient information for each analysis so that a verified analysis can subsequently be performed.

NOTE: *Information sufficient for performing a verified analysis includes the orientation of the grid at the analysis magnification, a sketch (or image) for each structure and the size of each structure (the recording of the location of the structure is also of use). Recording this information will allow for random quality assurance checks of any analysis and removes the bias that can occur when verification is done with the analysts' foreknowledge. The laboratory may want to refer to E.S. Windsor, S. Turner and E.B. Steel, NISTIR 5358, in which a recording form suitable for verification is described.*

NOTE: See Appendix A of this checklist.

_____ 5.6.2.12 AEM analysts shall record a selected area electron diffraction pattern of one asbestos structure on from every five samples that contain asbestos. The identification of diffraction patterns shall be verified by a qualified individual. It shall be shown that AEM analysts are correct 80 % of the time in identification of recorded diffraction patterns.

_____ 5.6.2.13 AEM analysts shall record an EDS spectrum from one asbestos structure of each type of amphibole asbestos from every set of samples that contain asbestos. The identification of EDS spectra shall be verified by a qualified individual. It is shown that the AEM analysis is correct 80 % of the time in identification of EDS spectra.

_____ 5.6.2.14 The laboratory shall have written procedures for acquiring, recording, indexing, and interpreting SAED patterns, including Miller indices of diffraction spots and, for amphiboles, zone axis.

_____ 5.6.2.15 The laboratory shall have written procedures for verifying report calculations.

5.6.3 Reference standards

5.6.3.1 The following standards and any associated certificates shall be on hand:

_____ a) materials with a certified value for the loading of asbestos on filters, such as NIST SRM 1876b, if available;

_____ b) materials that are characterized as asbestos for training and analyst evaluation such as NIST SRMs 1866 and 1867 or NIST-traceable standard, if available;

_____ c) calibration material(s) for the x-ray system such as SRM 2063 or NIST-traceable standard, if available;

_____ d) standard optical grating replica or calibrated polystyrene spheres for magnification calibration;

_____ e) gold or aluminum film material for electron diffraction calibration.

NOTE: *(1) The laboratory must use SRM 1876b for calibration and not SRMs 1876 or 1876a. SRM 1876 and 1876a were certified using sets of counting rules that are no longer in use. (2) SRMs 1866 and 1867 contain bulk asbestos and, therefore, precautions need to be taken against contaminating the filter preparation area and AEM with these specimens. (3) The laboratory has the primary responsibility for developing or obtaining a set of standards useful for checking the identification, analysis and concentration of asbestos on filters. For example, internal standards can be drawn from samples received by the laboratory or developed by the laboratory through water filtration of asbestos mixtures or by other methods. The samples then must be well-characterized by the laboratory for use as standards. NVLAP proficiency testing samples do not qualify as NIST-traceable standards.*

5.7 Sampling

(See NIST Handbook 150 Checklist)

5.8 Handling of test and calibration items

5.8.1 The log-in system shall include documentation of:

_____ a) the date of receipt;

_____ b) identity of the client;

_____ c) unique identification for sample;

_____ d) air volume pulled through sample;

_____ e) filter pore size;

_____ f) condition of the samples; and

_____ g) acceptance or rejection of the samples.

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

NOTE: *Examples of rejection criteria include: insufficient sampling documentation, bulk samples included with air filter samples, filter cassettes open, filters overloaded with particulate, uneven particle loading, sampling parameters not meeting AHERA sampling criteria, filters not uniquely identified, filters of incorrect pore size, tampering with cassettes evident, sample that laboratory is not capable of preparing properly, etc.*

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

_____ a) location of sample;

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

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

5.8.4 The laboratory shall:

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

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

_____ c) store the filters and grids in a logical fashion so that specified samples can be retrieved within one working day.

