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# **SAMPLING AND ANALYSIS PLAN**

## **Tonopah Convention Center 301 Brougher Avenue Tonopah, NV NDEP Contract #10-008, Task M11-11**

*Prepared for:*

*State of Nevada  
Department of Conservation and Natural Resources  
Division of Environmental Protection  
Bureau of Corrective Actions  
901 S. Stewart Street, Suite 4001  
Carson City, Nevada 89701-5249*

| Soil and Groundwater Remediation

| Regulatory Compliance

| Environmental Audits

| Hydrogeology

| Hazmat Response

*May 02, 2011*

**Sampling and Analysis Plan for:**

Tonopah Convention Center

301 Brougner Avenue

Tonopah, Nevada

May 02, 2011

**Date**

MGA Principal: \_\_\_\_\_

MGA Project Manager: \_\_\_\_\_

MGA QA Manager: \_\_\_\_\_

NDEP QC Coordinator for the Nevada Brownfields Program: \_\_\_\_\_

NDEP Brownfields Program Coordinator: \_\_\_\_\_

**For EPA use:**

Approved by EPA Project Manager:

Date:

\_\_\_\_\_

\_\_\_\_\_

Expedited Review? ☐ Yes

☐ No

Received by QA Office:

Date: \_\_\_\_\_

Reviewed by:

Date

Approved:

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Region 9 Quality Assurance Manager

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## Table

Table 1	DQO Summary Table for Environmental Sampling, Tonopah Convention Center Building, Tonopah, NV
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## FIGURES

Figure 1	Project Location Map
Figure 2	Site Map

## APPENDICES

Appendix A	Laboratory Data Quality Objectives and Sample Handling Procedures
Appendix B	MGA SOPs
Appendix C	Chain-of-Custody Forms
Appendix D	Sample Labels
Appendix E	Site Health and Safety Plan

# 1. INTRODUCTION

McGinley and Associates, Inc. (MGA) has prepared this Sampling and Analysis Plan (SAP) for assessment activities to be conducted at the Tonopah Convention Center located in Tonopah, Nevada. These assessment activities are being funded by the State of Nevada Brownfields program. This SAP was prepared in accordance with the Nevada Division of Environmental Protection (NDEP) Quality Assurance Program Plan (QA Program Plan) for the Nevada Brownfields Program (NBP) (NDEP 2007).

The purpose of this project is to assess the presence of asbestos containing material (ACM), lead-based paint and mold within the Tonopah Convention Center building.

## 1.1 Site Name

Tonopah Convention Center.

## 1.2 Site Location

The site is located at 301 Brougner Avenue in Tonopah, Nevada. The site is located on assessor parcel number (APN) 008-125-08 which covers an area of approximately 0.28 acres. The 11,354 square foot building is currently being utilized as the Town of Tonopah Convention Center.

## 1.3 Responsible Agency

This project is being conducted for the NDEP through State of Nevada Brownfields program. The investigation will conform to the NBP's QA Program Plan (NDEP, 2007).

## 1.4 Project Organization

Title/Responsibility	Name	Phone
<b>Town of Tonopah</b>		
Administrative Supervisor	Susan Dudley	(775) 482-6336
<b>NDEP</b>		
Program Coordinator for the Nevada Brownfields Program – Project coordination, liaison with Town of Tonopah	Jeff Collins	(775) 687-9381
Case Officer – Review SAP, quality assurance	David Friedman	(775) 687-9385
Quality Coordinator for the Nevada Brownfields Program – Review SAP, quality assurance	Mary Siders	(775) 687-9496
<b>USEPA</b>		
USEPA Project Manager – Work plan review	Carl Brickner	(415) 972-3814
USEPA QA Manager – SAP review	Gail Morison	(415) 972-3807
<b>McGinley and Associates, Inc.</b>		
Principal – Senior review, regulatory liaison	Joe McGinley	(775) 829-2245
Project Manager – Project management, regulatory liaison, coordinate field activities, data review, report preparation.	Brett Bottenberg	(702) 260-4961
Quality Manager – Oversee implementation of SAP, review QA/QC	Brett Bottenberg	(702) 260-4961

procedures, data validation.		
Environmental Scientist – Conduct sampling activities	Gene Johnson	(775) 829-2245
CAD Operator – CAD support	Tim Dory	(775) 829-2245
Administrative Assistant – Administrative support	Linda Comstock	(775) 829-2245
<b>Contractors/Vendors</b>		
Natural Link Mold Lab – Analysis of mold samples	Sean Abbott	(775) 746-3838
Asbestos TEM Laboratories, Inc. - Analysis of paint and ACM samples	Sue Ehrlich	(775) 359-3377

## 1.5 Statement of the Specific Problem

The Town of Tonopah is receiving a grant from the USDA to rehabilitate the existing Convention Center and an adjacent apartment complex for the purpose of promoting economic development within the Town of Tonopah. A previous asbestos survey was conducted on the Convention Center which did reveal asbestos containing materials in the building. In order to renovate the property for reuse, potential environmental concerns in the building including ACM, lead-based paint, and mold must be addressed and mitigated prior to renovation.

## 2. BACKGROUND

The existing Tonopah Convention Center facility was built in the 1940s as the USO during World War II. The 11,354 square foot facility is approaching 70 years old and has undergone several additions. The facility has been used for many years to accommodate a variety of activities, including community events such as graduations, funerals, and weddings.

The Tonopah Town Board uses this facility to hold Town Board Meetings. The community has come to rely heavily on this facility for its community based events. Due to the age and condition of the facility, the Town wants to rehabilitate it to include it in the Town's plan to promote more outside events and subsequently promote economic development within the Town of Tonopah.

The building contains suspect ACM and lead-based paint and the potential exists for the presence of mold.

### 2.1 Sampling Area Description

The parcel occupies approximately 0.28 acres in a commercial area in downtown Tonopah. The property is bounded on the north commercial properties, on the south by Brougner Ave, on the west by S. Summit Street and on the east by S. Central Street (Figure 2). Sampling will be conducted in the building located on the parcel. The building is 11,354 square feet in size. Samples will be collected from suspect ACM (insulation, dry-wall, etc.), painted surfaces, and visible mold within the building.

### 2.2 Operational History

The existing Tonopah Convention Center facility was built in the 1940s as the USO during World War II. The 11,354 square foot facility is approaching 70 years old and has undergone several additions. The facility has been used for many years to accommodate a variety of activities, including community events such as graduations, funerals, and weddings.

## **2.3 Previous Investigations/Regulatory Involvement**

A limited asbestos investigation was performed by the Town of Tonopah in 2009. The investigation focused only on the roof of the Convention Center and revealed ACM within samples collected. According to the Town of Tonopah, the grant funding source is requiring that asbestos, lead based paint, and mold risks, if any, be mitigated prior to renovation.

## **2.4 Geological Information**

The geology of the subject property has been mapped as Tertiary-age sedimentary strata and interbedded tuffs, including the Fraction Tuff (Kleinhampl & Ziony 1985). The sedimentary strata and interbedded tuff deposits are described as chiefly volcanogenic sedimentary rocks of lacustrine and fluvial origin interbedded with rhyolitic air-fall tuffs. Deposits of local algal reefs, marly and coquinoid limestones, and diatomitic beds are also present. The Fraction Tuff is described as a lithic-rich rhyolitic to quartz latitic ash-flow tuff.

Groundwater is estimated to be several hundred feet below ground surface.

## **2.5 Environmental and/or Human Impact**

No adverse human health effects associated with the contamination at this site have been reported or documented.

# **3. PROJECT DATA QUALITY OBJECTIVES**

## **3.1 Project Task and Problem Definition**

The purpose of this investigation is to assess for the presence of asbestos, lead-based paint, and mold in the onsite building. The assessment will provide adequate data to determine the extent of abatement necessary prior to renovation.

## **3.2 Data Quality Objectives (DQOs)**

The DQO process (EPA 2006) is a systematic planning tool that is used to establish performance or acceptance criteria. These criteria, in turn, serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of a study. The DQO process consists of seven iterative steps, as described in the following sections and summarized in Table 1.

### **3.2.1 Step 1: State the Problem**

The property will be used to house the Town of Tonopah's Convention Center, Tonopah Public Library, Town of Tonopah Offices and commercial office space. Prior to renovation of the property, potential environmental concerns in the building including ACM, lead-based paint, and mold must be addressed. Analytical data is needed to assess for the presence of these materials and determine if abatement and/or additional assessment activities are needed.

### **3.2.2 Step 2: Identify Decisions**

Analytical data for collected samples will be evaluated to determine if concentrations of asbestos, mold and lead-based paint exceed regulatory action levels. Asbestos data will be compared to levels established in OSHA 29 CFR 1926.1101, NAC 618.850 to 618.986, NESHAPS 40 CFR 61.141 and AHERA 40 CFR Part 763. Mold data for samples collected inside the building will be compared to mold levels in samples collected outside the building (background/baseline concentration). Lead data for paint samples will be compared to levels established in 40 CFR Part 745 and TSCA 402(c). Results of the investigation will be used to determine if additional assessment and/or abatement is required.

### 3.2.3 Step 3: Identify Inputs

Information required to address project objectives includes regulatory action levels and quantitative analytical data for samples collected during this investigation.

### 3.2.4 Step 4: Define Study Boundaries

Samples shall be collected within the on-site building. A mold sample (air) shall also be collected outside the building to establish a background mold concentration. Sampling shall be limited to suspect ACM, painted surfaces, and mold (if observed based on visual inspection). The duration of the assessment activities described in this SAP is approximately one week.

### 3.2.5 Step 5: Develop Decision Rules

Decision rules are specified in Table 1, and describe actions based on qualitative and definitive data.

Laboratory data for ACM, lead-based paint and mold samples will be compared to regulatory action levels. If asbestos, lead, and/or mold concentrations exceed the regulatory levels, an abatement plan shall be prepared to remove the subject material.

### 3.2.6 Step 6: Specify Tolerable Limits on Decision Errors

This is not a statistically based study; therefore, sampling locations will be selected based on professional judgment and site knowledge.

### 3.2.7 Step 7: Optimize the Sampling Design

The number of samples will be determined in the field using professional judgment such that samples are representative of site conditions.

## 3.3 Data Quality Indicators (DQIs)

Data quality indicators (precision, accuracy, representativeness, completeness, comparability and sensitivity [i.e., PARCCS parameters]) refer to quality control criteria established for various aspects of data gathering, sampling, and/or analyses. Precision is the degree of mutual agreement between or among independent measurements of a similar property (usually reported as standard deviation (SD) or relative percent difference) and relates to the analysis of duplicate laboratory or field samples. Accuracy is the degree of agreement of a measurement with a known or true value and is determined by comparing the reported laboratory value for a sample to a known or true concentration (i.e. matrix spikes, surrogate spikes, laboratory control samples and performance samples. Representativeness is the expression of the degree to which data accurately and precisely represent a characteristic of an environmental condition or population and relates to the method of collecting samples and determining sample locations (i.e. statistical sampling, professional judgment, etc.). Completeness is expressed as the percent of valid usable data obtained compared to the amount that was expected. Comparability expresses the degree of confidence with which one data set can be compared to another. Sensitivity is defined by the laboratory detection limits and are generally expressed in terms of method detection limits (MDLs) or reporting limits (RLs).