5.9 Assuring the quality of test and calibration results

(See NIST Handbook 150 Checklist)

5.10 Reporting the results

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

_____ a) area of filter analyzed;

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

_____ c) analytical sensitivity used for the analysis;

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

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

_____ f) statement of analytical uncertainty, including 95 % confidence limits on the reported concentration and laboratory and analyst accuracy and precision;

_____ g) micrograph number of any recorded diffraction patterns;

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

_____ i) descriptions of any departures from the test method.

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

______ a) calculation formulas;

______ b) all calculation variables and constants;

______ c) all calculation results.

APPENDIX A

Verified Asbestos Analysis

A.1 Verified analysis

Verified asbestos analysis is currently the best way to compare results among analysts and to check for the accuracy of an analyst on an unknown sample (see Steel, E. B. and J. A. Small, in clause 1.4). It consists of multiple operators independently analyzing a grid square, comparing the results, and 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 several operators in a verified analysis of a grid square to characterize initial operator performance if an analyst with verified status is not available (to achieve verified status an AEM analyst shall attain an average accuracy of ≥ 80 % true positives, ≤ 20 % false negatives, and ≤ 10 % false positives on verified analyses). The number of analysts who can analyze a grid square is limited by beam damage to the particles (four operators is typically the maximum number of analysts that can be used). Multiple analysts from different laboratories can be used if the initial orientation of the grid and grid opening is recorded and made known to subsequent laboratories.

One of the most common causes of false negatives in an AEM analysis of asbestos occurs when the operator fails to find or observe a fiber or structure. This is commonly due to the operator missing a whole or partial traverse of the grid square. A source of both false negatives and false positives is the individual operator's interpretation of asbestos structure counting rules as it is applied to complex structures. These types of errors can be uncovered and corrected by the use of verified analyses. As analysts within a laboratory count structures in a more uniform manner, the imprecision (due to the analysts) within the laboratory will decrease.

Verified asbestos analyses shall be performed by the laboratory on a routine basis and on sufficient types of samples to determine each operator's initial and continuing performance. Samples having approximately 6-40 structures/grid opening shall be used to achieve statistically significant information on new analysts. During training, all counts used in reports shall be verified until verified status is attained. After initial training, a variety of asbestos loadings, including routine AHERA samples, shall be used to validate analysts' results. At least 20 % of verified analyses shall be performed on samples with 6-40 structures/grid opening loadings and at least 5 grid openings with 6-40 structures/grid opening shall be verified annually. Filter blanks, unless known to be contaminated, shall be rarely used for verified counting. After verified status is attained, the frequency of verified analysis shall be at least 1 per 100 grid opening analyses.

A.2 NISTIR 5351

It is suggested that TEM laboratories follow NISTIR 5351, *Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0.* (Available from NVLAP.)

APPENDIX B

TEM Grid Square Overloading Criterion

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B.1 Introduction

When grids are prepared for TEM asbestos analysis, some of the grid squares may be observed to be heavily loaded with particulate matter. Overloaded squares are not to be analyzed, so there must be a criterion to define which squares are overloaded and must be rejected. In both the NVLAP TEM NIST Handbook 150-13 Checklist and the Asbestos Hazard Emergency Response Act (AHERA), the overload criterion is stated as a percentage of the grid square that is covered with particulate matter, but there is a discrepancy between the criteria stated in the NVLAP 150-13 Checklist and the AHERA. Item 5.6.2.9 b) in the Handbook 150-13 Checklist required grid square with > 10 % by area particulate loading to be rejected for analysis, while the AHERA puts the rejection criterion at > 25 % by area particulate loading.

This has led to some disagreement among TEM laboratories, their clients, and NVLAP assessors regarding the overload criterion; is the NVLAP requirement 10 % or 25 %?