**Precision and Accuracy:** The measurement quality objectives (MQOs) for precision and accuracy for the analyses of the specific chemicals of concern (CoCs) in the soil are summarized in Table 3.

**Representativeness:** Sampling locations will be selected using professional judgment and will adequately represent site conditions for the area(s) being investigated.

**Completeness:** The project goal is to obtain an adequate number of samples to characterize site conditions.



**Comparability:** Previous asbestos sampling has been conducted on the roof of the building. However, it does not appear that the samples were collected by AHERA certified personnel and the data may not be reliable. Therefore, this data may not be utilized.

**Sensitivity:** The laboratory reporting limits for the each analyte are summarized in Table 2. The reporting limits are well below the action levels and are adequate for this investigation.

### 3.4 Data Review and Validation

Data verification is the process of evaluating the completeness, correctness, conformance, and compliance of a specific data set against the method, procedural, or contractual requirements. Data verification evaluates whether sampling protocols, SOPs, and analytical methods were followed during data generation. Verification also involves examining the data for errors or omissions. Field and laboratory staff will verify that the work is producing appropriate outputs.

Data validation is a systematic process for reviewing a body of data against a pre-established set of acceptance criteria defined in this plan. Data validation is an analyte- and sample-specific process that extends the evaluation of data beyond data verification and is performed to determine the analytical quality of a specific data set. Validation involves a detailed examination of the data package to determine whether MQOs for precision, accuracy, and sensitivity have been met. For this environmental assessment, the intent of the data review and validation process is to verify that the specified levels of precision, accuracy, reproducibility, completeness, comparability, and analytical sensitivity of the final results are achieved, with respect to the project MQOs, and that the data fulfill project DQOs.

MGA's QA officer will supervise or perform data quality assessment tasks. MGA will consistently evaluate and document measurement data to monitor consistency with MQOs, to quantitatively assess data quality, and to identify potential limitations to data use. MGA will review field and analytical laboratory data generated for this project, including the following:

- Chain of custody documentation;
- Laboratory batch QC frequency; and,
- Results of batch and field QC analyses;

**Laboratory Data:** The laboratories will generate and review all laboratory data. Each data point will be assessed as non-qualified or qualified based upon the acceptance criteria. Data may be qualified as "estimated" (J-qualified); these data are used as is. Some data may be qualified as "rejected" (R-qualified) if critical QC parameters are not met; these data are unusable for any purpose. Sample re-analysis, for data not meeting MQOs, will be considered as a possible corrective action. Third-party data validation will not be performed.

### 3.5 Data Management

Sampling will be conducted in accordance with MGA's standard operating procedures (SOPs). A unique identification number will be assigned to each sample. The number will be an alphanumeric sequence that serves as an acronym to identify the sample. The following format will be used for the sample designation:

ACM Samples:

**Sample ID:** BRN003-ACM 1-Piping Insulation

**BRN003** - MGA Project Number

**ACM 1** - Insulation, boiler1 - Sample type (suspect ACM), number and location (piping insulation)

Paint Samples:

**Sample ID:** BRN003-PS 1 - Wall

**BRN003** - MGA Project Number

**PS 1** - Insulation, boiler1 - Sample type (paint sample), number and location (wall)

Fungal/Air Samples:

**Sample ID:** BRN003-AS 1 - Basement

**BRN003** - MGA Project Number

**AS 1** - Insulation, boiler1 - Sample type (air sample), number and location (wall)

Field logs shall be maintained throughout the project. The following information shall be included on the field logs: description of activities conducted, dates and times, field observations, deviations from sampling program, names of onsite personnel, sampling locations.

ACM, paint and air samples shall not be preserved or cooled. Samples shall be delivered or shipped to the laboratory under chain-of-custody protocol.

### 3.6 Assessment Oversight

Prior to commencing with field activities, the SAP will be reviewed by the Project Team. The MGA QA Officer will oversee QC of all field activities. If modifications to the proposed sampling program are required due to field conditions, the Project Manager shall be notified for direction. Any modifications to the sampling plan will be documented in the field logs and in the project report as "deviations from the sampling plan."

## 4. SAMPLING RATIONALE

Asbestos samples shall be collected from suspect ACM, and paint samples shall be collected from painted surfaces. Mold samples will be collected at locations where mold is suspected to be present, based on visual observations. An adequate number of samples shall be collected to represent site conditions. Professional judgment shall be used to select sampling locations that are likely to provide data to address project DQOs (Table 1). Decision statements formulated in the project DQOs are largely concerned with assessing for the presence of asbestos, lead-based paint and mold.

### 4.1 Soil Sampling

Sampling of soil is not included in the scope of this investigation.

## **4.2 Sediment Sampling**

Sampling of sediments is not included in the scope of this investigation.

## **4.3 Water Sampling**

Sampling of water/groundwater is not included in the scope of this investigation.

## **4.4 Biological Sampling**

Biological sampling is not included in the scope of this investigation.

## **4.5 Asbestos Sampling**

Samples will be collected from suspect ACM. An estimated 30 to 50 samples will be collected. Samples shall be removed using a clean knife and shall be at least two square inches or two tablespoons in size. Care shall be taken to minimize disturbance to the material. The material shall be placed in a zip-lock bag, which will be sealed and labeled.

## **4.6 Paint Sampling**

Samples will be collected from suspect lead-based paint. An estimated 15 to 25 samples will be collected.

## **4.7 Mold Sampling**

Samples shall be collected from apparent mold spores. An estimated 5 to 10 samples will be collected. Air samples shall be collected from inside and outside the building to establish a background/baseline concentration.

# **5. REQUEST FOR ANALYSIS**

Laboratory analyses are discussed in Section 5.1 below.

## **5.1 Analyses Narrative**

### **5.1.1 Suspect ACM**

Samples collected from suspect ACM shall be analyzed using polarizing light microscopy (PLM)/Stereomicroscopy for bulk asbestos samples. These methods are described in 40 CFR Part 763, Appendix E to Subpart E (interim and EPA 600/R-93/116, improved).

### **5.1.2 Paint**

Paint samples shall be analyzed by atomic absorption spectrometry (AAS) using method SW-846-7420.

### **5.1.3 Mold**

Mold samples shall be analyzed by Fungal Microscopic Exam/25152-R01

## **5.2 Analytical Laboratory**

Analytical testing on suspect ACM and paint samples shall be conducted by Crisp Analytical LLC of Carrollton, Texas. Analytical testing on mold samples shall be conducted by Natural Link Mold Lab of Reno, Nevada. Analytical testing and sampling handling shall be conducted in accordance with the standard operating procedures (SOPs) for each laboratory.

## **6. FIELD METHODS AND PROCEDURES**

### **6.1 Field Equipment**

#### **6.1.1 List of Equipment Needed**

##### **6.1.1.1 Suspect ACM, Paint, Fungal Mold**

- field logbook and field data sheets
- personal protective equipment (Level D)
- knife/box cutter with retractable blade
- tape measure
- camera
- zip-lock bags
- surface tape

##### **6.1.1.2 Air Samples (Mold)**

- field logbook and field data sheets
- low flow (15 liters/minute) air pump (Zefon Bio-Pump, or equivalent)
- spore trap sampler (Air-O-Cell, or equivalent).

#### **6.1.2 Calibration of Field Equipment**

All field equipment will be calibrated according to the manufacturer's guidelines and specifications.

### **6.2 Field Screening**

Field screening will not be utilized in this investigation.

### **6.3 Soil Sampling**

Not applicable, soil sampling is not included in the scope of this investigation.

### **6.4 Sediment Sampling**

Not applicable, sediment sampling is not included in the scope of this investigation.

### **6.5 Water Sampling**

Not applicable, water sampling is not included in the scope of this investigation.

### **6.6 Biological Sampling**

Not applicable, biological sampling is not included in the scope of this investigation.

### **6.7 Asbestos Sampling**

Samples shall be collected from suspect ACM by cutting material using a clean knife. Samples shall be at least two square inches or two tablespoons in size. Care shall be taken to minimize disturbance to the material. The samples shall be placed in a zip-lock bag which will be sealed and labeled.

### **6.8 Paint Samples**

Samples shall be collected from painted surfaces using a clean knife. The samples shall be placed in a zip-lock bag which will be sealed and labeled.

## **6.9 Fungal/Mold Samples**

Samples shall be collected from fungal material using surface tape. Air samples will be collected in an Air-O-Cell cassette (or equivalent) using a low-flow air pump. Samples shall be collected for a period of five minutes. Cassette shall be sealed, labeled and placed in a zip-lock bag.

## **7. SAMPLE CONTAINERS, PRESERVATION AND STORAGE**

### **7.1 Asbestos Samples**

Asbestos samples shall not be chilled. Care shall be taken to prevent deterioration or damage to samples during transit.

### **7.2 Paint Samples**

Paint samples shall not be chilled. Care shall be taken to prevent deterioration or damage to samples during transit.

### **7.3 Mold Samples**

Mold samples shall not be chilled. Care shall be taken to prevent deterioration or damage to samples during transit.

## **8. DISPOSAL OF RESIDUAL MATERIALS**

No investigation-derived waste is anticipated to be generated during this investigation. Any investigation-derived waste that is generated will be disposed of properly.

## **9. SAMPLE DOCUMENTATION AND SHIPMENT**

### **9.1 Field Notes**

#### **9.1.1 Field Logbooks**

Field logs will be completed describing all field activities. The following information will be included in the field logs:

- project name and location
- sampling location and description
- site plan showing sample locations
- sampler's name (s)
- date and time of sample collection
- type of sample (e.g., soil, ACM, paint, fungal)
- type of sampling equipment used
- field instrument readings and calibration
- field observations and details related to analysis or integrity of samples (e.g., noticeable odors, colors, etc.)
- sample preservation
- lot number of the sample containers, sample identification numbers and explanatory codes, and chain-of-custody form numbers
- name of recipient laboratory

### **9.1.2 Photographs**

Photographs will be taken at the select sampling locations. They will serve to verify information entered in the field logbook. For each photograph taken, the following information will be written in the logbook:

- time, date, location, and weather conditions
- description of the subject photographed
- name of person taking the photograph

## **9.2 Labeling**

All samples collected will be labeled in a clear and precise manner for proper identification in the field and for tracking in the laboratory. The samples will have pre-assigned, identifiable, and unique numbers. At a minimum the sample labels will contain the following information: sample location, date and time of collection, analytical parameter(s), and method of preservation. Every sample will be assigned a unique sample number.

## **9.3 Sample Chain-of-Custody Forms and Custody Seals**

All samples shall be delivered to the laboratory under chain-of-custody protocol.

## **9.4 Packaging and Shipment**

Samples shall be shipped in a manner as to prevent damage or deterioration during transit.

# **10. QUALITY CONTROL**

## **10.1 Field Quality Control Samples**

Samples will be collected in accordance with industry standard procedures.

### **10.1.1 Assessment of Field Contamination (Blanks)**

No equipment blanks will be collected during this investigation.

## **10.2 Background Samples**

An air sample shall be collected from outside the building to establish background/baseline conditions.

## **10.3 Field Screening and Confirmation Samples**

No confirmation samples will be collected during this investigation.

## **10.4 Laboratory Quality Control Samples**

Laboratory QC (e.g., matrix spike/matrix spike duplicate samples) samples will be analyzed to monitor the precision and accuracy of its analytical procedures.

# **11. FIELD VARIANCES**

As conditions in the field may vary, it may become necessary to implement minor modifications to sampling as presented in this SAP. Modifications to the approved SAP will be documented in the sampling project report.

## **12. FIELD HEALTH AND SAFETY PROCEDURES**

Care shall be taken to minimize disturbance of suspect ACM material and fungal material during sampling activities.

## **13. SCHEDULE FOR SAMPLING ACTIVITIES**

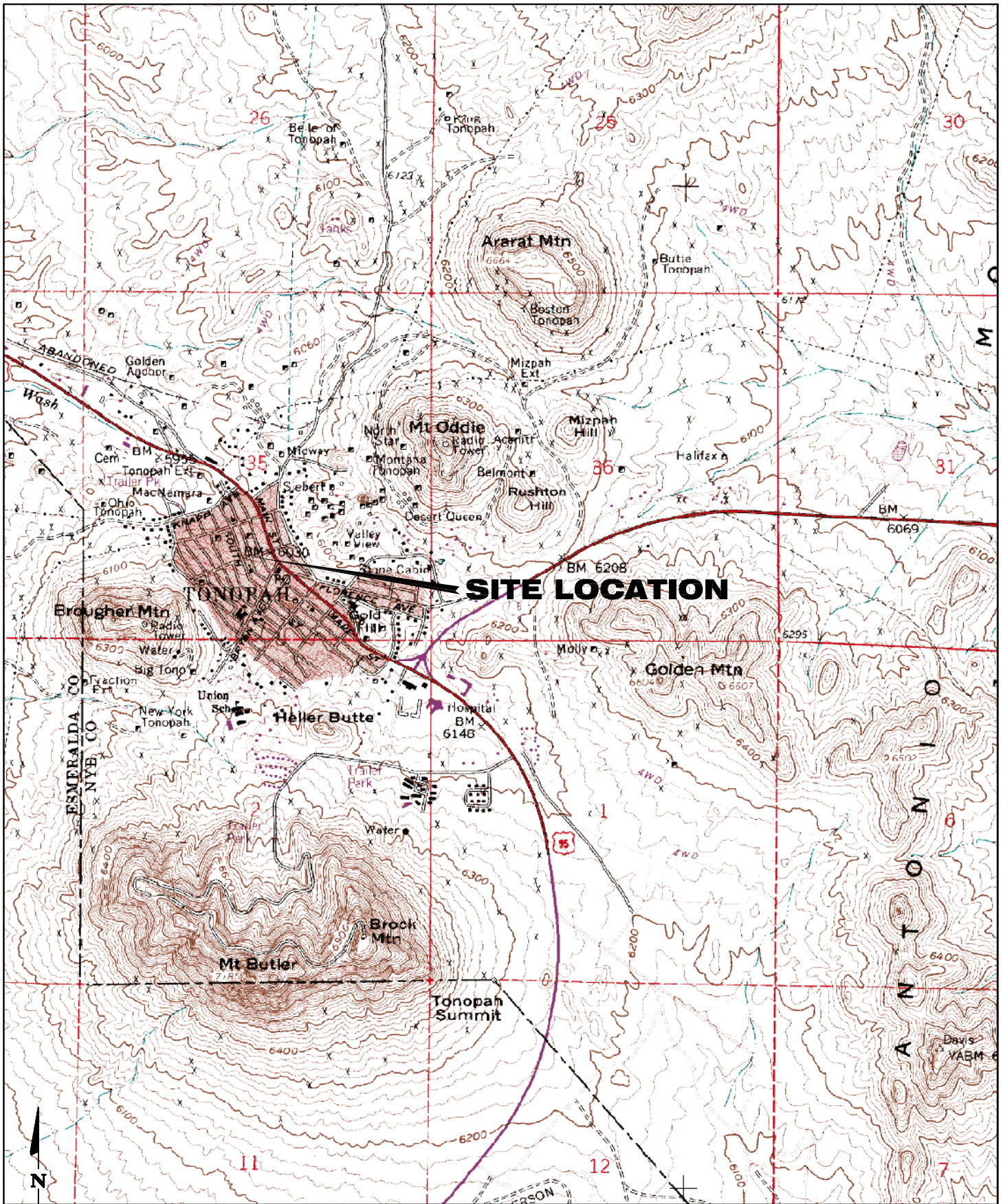
MGA will commence with the activities proposed herein upon receiving NDEP approval of this SAP. It is anticipated that field activities will be completed within 30 days of receiving SAP approval. A draft report of findings will be submitted before June 30, 2011.

## **14. REFERENCES**

*Final Nevada Brownfields Program Quality Assurance Program Plan*, May 20, 2007, Nevada Division of Environmental Protection.

US EPA. 2006. *Guidance on Systematic Planning using the Data Quality Objectives Process*. February. EPA QA/G-4, EPA/240/B-06/001





**SITE LOCATION**

**FIGURE 1**

**PROJECT LOCATION MAP  
-SHOWING-  
APN 008-125-08  
301 BROUGHER AVE  
TONOPAH, NEVADA**

0 1000 2000  
FEET

DESIGNED DRAWN CHECKED APPROVED				BH
				TAD
REVISIONS	No.	DESCRIPTION	BY	DATE
1				
2				
3				




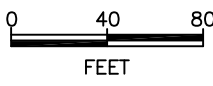

McGinley & Associates

SCALE: AS SHOWN  
JOB NO. BRN-003

REVISION  
A





 	DESIGNED	BH		<b>FIGURE 2</b>	 McGinley & Associates SCALE: AS SHOWN JOB NO. BRN-003 REVISION <div style="border: 1px solid black; width: 20px; height: 20px; text-align: center; line-height: 20px;">A</div>
	DRAWN	TAD			
	CHECKED				
	APPROVED				
	REVISIONS	No.	DESCRIPTION	BY	

**SITE MAP**  
**-SHOWING-**  
**APN 008-125-08**  
**301 BROUGHER AVE**  
**TONOPAH, NEVADA**

**Table 1. DQO Summary Table for Environmental Sampling, Tonopah Convention Center Building, Tonopah**

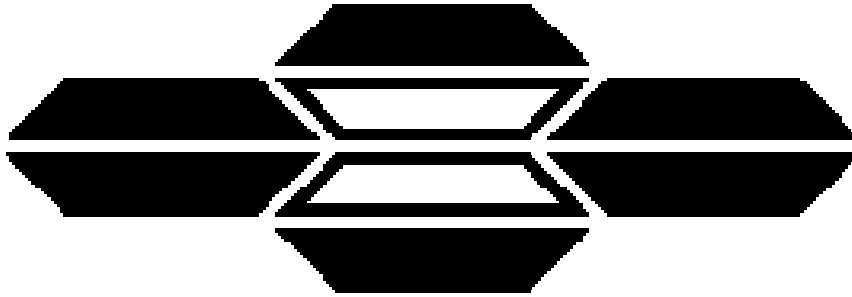
STEP 1	STEP 2	STEP 3	STEP 4	STEP 5	STEP 6	STEP 7
State the Problem	Identify the Decisions	Identify the Inputs to the Decisions	Define Study Boundaries	Develop Decision Rules	Specify Tolerable Limits on Errors	Optimize Sampling Design
<p>The property will be used to house the Town of Tonopah's Convention Center, Tonopah Public Library, Town of Tonopah Offices and commercial office space.</p> <p>Prior to renovation of the property, potential environmental concerns must be addressed:</p> <p>ACM and lead-based paint may be present in building.</p> <p>There is the potential presence of mold.</p> <p>Analytical data are needed to assess the presence of these materials in the building.</p>	<p>1) Is asbestos present in the building materials?</p> <p>2) Does paint in building contain lead in excess of acceptable levels?</p> <p>3) Is mold present in the building?</p> <p>4) Is there a potentially complete pathway for contaminated materials to adversely affect human health or the environment?</p> <p>5) Are abatement actions required?</p>	<p>Analytical data for collected samples (quantitative data)</p> <p>Regulatory action levels for asbestos and lead.</p> <p>Potential receptors and completed exposure pathways</p> <p>Pathways for impacts to human health</p>	<p>Samples shall be collected within the onsite building.</p> <p>A mold sample (air) shall also be collected outside the building to establish a background mold concentration.</p> <p>Sampling shall be limited to suspect ACM, painted surfaces, and mold (if observed based on visual inspection).</p> <p>The duration of the assessment activities described in this SAP is approximately one week</p>	<p>1) If asbestos exceed regulatory levels, an abatement plan shall be prepared to remove these materials.</p> <p>2) If paint in the building contains concentrations of lead in excess of acceptable levels, then an abatement plan will be prepared to remove this material.</p> <p>3) If the fungal levels in the building exceed acceptable levels, and/or mold is discovered in the building, then an abatement plan will be prepared to mitigate threats to human health.</p>	<p>The number of samples to be collected is not statistically based and will be determined in the field based using professional judgment.</p> <p>MQOs and DQIs established for analytical data are described in the NBP QA Program Plan.</p>	<p>The quantity of samples shall be adequate to represent site conditions.</p>

# **APPENDIX A**

---

## **Laboratory Data Quality Objectives and Sample Handling Procedures**

**Asbestos TEM Labs, Inc.**  
**630 Bancroft Way**  
**Berkeley, CA 94710**



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# **Laboratory Quality Assurance Plan**

Quality Assurance Director: Robert E. Butler

President: Mark R. Bailey, R.G.

Date of Revision: 12/10/2009



**Asbestos TEM Labs, Inc.**

## **Laboratory Quality Assurance Plan**

**Issue Date:**  
**2009/12/10**  
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### **Quality Manual**

This Quality Manual follows the requirements of ISO 17025 and ISO 9001. This Quality Manual is confidential and assigned as outlined below.

Issued to: Asbestos TEM Laboratories, Inc.

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**Asbestos TEM Labs, Inc.**

# **Laboratory Quality Assurance Plan**

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# Laboratory Quality Assurance Plan

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## Introduction

### Purpose

This Quality Manual contains all the requirements that our laboratory uses to demonstrate our quality system, technical competence, and valid results.

Section 4 specifies how we demonstrate sound management and maintain client satisfaction.

Section 5 specifies how we demonstrate technical competence in our laboratory.