______

B.2 NVLAP requirement

The NVLAP requirement is 10 % coverage, just as written in the NIST 150-13 Checklist, Item 5.6.2.9. Item 5.6.2.9 requires laboratories to have written procedures for examining a grid square and for counting and analyzing particles. Part b) under 5.6.2.9 states "particle loading acceptance criterion (> 10 % by area particulate loading or uneven particle loading is rejected)." Thus, for NVLAP TEM laboratories, the 10 % criterion is to be written into their procedures, and the laboratories will be assessed to the 10 % requirement.

The NIST 150-13 Checklist requirements were written after careful study of AHERA and examination of numerous prepared grids. In the case of the criterion for acceptance/rejection of loaded grid squares, the Technical Experts at NIST decided 25 % or greater overloading coverage would allow asbestos structures to be obscured, so a lower coverage of 10 % was adopted as the NVLAP requirement to prevent the underestimation of asbestos structure concentrations during routine analysis of airborne asbestos samples.

DATE: ☐ **NVLAP LAB CODE:** ☐

COMMENTS AND NONCONFORMITIES

Instructions to the Assessor: Use this sheet to document comments and nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a "C" and nonconformities with an "X." If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	Comments and/or Nonconformities

Table 5.1 (Cont.) - Demonstration of Capability Acceptance Criteria

AIHA-LAP, LLC METHODS		
Certified Method	DOC Requirement	Control Limits/ Acceptance Criteria*
SW3050B / N7082 (Lead Paint)	IDOC: 4 sets of 5 Ref CDOC: Batch QC or PT	75% within 80-120%Rec LCS Control Limits, MDLs or PT Acceptance Criteria
SW3050B / 7420 (Lead in Soil)	IDOC: 4 sets of 5 Ref CDOC: Batch QC or PT	75% within 80-120%Rec LCS Control Limits, MDLs or PT Acceptance Criteria
SW3050B / 7000B (Lead in Soil)	IDOC: 4 sets of 5 Ref CDOC: Batch QC or PT	75% within 80-120%Rec LCS Control Limits, MDLs or PT Acceptance Criteria
N7082 (Lead in Dust Wipe)	IDOC: 4 sets of 5 Ref CDOC: Batch QC or PT	75% within 80-120%Rec LCS Control Limits, MDLs or PT Acceptance Criteria
N7303 (Lead in Air)	IDOC: 4 sets of 5 Ref CDOC: Batch QC or PT	75% within 80-120%Rec LCS Control Limits, MDLs or PT Acceptance Criteria
N7400 (Asbestos PCM)	PT Samples	PT Acceptance Criteria
Fungal Air Direct Exam (Micro)	PT Samples	PT Acceptance Criteria
Fungal Bulk Direct Exam (Micro)	PT Samples	PT Acceptance Criteria
Fungal Surface Direct Exam (Micro)	PT Samples	PT Acceptance Criteria

5.7.10.4 The large number of analytes in multi-element analyses presents a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined. Should this occur, re-analyze only the failed analytes, following the procedures discussed in this section.

5.7.10.5 When one or more of the analytes tested fails at least one of the acceptance criteria, the analyst must proceed according to the out-of-control procedures discussed in Section 5.8.

5.7.10.6 Due to the nature of several test methods, IDOCs cannot be performed. These tests represent methods where samples of known concentrations cannot be prepared in the laboratory. Tests that are included in this category are EPA 110.2, 160.3, 160.4, 160.5, 150.1, 9040, 9045, 1010, SM 2340B, SM2340G, SM9223, and SM9222. To complete IDOCs for these tests, the analyst(s) must satisfactorily pass available PE samples for all appropriate matrices.

5.7.10.7 Analyst Demonstration of Capability and training includes the following:

Quality Assurance Manual Training (annually)
Legal & Ethical Training (annually)
SOP Training (initially and as updated)
ICNs associated with the SOPs (initially and as updated)
Demonstration of Capability (program specific)
Procedure and Checklist Training (initially and as updated)

Individuals are authorized to perform analysis when these documents have been completed and signed by the individual(s) and referenced managers.