In addition, this Quality Manual outlines how we meet:

- Ø ISO 17025-2005 Requirements
- Ø AIHA ELLAP Accreditation Requirements

All personnel are to take an active role in establishing, implementing, and maintaining our quality management program. We do not separate quality from our daily business. Quality cannot be something that we do just to pass audits. Quality is involved in every facet of the decision-making process in the management of our laboratory and the science that we practice.

### Distribution List

The Quality Assurance Director maintains a distribution list for this Quality Manual.

## 1. Scope

This Quality Manual facilitates:

- Ø recognition of technical competence for standardized methods, non-routine methods, and laboratory-developed methods we perform
- Ø inspection and product certification capabilities and/or services we provide
- Ø total quality for our administrative and technical systems
- Ø audits by clients, regulatory authorities and accreditation bodies
- Ø meeting the requirements of ISO 17025 and ISO 9001
- Ø client satisfaction





## 2. Normative References

### Reference List

ISO/IEC Guide 2 - General terms and their definitions concerning standardization and related activities.

VIM: 1993 - International vocabulary of basic and general terms in metrology, issued by BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML.

ISO 9001:2000 – Quality Management Systems – Requirements.

ISO 17025:2005 – General Requirements for the Competence of Testing and Calibration Laboratories.

National Institute of Standards and Technology Handbook 150, 2006 Edition  
Natl. Inst. Stand. Technol. Handb. 150, 2006 Ed., 65 pages (February 2006)  
CODEN: NIHAЕ2.

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

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

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

### Cross-references

This manual is numerically aligned with the international standard ISO 17025. It is expected that this will prove useful during accreditation audits and expedite the process.

Furthermore, each section cross-references the ISO 9001 standard to assist the laboratory during the ISO 9001 registration process (if applicable).

For ease of use, each section starts with a brief summation of what the section addresses and a listing of the quality terminology and key words.





### 3. Terms and Definitions

For the purposes of this manual, the following relevant definitions apply: ISO/IEC Guide 2; ISO/IEC Guide 30; ISO Council Committee on Conformity Assessment (CASCO); ISO 8402; ISO 10015; ISO 5725-1; ISO 17025; the Food Laboratory Accreditation Working Group (FLAWG); AOAC; American Chemical Society (ACS); and International Vocabulary of Basic and General Terms in Metrology (VIM).

**Accreditation** – formal recognition of a laboratory by an independent science-based organization that the laboratory is competent to perform specific tests (CASCO).

**Accuracy** – the closeness of agreement between a test result and the accepted reference value (ISO 5725-1, ISO Guide 30).

**Calibration** - a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system or values represented by a material measure or a reference material, and the corresponding values realized by the standard (VIM).

#### Notes

1. The result of a calibration permits either the assignment of values or measurands to the indications or the determination of corrections with respect to indications.
2. A calibration may also determine other metrological properties such as the effect of influence quantities.
3. The result of a calibration may be recorded in a document sometimes called a calibration certificate or a calibration report.

**Certification** - procedure by which a third party gives written assurance that a product, process, or service conforms to specified requirements (ISO 8402).

**Certified Reference Material** – a reference material, one or more of, whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body (ISO Guide 30).

**Client** – an entity (customer, agency, company, person, etc.) who receives a test result done according to specified requirements (FLAWG).

**Competence** – ability consisting of theoretical knowledge, practical skills, and attitudes (ISO 10015).



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**Corrective Action** – action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence (ISO 8402).

**Holding Time** – elapsed time between sample collection and either sample preparation or analyses, as appropriate.

**Inspection** - evaluation for conformity by measuring, observing, testing, or gauging the relevant characteristics (ISO/IEC Guide 2). Activity such as measuring, examining, testing or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformity is achieved for each characteristic. (ISO 8402)

**Limit of Detection** – the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte (ACS). The mean value of the matrix blank readings plus 3 standard deviations of the mean, expressed in analyte concentration. For methods with less than 100% recovery the limit of detection should be corrected for recovery (AOAC).

**Limit of Quantification** – lowest concentration of analyte that can be determined with an acceptable level of accuracy and precision. Determined by actual analysis of at least 6 fortified test samples per matrix. It is not determined by extrapolation (AOAC).

**Linearity** – is determined by the analysis of samples with analyte concentrations spanning the claimed range of the method. The results are used to calculate a regression line against analyte calculation using the least squares method. It is convenient if a method is linear over a particular range but it is not an absolute requirement. Where linearity is unattainable for a particular procedure, a suitable algorithm for calculations should be determined (AOAC).

**Measurement Uncertainty** - parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand (International Vocabulary of Basic and General Terms in Metrology).

**Precision** – the closeness of agreement between repeated test results obtained under stipulated conditions (ISO 5725-1, ISO Guide 30); repeatability.

**Preventive Action** – action taken to eliminate the causes of a potential nonconformity, defect, or other undesirable situation in order to prevent occurrence (ISO 8402).

**Proficiency Testing** – determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons (ISO/IEC Guide 2).



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**Quality Assurance** – all those planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality (ISO 8402).

**Quality Control** – the operational techniques and activities that are used to fulfill requirements for quality (ISO 8402).

**Quality Manual** – a document stating the quality policy, quality system, and quality practices of an organization (ISO 8402).

**Quality System** – the organizational structure, responsibilities, procedures, processes, and resources for implementing quality management (ISO 8402).

**Range** – the difference between the largest and smallest observed value of a quantitative characteristic. For quantitative analysis the working range for a method is determined by examining samples with different analyte concentrations and determining the concentration range for which acceptable accuracy and precision can be achieved. The working range is generally more extensive than the linear range. The working range is determined by the analysis of a number of samples of varying analyte concentrations and calculating the regression from the results, usually using the method of least squares. The relationship of analyte response to concentration does not have to be perfectly linear for a method to be effective (AOAC).

**Reference Material** – a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (ISO Guide 30).

**Reference Standard** – a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived (VIM). Generally, this refers to national traceable standards such as those from the National Institute of Standards and Technology (NIST).

**Repeatability (r)** – precision under the *same conditions* (same method, same test item, same operator, same apparatus, same laboratory, short interval of time) (ISO 5725-1).

**Reproducibility (R)** – precision using the *same method* on identical items obtained by operators in different laboratories using different equipment (ISO 5725-1).

**Ruggedness** – the ruggedness of a method is tested by deliberately introducing small changes to the method and examining the consequences. A large number of factors may need to be considered, but because most of these will have a negligible effect, it will normally be possible to vary several at once (AOAC).

**Selectivity** – the extent that a specific analyte can be determined from a complex mixture without interference from the other components in the mixture. A method that is perfectly



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selective for an analyte or group of analytes is said to be specific. The applicability of the method should be studied using various samples, ranging from pure standards to mixtures with complex matrices. In each case the recovery of the analyte(s) of interest should be determined and the influences of suspected interference duly stated. Any restrictions in the applicability of the technique should be documented in the method (AOAC).

**Sensitivity** – the difference in analyte concentration corresponding to the smallest difference in the response of the method that can be detected. It is represented by the slope of the calibration curve and can be determined by a least squares procedure, or experimentally, using samples containing various concentrations of the analyte (AOAC).

**Skill** – ability to apply knowledge effectively and readily in performance (ISO 10015).

**Specific** – see selectivity.

**Standard Operating Procedure** – a document that specifies or describes how an activity is to be performed. It may include methods to be used and sequence of operations (FLAWG).

**Test** - technical operation that consists of the determination of one or more characteristics of a given product, process, or service according to a specified procedure (ISO Guide 2: 1991).

**Traceability** – the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons (VIM).

**Training** – a process to provide and control competence to meet requirements (ISO 10015).

**Validation** - confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled (ISO 8402).

**Verification** - confirmation by examination and provision of objective evidence that specified requirements have been fulfilled (ISO 8402).



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### 4. Management Requirements

#### 4.1 Organization

This section tells you our laboratory has:

1. Appointed a Quality Manager
2. Organized the workforce to achieve quality
3. Provided adequate resources to ensure quality

#### Key Words

Quality Manager  
Organizational Chart  
Authority  
Resources  
Confidential Information  
Proprietary Rights  
Responsibilities  
Undue Pressure

#### Cross-references

ISO 17025:2005 Section 4.1  
ISO 9001:2000 Section 5.1, 5.3, 5.4.1, 5.5.1, 5.5.2, 6.1, 6.2.1, 6.3



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### **4.1.1 Legal Identification / Registration**

Asbestos TEM Laboratories, Incorporated  
630 Bancroft Way, Berkeley, CA 94710  
Voice: 510-704-8930  
Fax: 510-704-8429

### **4.1.2 Laboratory Requirements**

The divisions of Asbestos TEM Laboratories, Inc. have been organized to satisfy the needs of the client and regulatory authorities and to operate to the international standards ISO 17025 and ISO 9001. Asbestos TEM Laboratories, Inc. is composed of the following divisions:

- Ø President's Office
- Ø Administrative Division
- Ø TEM Analysis Division
- Ø PLM Analysis Division
- Ø PCM Analysis Division
- Ø AA Analysis Division

### **4.1.3 Scope of Management System**

The management system covers activities in the laboratory's permanent facility. The fields of activities include the analysis of:

- Ø Air
- Ø building material
- Ø soil
- Ø water
- Ø other samples

for the presence of:

- Ø asbestos
- Ø lead

By

- Ø Transmission Electron Microscopy (TEM)



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- Ø Polarized Light Microscopy (PLM)
- Ø Phase Contrast Microscopy (PCM)
- Ø and Atomic Absorption Spectroscopy (AA)

The laboratory's scope of tests is listed in the Standard Operating Procedures Manual maintained by the Quality Assurance Director.

### **4.1.4 Potential Conflicts of Interest**

The laboratory is not part of a larger organization and thus is not subject to conflicts of interest at the organizational level. Employees and management are subject to the guidelines of Section 4.1.5B of this document regarding personal conflicts of interest.

### **4.1.5 Organization**

#### **A) Management and Technical Personnel**

##### **Policy:**

The laboratory managerial and technical personnel have the necessary authority and resources needed to meet the mandates assigned to their areas irrespective of other responsibilities.

##### **Details:**

Responsibilities are detailed in 4.1.5 (F).

Implementation, maintenance and improvement of the management system are the responsibilities of the President.

Departures from the organizational and management policies in this manual can only be approved by the President.

Departures from quality system procedures can only be approved by the Quality Assurance Director.

Departures from test methods or technical standard operating procedures (SOPs) can only be approved by the Quality Assurance Director.

See also section 5.2.



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### B) Undue Pressure

#### Policy:

Management and personnel are to be free from any undue internal and external commercial, financial and other pressures that may adversely affect the quality of their work. The integrity of test results is the responsibility of all personnel. Management ensures that employees are never instructed or forced to alter or falsify data.