5.7.10.8 AIHA-LAP, LLC Training Requirements
AIHA-LAP, LLC Technician/Analyst Training Requirements. All technicians and analysts must complete training and demonstrate proficiency prior to analysis of any ELLAP or IHLAP program samples. The training and proficiency demonstrations must meet the requirements specified in the AIHA-LAP, LLC LQAP Policy Document, Modules 2A, 2B and 2C and are described in Section 1.2 and 1.3 below.

5.7.10.8.1 ELLAP Specific Technician/Analyst Training Requirements:

5.7.10.8.1.1 Initial demonstration of capability.

Each technician/analyst must complete at least 20 days work/training in the prep and / or metals analysis lab using technologies/instrumentation similar to that to be used for ELLAP samples under the direct supervision of an ELLAP trained technician / analyst prior to unsupervised prep / analysis of ELLAP regulated client samples.

Each analyst/technician must read, understand & agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form. Each technician / analyst must prep and/or analyze as appropriate at least 2 blind reference material test samples (concentration unknown to the technician/analyst). These samples may be AIHA-LAP, LLC provided PT samples or laboratory prepared Certified Reference Material of the appropriate matrix, i.e. soil, paint, wipe(spiked with baghouse dust) or air filter. Results must fall within the PT acceptance range or laboratory LCS range as appropriate.

Each technician/analyst must complete a minimum of 4 independent test runs of sample preparation/analysis prior to prepping/analyzing actual samples. This test is performed through the digestion/analysis of four separate groups of 5 replicate, matrix specific Certified Reference Material samples, with each group separated by at least one day. To be deemed acceptable per ELLAP requirements, 75% of the replicates in each group must recover within 90-110% of the true value. Any individual group that fails to meet the ELLAP criteria must be repeated in its entirety (all 5 replicates repeated).

Once all requirements in 5.7.10.8.1.1 have been met, the technician/analyst will be approved to begin unsupervised prep/analysis of client samples. Documentation of approval to begin work is defined as the date signed by the Technical Director (or designee) on the Demonstration of Capability Certification form.

5.7.10.8.1.2 Continuing Demonstration of Capability (CDOC). Each technician/analyst must demonstrate continued capability at least every 6 months through the analysis of AIHA-LAP, LLC provided PT samples or in house laboratory QC samples, i.e. LCS samples. Results must fall within the AIHA-LAP, LLC PT acceptance criteria or Policy Module 2C, Table 2C-1 LCS control limits per samples used.

5.7.10.8.1.3 All IDOC and CDOC documentation for ELLAP related procedures is maintained and available for review for at least 5 years.

5.7.10.8.2 IHLAP Chemistry Specific Technician/Analyst Training Requirements:

5.7.10.8.2.1 Initial demonstration of capability.

Each technician/analyst must complete at least 20 days of work/training in the prep and/or metals analysis lab using technologies/instrumentation similar to that used for IH samples under the direct supervision of an IH trained technician/analyst prior to unsupervised prep and/or analysis of IH regulated client samples. Each analyst /technician must read, understand and agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form. Each technician / analyst must prep and/or analyze as appropriate at least 2 blind reference material samples (concentration unknown to the technician/analyst). These samples may be AIHA-LAP, LLC provided PT samples or laboratory prepared Certified Reference