#### Details:

The following list provides some guidelines on how employees avoid conflict of interest situations. Employees shall not:

- Ø falsify records, prepare fraudulent reports, or make false claims
- Ø seek or use privileged or confidential company information, or data from any client, for any purpose beyond the scope of employment
- Ø conduct non-laboratory business on laboratory time, or use company facilities or equipment to conduct outside interests in business, unless prior approval has been obtained
- Ø solicit business on their own behalf (rather than the laboratory) from a client
- Ø be employed by, or affiliated with, organizations whose products or services compete with laboratory products or services
- Ø have employment that negatively affects or interferes with their performance of laboratory duties
- Ø compete with the laboratory in the purchase, sale, or leasing of property or goods
- Ø allow association, family, or friends to influence business decisions to their benefit - decisions must be made on a strictly business basis, always in the best interest of the laboratory
- Ø make any decision that provides gains or benefits to the employee and/or others
- Ø have personal financial dealings with an individual or company that does business with the laboratory which might influence decisions made on the laboratory's behalf

Firm adherence to this code of values forms the foundation of our credibility. Personnel involved in dishonest activities are subject to a range of disciplinary action including dismissal.

### C) Client Confidentiality

#### Policy:

It is the policy of our laboratory to protect the confidential information and proprietary rights of our client including the electronic storage and transmission of results. The procedures are out detailed in SOP 4-13-1 Control of Records.





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### **Details and Procedures:**

All employees sign an Employee Confidentiality Agreement. The signed agreement is retained in each employee's Human Resources file. All employees have read and understand the Laboratory Quality Assurance Manual.

Test results are only released to the client. Release to someone other than the client requires the express permission of the client, except when made in response to a valid order from a State or Federal Court. The release of test results to anyone other than the client requires the permission of management. Laboratory reports are reviewed for accuracy prior to release.

### **D) Operational Integrity**

#### **Policy:**

The laboratory will avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment, or operational integrity.

#### **Details and Procedures:**

To ensure confidence in laboratory operations a formal quality assurance program is implemented. Technical competence is ensured through check sample programs. Impartiality is assessed through audits and approvals. Judgment is ensured through the hiring of qualified personnel and by continuously refining, upgrading and improving his or her skills. Operational integrity is reviewed by management on a regular basis at management review meetings to ensure continued suitability and effectiveness of laboratory policies and procedures. Any problems are acted on immediately through corrective action procedures.

### **E) Organizational Structure**

#### **Policy:**

The organization and management structure of the laboratory and the relationships between management, technical operations, support services, and the quality system is defined through the aid of an organizational chart.

#### **Details:**



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Senior management keeps the most current organizational chart on file. An organizational chart is available with this manual as a reference record and is considered the official record on the date it is marked.

### **F) Responsibility and Authority**

#### **President**

- Ø develops primary goals, operating plans, policies, and short and long range objectives for the laboratory
- Ø directs and coordinates activities to achieve profit and return on capital
- Ø establishes organizational structure and delegating authority to subordinates
- Ø leads the laboratory towards objectives, meets with and advises other executives, and reviews the results of business operations
- Ø determines action plans to meet the needs of stakeholders
- Ø represents organization to major clients, government agencies, shareholders and the public

#### **Laboratory Manager**

- Ø is knowledgeable of the scope of all processes under his or her supervision
- Ø provides the necessary resources (personnel, equipment, supplies) for the quality assurance program, in order to ensure confidence in the laboratory's results
- Ø ensures personnel are trained for the duties they perform - including substitutes when regular personnel are absent
- Ø maintains current job descriptions
- Ø hires and dismisses personnel
- Ø orients new personnel
- Ø determines technical training needs of personnel
- Ø conducts employee performance reviews
- Ø schedules vacation and coverage
- Ø ensures that all health and safety regulations are followed
- Ø ensures that all Human Rights Laws and Regulations are complied with
- Ø oversees quality, standard pricing, customized quotations, and invoicing for tests performed
- Ø ensures equipment is maintained and calibrated, reporting all deficiencies (e.g., equipment malfunctions) in the appropriate manner
- Ø maintains records and manages all aspects of testing activities
- Ø acting in collaboration with the President and appropriate staff scientists, the Laboratory Manager also coordinates research activities in the laboratories, including



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- provides vision and direction for research activities
- develops and reviews research proposals
- ensures adequate training is completed for research personnel
- monitors the progress of research projects
- reviews research reports for clients or publication
- ensures quality policies and procedures are followed
- controls the flow of communication between the client and the laboratory

### **Quality Assurance Director**

- Ø ensures that the Quality System is established, implemented and maintained in accordance with ISO 17025 standards
- Ø manages the internal audit program
- Ø coordinates laboratory accreditation activities
- Ø handles the maintenance and distribution of the Quality Manual and associated documents
- Ø maintains a master list of current versions of quality documentation
- Ø trains personnel on Quality System activities
- Ø monitors the Quality System
- Ø reports on the performance of the Quality System to senior management for review as a basis for improvement of the Quality System
- Ø supervises the laboratory's inter-laboratory proficiency testing program
- Ø writes, edits, revises and approves SOPs

### **Senior Analysts**

- Ø responds to client inquiries and provides professional advice
- Ø prioritizes section's workload
- Ø facilitates operational concerns in their area
- Ø ensures accurate and consistent testing procedures through the validation of all current procedures within their area and by developing, validating and implementing new procedures as delegated by the Laboratory Manager
- Ø coordinates purchasing requests
- Ø edits and writes SOPs and test methods as delegated and supervised by the QA Director
- Ø signs reports when designated with signing authority

### **Senior Analysts and Analysts**

- Ø maintains records of all quality activities as documented in SOPs and test methods
- Ø handles samples and performs analyses according to SOPs and test methods
- Ø maintains and calibrates equipment
- Ø reports deficiencies or malfunction to the supervisor or Laboratory Manager
- Ø identifies and records nonconformance events on *Corrective Action Requests*
- Ø identifies and records potential nonconformance events on *Preventive Action Requests*



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- Ø corrects potential and actual nonconformance event causes
- Ø improves laboratory and/or quality activities on a continuous basis

### Sample Coordinators and Administrative Personnel

- Ø performs work functions and record keeping as per approved SOPs and/or laboratory policies
- Ø identifies and records nonconformance events related to sample receipt, packaging, and Chain of Custody entries on *Sample Receipt/Login Checklist*
- Ø identifies and records potential nonconformance events related to sample receipt, packaging, and Chain of Custody entries on *Sample Receipt/Login Checklist*
- Ø corrects nonconformance events and potential nonconformance events related to sample receipt, packaging, and Chain of Custody entries
- Ø improves laboratory and/or quality activities on a continuous basis

### G) Laboratory Supervision

#### Policy:

Adequate supervision is provided in each area of the laboratory for all testing and calibration personnel, including trainees, by persons familiar with the methods and procedures.

#### Details:

Adequate supervision is ensured through designated supervisors as well as through documentation such as this Quality Manual, test methods and SOPs. A thorough orientation and training program is adhered to for all new employees. Ongoing training for regular personnel is required.

### H) Technical Management

#### Policy:

A technical supervisor is assigned to each major division of the laboratory. They have overall responsibility for the technical operations and the provision of resources needed to ensure the required quality of laboratory operations within that division.

#### Details:

While the technical supervisor may at times delegate duties to other personnel, the technical supervisor is accountable for any nonconforming activities within their division.



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### I) Quality Assurance Director

**Policy:**

The Quality Assurance Director is appointed by the highest level of management. The Quality Assurance Director, who, irrespective of other duties and responsibilities, has defined responsibility and authority for ensuring that the quality system is implemented and followed. The Quality Assurance Director has direct access to the highest level of management where decisions are taken on laboratory policy or resources.

**Details:**

This statement notifies all laboratory personnel that Robert E. Butler is the Quality Assurance Director as authorized below by the President. Any change in this position requires the reissue of this section to all holders of controlled copies of the Quality Manual.

### J) Managerial Substitutions

**Policy:**

Deputies for key personnel are appointed to fulfill the key personnel's duties in their absence.

**Details:**

The Senior Analyst in all Laboratory Divisions has authority to make temporary decisions in the absence of the Quality Assurance Director or Laboratory Manager.

In the event of an extended absence of the Quality Assurance Director, the President will assume his/her responsibilities.

In the event of an extended absence of the Laboratory Manager, the President will assume his/her responsibilities.

Management is responsible for ensuring that current and/or increased workload requirements are met. This includes making adjustments as a result of employee absence. Only fully trained employees are utilized to fulfill the duties of personnel who are absent.



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If sufficient human resources are not available, management will identify the best possible solution to meet operational requirements.

### **K) Personnel Awareness**

#### **Policy and Details:**

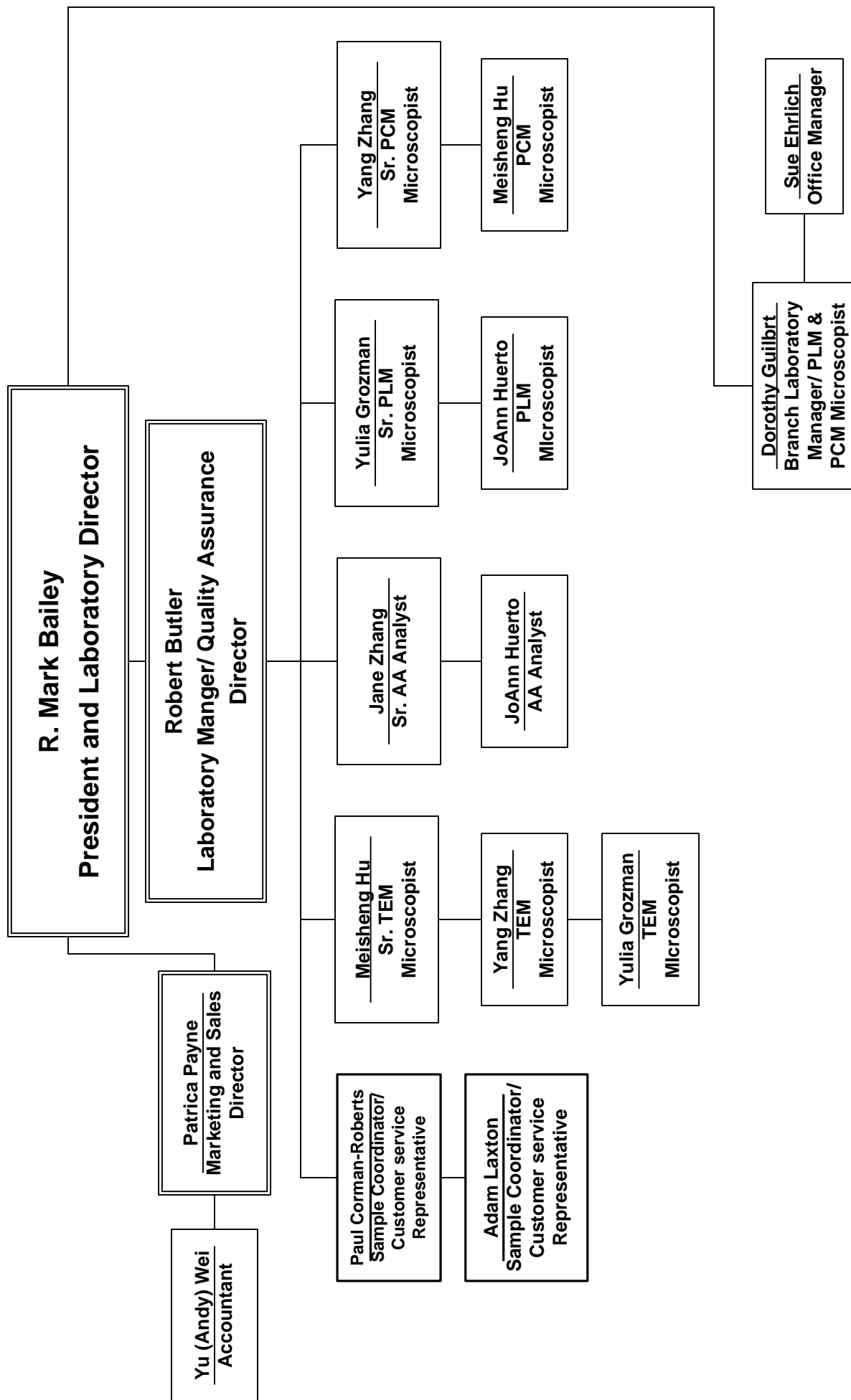
Staff meetings are held regularly to inform the laboratory personnel are aware of the relevance and importance of their activities and how they contribute to the achievement of the objectives of the management system.