Analysis	Matrix*	Holding Time	Container	Preservative
Density / Specific Gravity	Water	7 days	P, G	0 - ≤6°C
Density / Specific Gravity	Soil / Sludge	6 months	P, G	0 - ≤6°C
DRO	Water	7 days (ext)	G	0 - ≤6°C
DRO	Soil	14 days (ext)	G	0 - ≤6°C
EDB, DCBP	Water	14 days (Ext)	40 mL VOA	0 - ≤6°C
Ferrous Iron	Water	24 hours	P, G	None, 0 - ≤6°C
Flash Point/Ignitability	Liquid	6 months	P, G	None
Flash Point/Ignitability	Solid	6 months	P, G	None
Ignitability	Solids	6 months	P, G	None
FL-PRO	Water	7 days	G	1:1 H_2SO_4 (pH<2), 0 -≤6°C
FL-PRO	Soil	14 days	G	0 - ≤6°C
Fluoride	Water	28 days	P, G	None
FOC/FOM	Solid	28 days	G	0 - ≤6°C
Formaldehyde	Water	72 hrs, to tumble, 72 hrs. to analyze	250 mL G (amber)	0 - ≤6°C
Formaldehyde	Soil	From leachate 72 hours	G	0 - ≤6°C
GRO	Water	14 days	40 mL VOA	1:1 HCl (pH<2), 0-≤6°C
GRO	Soil	48 hours, 14 days after pres.	Pre-weighed vials or Encore*	Sodium Bisulfate, Methanol, 0-≤6°C
Hardness, calculation	Water	6 months	P, G	1:1 HNO_3 (pH<2)
Herbicides	Water	7 days (Ext)	G (amber)	0 - ≤6°C
Herbicides	Soil	14 days (Ext)	G	0 - ≤6°C
Hexavalent Chromium	Water	24 hours	P, G	0 - ≤6°C
Hexavalent Chromium	Soil	30 days (Ext)	P, G	0 - ≤6°C
Lead	Air	6 months	Cartridge**	None
Lead	Wipe	6 months	Bag***	None
Lead	Paint	6 months	Bag	None

12.11.2 The laboratory utilizes Type A approach for the Estimation of Uncertainty. One or more of the following options are utilized:

12.11.2.1 Uncertainty specified within a standard method. In those cases where well recognized test method (such as NIOSH, OSHA, etc. method), specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, laboratories need not do anything more than to follow the reporting instructions as long as they can demonstrate they follow the reference method without modification and can meet specified reliability.

12.11.2.2 Laboratory Control Samples (LCS) and Matrix Spikes. In cases where matrix specific LCS (CRM or media spikes) and/or matrix spike data are available, include uncertainty estimated from the standard deviation of long term data collected from routine sample runs for existing test methods or from the standard deviation of the LCS or matrix spike data for method validation/verification studies for new test methods.

12.11.2.3 Duplicate Data. In cases where sub-sampling occurs and there are data over the reporting limit, include uncertainty estimated from long term duplicate data collected from routine sample runs for existing test methods or method validation/verification studies for new test methods.

12.11.2.4 Proficiency Testing (PT) Sample Data. In cases where the previous options are not available and where PT samples are analyzed with sufficient data above the reporting limit, pooled PT sample data can be used to estimate uncertainty.

12.11.3 Uncertainty determinations specific to each type of testing for AIHA-LAP, LLC is as follows:

12.11.3.1 Industrial Hygiene Chemical/Gravimetric Analysis.

The laboratory uses the Type A approach to Measurement Uncertainty. Acceptance limits are determined using historical LCS (CRM or media spikes) data for each procedure/target analyte. Once at least twenty values are available, the mean and standard deviation of the data set are calculated. Bias is noted and available for reporting. The data is evaluated for outliers using standard Grubbs Outlier calculations with statistical outliers omitted. Control limits are set at ±3 standard deviation and for measurement uncertainty k=2, or ± 2 standard deviation are used.

Where target analyte spiking is not applicable such as for gravimetric testing, only precision limits are used for uncertainty determinations. If less than 50 points are available for calculation, the limits are considered interim limits.

12.11.3.2 Industrial Hygiene Asbestos by PCM Analysis. Ranges of uncertainty for IH asbestos by PCM testing are determined for precision only using daily reference slide and blind recount analyses as described below.