#### **4.1.6 Internal Communication**

The President shall ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

# Asbestos TEM Laboratories, Inc.

Organizational Chart  
December 10, 2009





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### 4.2 Quality System

This section tells you that our Quality System is based on the premises:

1. Define your policy on quality
2. Say what you do (through documentation)
3. Do what you say (following your documentation)
4. Record what you did

### Key Words

Establish, Implement, and Maintain  
Policies, Systems, Processes, Programs, Procedures, Instructions  
Communicate, Understand  
Quality Policy Statement  
Quality Manual  
SOP  
Test Method

### Cross-references

ISO 17025:2005 Section 4.2

ISO 9001:2000 Section 4.1, 4.2.1, 4.2.2, 5.1, 5.4.1, 5.4.2, 5.5.1, 6.2.1, 7.1





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### 4.2.1 Policies and Procedures

**Policy:**

The Quality System is established, implemented, and maintained by management. It is applicable to all the fields of testing and activities in which the laboratory is involved and undertakes. All policies, systems, programs, procedures and instructions are documented to the extent necessary to enable the laboratory to assure the quality of results generated. These documents are communicated to, understood by, available to, and implemented by the appropriate personnel.

**Details:**

The purpose of our Quality System is to ensure that all services and products satisfy the client's requirements and have been designed, manufactured, and delivered under controlled conditions.

The effectiveness of the Quality System is assessed in several ways:

- Ø by a program of planned internal audits, covering all aspects of the operation of the quality system
- Ø by regular management reviews of the suitability and effectiveness of the quality system
- Ø by analysis of potential and actual problems as shown by client complaints and supplier and subcontractor assessments
- Ø by other methods approved from time to time by the Technical Quality Manager

This Quality Manual and associated documents (including procedures) and records serve as the quality plan for the laboratory. Other documents and records include:

- Ø standard operating procedures
- Ø quality control plans in test methods
- Ø organizational charts
- Ø proposals
- Ø project management schemes

### 4.2.2 Quality Policy Statement

**Policy:**

The policies and objectives for laboratory operations are documented in this Quality Manual. The overall objectives are set out in the Quality Policy Statement, and are reviewed during Management review. The Quality Policy Statement is issued under the authority of the President on the effective date.

**Quality Policy Statement:**

To ensure accurate and timely *analytical* services and to continuously meet or exceed the



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stated or implied expectations of our clients through day-to-day interactions, Asbestos TEM Laboratories, Inc., has adopted the following Quality Policy:

a) *Management is committed to good professional practice and providing the highest possible quality of services to the client and to continually improve the effectiveness of the management system:* Tests and calibrations are always carried out in accordance with stated standardized methods and clients' requirements. Requests to perform tests that may jeopardize an objective result or have a low validity are rejected.

b) *Standards of service include:*

- Ø Client Satisfaction
- Ø Accuracy
- Ø Timeliness

Management promotes excellence in the workplace by providing all employees with the knowledge, training, and tools necessary to allow for the completion of accurate and timely work.

c) *Personnel:* Asbestos TEM Laboratories, Inc., staff are required to familiarize themselves with quality documentation and to implement the policies and procedures in their work.

d) *Management is committed to complying with ISO 17025 and ISO 9001 international standards, Nist Handbooks 150, 150-3 and 150-13:* the objective of this Quality Manual is to document the compliant policies and associated procedures that are integrated into our daily activities.

Additional objectives include:

- Ø to establish the level of the laboratory's performance
- Ø when possible, to make test method changes to improve performance
- Ø to participate in proficiency testing or quality evaluation programs with peer laboratories
- Ø to ensure that all personnel are trained to a level of familiarity with the quality system appropriate to the individual's degree of responsibility
- Ø to improve and validate laboratory methodologies by participation in method validation collaborative tests
- Ø to establish and report on quality savings



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### 4.2.3 Commitment Evidence

**Policy:**

The President shall provide evidence of commitment to the development and implementation of the management system and to continually improving its effectiveness.

### 4.2.4 Customer/Regulatory Requirements

The President and the Laboratory Manager communicate to the laboratory staff the importance of meeting customer requirements as well as statutory and regulatory requirements.

### 4.2.5 Quality Manual

**Policy:**

This Quality Manual outlines the structure of the documentation used in the quality system. This Quality Manual makes reference to supporting procedures including technical procedures and is maintained up to date.

**Details:**

This quality system is structured in three tiers of documentation. The tiers are as follows

- I. Quality Manual
- II. Standard Operating Procedures and Test Methods
- III. Records

For most clients, this Quality Manual and the associated documents form a general Quality Plan. If necessary, specific Quality Plans will be prepared on a 'per-client' basis. These Quality Plans will modify the general requirements stated in the Manual and associated documents.

All of the above documents are controlled documents under the custodianship of the Quality Assurance Director. Only authorized copies are distributed within the laboratory.

The following records and directive documents are referenced in the Quality Manual, but maintained separately:

- Ø organizational chart (section 4.1.5.E)
- Ø copies of the Quality Policy Statement posted in the laboratory (section 4.2.2)
- Ø identification of resources and management review (section 4.15.1)
- Ø job descriptions (section 5.2.4)



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- Ø statistical techniques (section 5.9)
- Ø test reports (section 4.13.2)
- Ø identification of the laboratory's approved signatures (section 5.10.2)
- Ø laboratory's scope of tests (section 4.1.3)
- Ø equipment inventory and records (sections 5.5.4 and 5.5.5)
- Ø calibration status indicators (section 5.5.8)
- Ø reference standards inventory (section 5.6.3)
- Ø verification records (section 5.9)
- Ø quality control plan / criteria for workmanship (section 5.4.1)
- Ø corrective action records (section 4.12)
- Ø preventive action records (section 4.12)
- Ø client complaint records (section 4.8.1)
- Ø audit schedule and records (section 4.14.3)
- Ø procurement and subcontracting records (sections 4.6 and 4.5.4)
- Ø training records (section 5.2.5)
- Ø master list of documentation (section 4.3.2)
- Ø confidentiality agreements (section 4.1.5 C)
- Ø contract review (section 4.4.2)
- Ø validation of test methods (section 5.4.5)
- Ø facility floor plan (section 5.3.1)

### **4.2.6 Technical Management and the Quality Assurance Director**

The roles and responsibilities for technical management and the Quality Assurance Director are outlined in section 4.1.4 (F) of this manual.

Laboratory Manager ensures that section 5 of this manual is implemented and maintained. The Quality Assurance Director ensures that section 4 of this manual is implemented and maintained.

### **4.2.7 Management System**

The President ensures that the integrity of the management system is maintained when changes to the management system are planned and implemented.



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### 4.3 Document Control

This section tells you that Document Control involves:

1. Writing good procedures
2. Getting them to the users
3. Keeping procedures good

#### Key Words

Controlled Document  
Master List  
Unique Identification  
Revise  
Revision Number  
Effective Date  
Review and Approval  
Obsolete  
Archive  
Hand-written changes

#### Cross-references

ISO 17025:2005 Section 4.3

ISO 9001:2000 Section 4.2.1, 4.2.3



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### 4.3.1 Policies and Procedures

#### **Policy:**

The SOP# 4-3-1 is used to control all quality system documents (internally generated and from external sources). These include documents of external origin, such as regulations, standards, other normative documents, test and/or calibration methods, as well as drawings, specifications, instructions, and manuals.

#### **Details:**

Document means any information or instructions including policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, and plans. These may be in various media, whether hard copy or electronic and they may be digital, analog, photographic or written.

The documents to be controlled include:

- Ø Quality Manual
- Ø Standard Operating Procedures and test methods
- Ø Forms
- Ø Standards

The control of data related to testing and calibration is covered in section 5.4.7. The control of records is covered in section 4.12.

### 4.3.2 Document Approval and Issue

#### 4.3.2.1 Review / Approval / Master List

#### **Policy and Details:**

All documents issued to personnel in the laboratory as part of the quality system are reviewed and approved for use by authorized personnel prior to issue (i.e., reviewed by personnel knowledgeable in the documented activity and then approved by management). A master list identifying the current revision status and distribution of documents in the quality system is readily available in order to preclude the use of invalid and/or obsolete documents (see SOP# 4-3-1). A revision history of documents is also maintained. Documents are formally reviewed on a biennial basis to ensure their continuing suitability.



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### 4.3.2.2 Availability and Obsolete Documents

#### Policy and Details:

The master list shows the current status of all controlled documents. The master list document is organized with the following information:

- Ø Document #
- Ø Title
- Ø Revision #
- Ø Date of issue
- Ø Date of last review
- Ø Locations

Controlled documents are approved before issue.

The SOP# 4-3-1 for document control ensures that:

- Ø authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed
- Ø documents are periodically reviewed and where necessary revised to ensure continuing suitability and compliance with applicable requirements
- Ø invalid or obsolete documents are promptly removed from all points of issue or use to assure against unintended use
- Ø obsolete documents retained for either legal or knowledge preservation purposes are suitably marked (i.e., stamped "OBSOLETE" and dated)

### 4.3.2.3 Identification

#### Policy and Details:

All quality system documentation is identified by:

- Ø date of issue and/or revision number
- Ø page numbering
- Ø total number of pages (e.g., page 5 of 5)
- Ø issuing authority (i.e., approval signature)

## 4.3.3 Document Changes

### 4.3.3.1 Review / Approval

#### Policy:

Changes to documents are reviewed and approved by the same function (i.e., personnel or position) that performed the original review.



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### **Details:**

Developments in policies and procedures require documents to be changed from time to time. Changes to documents receive the same level of review and approval as the originals.

The Quality Manual is reviewed annually by the Quality Manager. Records are kept of this review.

Test methods and SOPs are reviewed on a biennial basis. Procedures for this are outlined in SOP# 4-3-1.

Obsolete documents are withdrawn, but are retained for archive purposes and clearly labeled as obsolete.

### **4.3.3.2 Identification of Changes**

#### **Policy:**

The nature of document changes is identified in the document.

#### **Details:**

As outlined in SOP# 4-3-1.

In general, the nature of changes is identified in the document with a vertical bar in the left-hand margin. Revision history is recorded at the end of the document.

### **4.3.3.3 Amendments by Hand**

#### **Policy and Details:**

Hand-written amendments to documents are not permitted, however Analysts and Senior Analysts may make hand-written entries in printed copies of the Quality Manual which indicate suggested revisions or improvements in the manual. Such hand-written entries do not become part of the approved text of the Manual until the document is formally revised and re- issued according to the procedures in SOP# 4-3-1.

### **4.3.3.4 Computerized Documents**

#### **Policy and Details:**

The SOP# 4-3-1 details how changes in documents maintained in computerized systems are made and controlled.





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### 4.4 Review of Requests, Tenders, and Contracts

This section tells you that we must:

1. Clearly understand client requirements

#### Key Words

Requirements  
Subcontractor  
Request  
Tender  
Contract  
Review

#### Cross-references

ISO 17025:2005 Section 4.4

ISO 9001:2000 Section 5.2, 7.2.1, 7.2.2, 7.2.3



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### 4.4.1 Policies and Procedures

#### **Policy:**

The SOP# 4-4-1 is used to review requests, tenders, or contracts. This procedure ensures that:

- a) the client requirements including the methods to be used are adequately defined, documented and understood (see section 5.4.2)
- b) the laboratory has the capability and resources to meet the requirements
- c) the appropriate test method(s) is(are) selected and capable of meeting the client's requirements (see section 5.4.2)

Any differences between the request or tender and the contract are resolved before any work commences. Each contract must be acceptable by both the laboratory and the client.

#### **Details:**

The request, tender and contract review is conducted in a practical and efficient manner, and the effect of financial, legal, and time schedule aspects are taken into account.

The review of capability establishes 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 in question. The review may also encompass results of earlier participation in inter- laboratory comparisons or proficiency testing and/or the running of trial test programs using samples or items of known value in order to determine uncertainties of measurement, limits of detection, and confidence limits.

The contract review ensures that each client's requirements are adequately defined and documented before the service or product is ordered or dispatched. This should ensure that any order, once accepted, can be completed without delay, and that the client's requirements including delivery date, technical specification, and cost can be met.

If the contract review highlights any ambiguities or uncertainties then the client will be contacted and the problem resolved before the order is accepted. All review notes and correspondence with the client will be documented.

The SOP# 4-4-1 also describes the activities that take place should there be a subsequent amendment to a client's order.

Typical types of contracts include:

- Ø approved service quotations
- Ø confidentiality agreements
- Ø non-disclosure agreements
- Ø sample submission requests



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- Ø memorandum of agreement
- Ø memorandum of understanding
- Ø research proposals and contracts
- Ø verbal orders (oral agreements)
- Ø activity plans

### 4.4.2 Records of Review

**Policy:**

Records of request, tender and contract review, including significant changes, are maintained. Records of pertinent discussions with a client relating to the client's requirements or the work during the period of execution of the contract are also maintained.

**Details:**

For review of routine and other simple tasks, the date and the identification (e.g., 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 grant of the contract for on-going routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged. For new, complex or advanced testing tasks, a more comprehensive record is maintained.

### 4.4.3 Review of Subcontracted Work

**Policy:**

Request, tender, and contract review also includes work that is subcontracted by the laboratory.

**Details:**

Subcontractor laboratories are reviewed as described in section 4.5.

### 4.4.4 Notification of Client

**Policy and Details:**

Clients are informed of deviations from the contract. This is typically communicated to the client prior to the performing the deviation.

### 4.4.5 Contract Amendment

**Policy and Details:**



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If a contract needs to be amended after the work has commenced, the same contract review process is repeated and any amendments are communicated to all affected personnel.

### 4.5 Subcontracting of Tests and Calibrations

This section tells you that we must:

1. Know what tests and calibrations need to be done by another laboratory
2. Check out the other laboratories

### Key Words

Competence  
Register of Subcontractors  
Assessment

### Cross-references

ISO 17025:2005 Section 4.5

ISO 9001:2000 Section 7.4.1, 7.4.3, 8.2.4



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### 4.5.1 Subcontractor Competence

**Policy:**

Work that must be subcontracted due to:

- Ø equipment failure
- Ø workload
- Ø large contracts
- Ø contracts requiring some extra technical expertise
- Ø unforeseen circumstances

is subcontracted to a technically competent laboratory.

**Details:**

The subcontracted laboratory demonstrates technical competence by possession or receipt of one or more of the following:

- Ø accreditation by the NIST-NVLAP or other recognized body covering the test methods used for the subcontracted work
- Ø registration under the ISO 9001 standard
- Ø satisfactory performance of appropriate quality control check samples (certified reference material, in- house reference material or replicate analysis)
- Ø audit of the subcontractor's quality system by our auditors

It is the responsibility of the Technical Quality Manager to assess and approve the competence level of subcontractor laboratories.

### 4.5.2 Client Approval

**Policy:**

Clients are advised of work (or any portion thereof) that is being subcontracted to another laboratory and their approval is obtained (preferably in writing).

**Details:**

Clients are advised of subcontracted work through fee schedules or any type of contract listed in section 4.4.1.

### 4.5.3 Assurance of Subcontractor Competence

**Policy:**



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The laboratory is responsible to the client for the subcontractor's work. Technical competence of subcontractor laboratories is demonstrated through various records.

**Note** – there may be circumstances where the client specifies which subcontractor is to be used. In such cases we may not be able to demonstrate the competence of the subcontractor and therefore are not responsible for the results.

**Details:**

Records of subcontractor competence include, but are not limited to, the following:

- Ø accreditation certificates or documentation
- Ø registration certificates
- Ø check sample results
- Ø audit results
- Ø approval by the Quality Assurance Director

### **4.5.4 Subcontractor Register**

**Policy:**

A register of all subcontractors performing tests and calibrations is maintained.

**Details:**

The approved register of subcontractors and all assessment records are maintained by the Quality Assurance Director.



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### 4.6 Purchasing Services and Supplies

This section tells you that we must:

1. Know what we want
2. Check out our suppliers

#### Key Words

Selection  
Verify  
Specifications  
History

#### Cross-references

ISO 17025:2005 Section 4.6

ISO 9001:2000 Section 7.4, 8.2.4



### 4.6.1 Policies and Procedures

**Policy:**

The SOP# 4-6-1 is used to select and purchase services and supplies. The SOP# 4-6-1 is used for procurement, reception, and storage of supplies.

**Details:**

Consumable materials are stored according to the appropriate test method, SOP, or work instruction.

### 4.6.2 Specifications

**Policy:**

Only services and supplies of the required quality are used. These quality requirements are detailed in laboratory SOPs under the “*Materials Required*” section and will identify the appropriate minimum specifications when necessary.

**Details:**

Packing slips are checked against package content labels and matched with the Request for Purchase or Purchase Order if accepted. Once accepted, the packing slip is dated and initialed as evidence of compliance. Certificates of analysis (COA) are maintained on file after the COA is checked to ensure the received item meets minimum specifications.

Chemicals are purchased with manufacturer’s certificates where possible. Uncertified chemicals are purchased from ISO 9000 registered companies. Whatever the source, the laboratory verifies the quality of the standards by comparing the new batch of standards to the old. Due regard is paid to the manufacturer’s recommendations on storage and shelf life.

Wherever possible, reagents are purchased from manufacturers who have a quality system based on ISO 9000. The grade of any reagent used (including water) is stated in the method together with guidance on any particular precautions to be observed in its preparation or use.

Where no independent assurance of the quality of procured goods or services is available or the supplier’s evidence is insufficient the laboratory ensures that purchased goods and services comply with specified requirements. Where possible and practical the laboratory ensures that goods are inspected, calibrated, or are otherwise in compliance with any standard specification relevant to the calibrations or tests concerned.





### 4.6.3 Purchasing Documents

**Policy:**

Purchasing requests are recorded on the Request for Purchase form and contain data describing the product ordered. The Request for Purchase is reviewed and approved for technical content prior to release.

**Details:**

The description may include type, class, grade, precise identification, specifications, drawings, inspection instructions, other technical data including approval of test results, quality required and quality system standard under which they were produced.

The completion of the Request for Purchase is the responsibility of the originator, normally the Senior Analyst. They review the Request for Purchase for accuracy and approve the technical content prior to release with their signature and the date.

### 4.6.4 Approved Suppliers

**Policy:**

Suppliers of critical services are evaluated and approved before use. An approved supplier list is maintained.

**Details:**

Audits or tender evaluation is conducted to qualify suppliers of critical services prior to use. The criteria for evaluation may include, but is not limited to the following:

- Ø references
- Ø accreditation
- Ø formal recognition

The records are maintained by purchasing personnel.



### 4.7 Service to the Client

This section tells you that we must:

1. Facilitate clarification of the client's request
2. Give client access to relevant testing area
3. Maintain client contact
4. Inform client of delays or deviations
5. Utilize client surveys

### Key Words

Clarification  
Deviations  
Delays  
Client Satisfaction Survey  
Advice

### Cross-references

ISO 17025:2005 Section 4.7

ISO 9001:2000 Section 7.2.1, 7.2.3, 7.4.3, 7.5.1



### 4.7.1 Services to the Client

**Policy:**

Client requests are clarified for the clients or their representatives. Furthermore the client or their representative will be afforded the right to monitor the performance of the laboratory in relation to the work performed.

**Details and Procedures:**

Service to the client includes:

- Ø Affording the client or the client's representative reasonable access to relevant areas of the laboratory for the witnessing of work performed for the client; it is understood that such access should not conflict with rules of confidentiality of work for other clients or with safety.
- Ø Preparing, packaging, and dispatching of test items needed by the client for verification purposes.
- Ø Maintaining of open contacts. The client values advice and guidance in technical matters, and opinions and interpretations based on results. Contact with the client, especially in large assignments, should be maintained throughout the work. The laboratory should inform the client of any delays or major deviations in the performance of the tests.

### 4.7.2 Customer Feedback

**Policy and Details:**

The laboratory obtains feedback from the client. Positive and negative feedback can be obtained passively through ongoing communications with the client or actively through client satisfaction surveys. The surveys are distributed by e-mail and by hand in the lobby. The feedback is used to improve the quality system, testing activities, and client service.



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### 4.8 Complaints

This section tells you that we must:

1. Maintain records of Complaints
2. Maintain records of Corrective Action

### Key Words

Resolving  
Investigation  
Corrective Action  
Follow-up Verification  
Root Cause

### Cross-references

ISO 17025:2005 Section 4.8

ISO 9001:2000 Section 7.2.3



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### 4.8.1 Policies and Procedures

**Policy:**

The SOP# 4-8-1 is used for resolving complaints received from clients or other parties. Records are maintained of all complaints and follow-up.