12.11.3.2.1 The laboratory's set of reference slides includes slides from previous PAT rounds, Round Robins and field samples. The laboratory acceptance limits have now been determined from data accumulated from blind recounts of these reference slides and established at 95% confidence limits. From blind repeat counts of reference slides, Sr values obtained for 3 following ranges: 5-20 fibers in 100 graticule fields; 20.5-50 fibers in 100 graticule fields; 50.5-100 fibers in 100 graticule fields.

12.11.3.3 Environmental Lead Analysis for reporting under the ELLAP Program. Ranges for uncertainty for ELLAP testing for precision and accuracy are determined by the laboratory. Monitoring of method performance and bias is accomplished using statistical process control (charts or database) for monitoring AES laboratory performance with QC sample analysis (LCS/LCSD, MS/MSD). SOPs (Sec. 13) for Lead in Paint, Lead in Wipes, Lead in Soil

(SW 7000D), and Lead in Airborne Dust describe the required minimum performance criteria for QC sample analysis and the method performance for the laboratory. Method performance and bias are evaluated on an annual basis by the QA Manager. If the calculated limits are more stringent than the required minimum performance criteria listed in the SOP tables, then the limits are updated in LIMS. All monitoring data in the form of control charts are maintained/posted to the portal server, the laboratory's archival system.

12.11.3.4 Quantifiable Fungal Analysis for reporting under the EMLAP Program. Ranges for uncertainty for quantifiable fungal testing are determined for precision only. Duplicate samples are counted for at least 5% of samples for inter-analyst precision monitoring and replicates samples are counted by different analysts for intra-analyst precision monitoring. Uncertainty ranges are determined using the mean of the range of the logarithm of each count obtained from a minimum of 20 duplicate/replicate pairs. This mean value is multiplied by 3.27 to obtain the final control limit. Once the control limit is determined, the logarithmic range for each ongoing duplicate/replicate pair is determined and must be < control limit value. Specific information used for control limits for each individual EMLAP test method are provided in Table 5-1.

The lab determines the measurement of uncertainty associated with Spore Trap Analysis by using the Type (A) methodology. QC reference slides are used that have varying spore count levels. 30 data points are used for each QC slide. From these counts the Mean and Standard Deviation are determined. Then the Coefficient of Variation (CV) is calculated for each set of data by dividing the standard deviation by the mean. Then the pooled CV is calculated by adding the squares of the CV values, averaging them and taking the square root. The expanded Measurement of Uncertainty (MU) is calculated by multiplying the pooled CV value by the appropriate coverage factor k. For a confidence level of 95%, k is approximately 2 for a data set of 30 points or more. This RSD value is then multiplied by the calculated or observed value of the sample to be expressed as a measurement of uncertainty. When reporting results for expanded Measure of uncertainty the test results and the expanded measurement of uncertainty are expressed in the same units.

Example with a calculated CV pooled of 0.114:

Expanded MU @ 95% C.L. (k=2) equals CV *pooled (.114)* X 2 = 0.23 (23% RSD)

Bias cannot be determined. No quantitative reference material available
Example analytical uncertainty for air sample with 500 spores/m3:

Expanded analytical uncertainty = 500 spores/m3 X 0.23 = 115 spores/m3
Example of reporting for air sample with 500 spores/m3:

500 spores/m3 with an analytical uncertainty of +/- 115 spores/m3 at the 95% confidence level

12.11.3.5 Qualitative Fungal Analysis for reporting under the EMLAP Program. In order to monitor consistency with regard to genus/species identification, acceptability criteria for taxon identification and taxon abundance ranking are described below. These are laboratory determined; interim criteria as no regulatory guidance or method specified criteria are available.

12.11.3.5.1 Taxon identification acceptability: On the replicate and duplicate analyses, daily reference slide analyses, monthly reference culture analyses and round robin study analyses with at least 3 different organisms present, 60% of all genus/species of fungi and/or genus/group of fungi identified on the original sample at levels >10x LOD should also be identified on the recount.

12.11.3.5.2 Taxon abundance ranking acceptability: On the replicate and duplicate analyses, daily