**Details:**

Records of complaints include the following information:

- Ø details of the complaint
- Ø investigation
- Ø corrective action(s)
- Ø root cause analysis
- Ø follow-up verification

See also section 4.12.

All personnel are responsible for recording and responding to complaints.



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### 4.9 Control of Nonconforming Testing and Calibration Work

This section tells you that we must:

1. Stop testing when nonconforming work is identified
2. Figure out what is causing nonconforming work

#### Key Words

Nonconforming  
Root Cause

#### Cross-references

ISO 17025:2005 Section 4.9

ISO 9001:2000 Section 5.5.1, 7.4.3, 7.5.1, 8.2.4, 8.3



### 4.9.1 Procedures to Control Nonconforming Work

**Policy:**

The SOP# 4-9-1 is used to control any aspect of testing and/or calibration work, or the results of this work, when they do not conform with the test methods or the agreed requirements of the client.

**Details:**

The procedure ensures that:

- Ø Responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports as necessary) are defined and taken into consideration when nonconforming work is identified
- Ø an evaluation of the significance of the nonconforming work is made
- Ø remedial actions are taken immediately, together with any decision about the acceptability of the nonconforming work
- Ø where necessary, the client is notified and the work is recalled
- Ø the responsibility for authorizing the resumption of work is defined

Identification of nonconforming work or problems with the quality system or with testing activities can occur at various locations within the quality system and technical operations such as:

- Ø client complaints
- Ø quality control
- Ø instrument calibration
- Ø checking of consumable materials
- Ø staff observations or supervision
- Ø test report checking
- Ø management reviews
- Ø internal or external audits

### 4.9.2 Root Cause Analysis

**Policy:**

Where evaluation indicates that 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 4.12 are followed to identify the root cause(s) of the problem and to eliminate this (these) cause(s).



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### **Details:**

The SOP# 4-12-1 outlines recording the root cause analysis for investigating nonconforming work.

Situations warranting corrective action investigation include:

- Ø failure to comply with test method including all applicable procedures necessary to ensure the integrity and representative nature of the sample
- Ø presentation of uncertain knowledge as to compliance with test methods including all applicable procedures necessary to ensure the integrity and representative nature of the sample
- Ø failure or suspected failure in method performance as demonstrated by results provided by quality control samples
- Ø lack of relevant evidence provided by quality audit, proficiency testing, or client feedback
- Ø lack of relevant evidence provided by data validation
- Ø neglect to check the inherent property of the sample that compromises the testing





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### **4.10 Improvement**

This section tells you that we must:

1. Continually improve the effectiveness of our management system

### **Key Words**

Improving  
Management System

### **Cross-references**

ISO 17025:2005 Section 4.10



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### **4.10 Policies and Procedures**

**Policy:**

The laboratory shall continually improve the effectiveness of our management system.

**Details:**

Management system is continually improved through the use of the follows:

- Ø quality policy
- Ø quality objectives
- Ø audit results
- Ø analysis of data
- Ø corrective and preventive actions
- Ø management review



## 4.11 Corrective Action

This section tells you that we must:

1. Identify problems
2. Determine why the problem occurred
3. Fix the cause of the problem
4. Verify that your changes worked

### Key Words

Corrective Action Request  
Root Cause  
Monitor  
Audit  
Nonconforming work

### Cross-references

ISO 17025:2005 Section 4.11

ISO 9001:2000 Section 5.5.1, 8.1, 8.2.2, 8.2.3, 8.4, 8.5.2, 8.5.3



### 4.11.1 General

**Policy:**

The SOP# 4-11-1 is utilized for implementing corrective action when nonconforming work or departures from policies and procedures in the quality system or technical operations have been identified. The procedure requires that appropriate authority be designated for the implementation of corrective actions. The procedure includes cause analysis, selection and implementation of corrective action, and monitoring of actions.

**Details:**

Problems with the quality system or 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, feed-back from clients, or staff observations.

Corrective action investigations are documented and required changes to operational procedures are implemented. The corrective action request (CAR), investigation and resolution are recorded on a CAR form.

### 4.11.2 Cause Analysis

**Policy:**

Corrective action always begins with an investigation to determine root cause(s) of the problem (see SOP# 4-11-1).

**Details:**

Potential causes of the problem could include client requirements, the samples, sample specifications, methods and procedures, personnel skills and training, consumable materials, or equipment and its calibration.

### 4.11.3 Selection and Implementation of Corrective Actions

**Policy and Details:**

After determining the cause(s) of the problem, potential corrective actions are identified. The most likely action(s) (this includes practical and/or reasonable) are selected and implemented to eliminate the problem and to prevent recurrence. It should be noted that any corrective actions taken to eliminate the cause(s) of nonconformance events or other departures are to an appropriate degree to address the magnitude of the problem and should be commensurate with the risks encountered. (Note – in plain language, this means determine whether the benefit outweighs the cost.) Controls are applied to prevent



recurrence. The laboratory documents and implements the required changes resulting from corrective action investigations.

## 4.11.4 Monitoring of Corrective Action

### **Policy:**

After implementing the corrective action(s), the laboratory monitors the results to ensure that the actions taken have been effective in overcoming the problems originally identified.

### **Details:**

Monitoring is assigned to an appropriate individual such as the originator of the CAR or the originator's manager. Changes resulting from corrective action are documented.

## 4.11.5 Additional Audits

### **Policy:**

Where the identification of nonconformance events or departures casts doubts on compliance of policies, procedures, regulations, international quality standards, the appropriate areas of activity are promptly audited in accordance with section 4.13.

### **Details:**

Special audits follow the implementation of corrective actions to confirm their effectiveness. A special audit is only necessary when a serious issue or risk to the business is identified. Special audits are carried out by trained and qualified personnel who are (whenever resources permit) independent of the activity to be audited. See section 4.13 for more details.



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### 4.12 Preventive Action

This section tells you that we must:

1. Identify potential problems
2. Determine why the problem could occur
3. Fix the cause of the potential problem
4. Verify that your changes worked

### Key Words

Preventive Action Request  
Potential Nonconformance  
Action Plan

### Cross-references

ISO 17025:2005 Section 4.12

ISO 9001:2000 Section 8.4, 8.5.2, 8.5.3



## 4.12.1 Preventive Action Identification

### Policy:

Opportunities for needed improvement and potential sources of nonconformance, either technical or with the quality system shall be identified. If action is required, action plans are developed, implemented and monitored, to reduce the likelihood of occurrence of such nonconformance events and to take advantage of the improvement opportunities.

### Details:

Records of preventive action include the following information:

- Ø details of potential non-conformances
- Ø investigation
- Ø preventive action
- Ø follow-up verification

These records are maintained in the files of the Quality Assurance Director.

## 4.12.2 Preventive Action Plans

### Policy:

The preventive action procedure includes the initiation of such actions and application of controls to ensure that they are effective.

### Details:

Preventive action may result from the review of operational procedures and analysis of data. Analysis of data includes trend analysis, analysis of proficiency testing results, and risk analysis.

The SOP# 4-12-1 is utilized to implement opportunities for needed improvement and prevent potential sources of non-conformances.



## 4.13 Control of Records

This section tells you that we must:

1. Identify the records to be kept
2. Keep identified records in a useful state
3. Destroy records when they are no longer needed

### Key Words

Collection  
Indexing  
Access  
Storage  
Maintenance  
Disposition  
Legible  
Traceable  
Retrievable  
Secure

### Cross-references

ISO 17025:2005 Section 4.13

ISO 9001:2000 Section 4.2.4, 6.3, 6.4, 7.1, 7.5.1, 7.5.2, 7.5.3, 8.1, 8.2.2, 8.2.3, 8.2.4





## 4.13.1 General

### 4.13.1.1 Procedures

#### Policy:

The SOP# 4-13-1 is used to identify, collect, index, access, file, store, maintain, protect, backup, and dispose quality and technical records. Quality records include reports from internal audits and management reviews as well as corrective and preventive action records.

#### Details:

Records are available to demonstrate conformance to requirements and effective operation of the Quality System. Quality records from suppliers are also controlled.

All records, including test reports, are safely stored and held secure in locked areas, and in confidence to the client. Records are maintained in designated storage locations either on-site or off-site for a minimum of ten years, beginning in December of 2004. Prior to this date, records management and retention practices were inconsistent. Older documents are available for varying periods but all records are now being retained for ten years.

The master list of records is organized with the following information:

- Ø Record No. / Form No.
- Ø Record Name
- Ø Filing Method (loose forms filed monthly, quarterly, semi-annual, annual or electronic)
- Ø Active Files (files referred to within the work area) / Retention Period / Location
- Ø Inactive Files (files referred to but not often and kept in storage) / Retention Period / Location
- Ø Persons / Positions Responsible / Users

The dating format for records is MM/DD/YYYY.

### 4.13.1.2 Record Integrity

#### Policy:

All records are to be legible and shall be 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.

#### Details:



The retention times for records are generally set at ten years.

Records may be in the form of any type of media, such as hard copy or electronic media.

#### **4.13.1.3 Record Security**

**Policy:**

All records are held secure and in confidence.

**Details:**

Access to records is secured through locked rooms and filing cabinets.

#### **4.13.1.4 Record Backup**

**Policy:**

The SOP# 4-13-1 is followed to protect and backup data/records held on computers at all times and to prevent unauthorized access to or amendment of data/records on computers.

**Details:**

Data is password protected.

Backups ensure integrity and availability of data / information in the event of a system / power failure.

### **4.13.2 Technical Records**

#### **4.13.2.1 Record Information**

**Policy:**

Original observations, calculations, derived data and sufficient information to establish an audit trail, calibration records, personnel records and a copy of each test report issued will be retained for ten years, beginning in December of 2004. Prior to this date, records management and retention practices were inconsistent. Older documents are available for varying periods but all records are now being retained for ten years.

The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the test uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original. The records include the identity of personnel responsible for sampling, performing of each test and/or calibration and checking of results.

**Details:**

Technical records are accumulations of data (see 5.4.7) and information that 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, note books, instrument printouts, magnetic media, check sheets, work notes, control graphs, test reports, calibration certificates, client's notes, papers and feedback, and test reports to clients.

The records for each test contain sufficient information to permit its repetition. Records include:

- Ø date of sampling
- Ø sample receipt
- Ø sample handling, storage, and disposal
- Ø identification of personnel
- Ø analyst proficiency
- Ø equipment identification and performance
- Ø calibration records
- Ø media performance, where appropriate
- Ø test organism batch # or lot #, where appropriate
- Ø results
- Ø reports (mailed, faxed)
- Ø review

*Note* – the above records may be stored in separate locations. They are cross-referenced for easy retrieval.

#### 4.13.2.2 Recording

**Policy:**

Observations, data, and calculations are clearly and permanently recorded and identifiable to the specific job at the time they are made.

**Details:**

Handwritten records must be legible and made with indelible ink immediately after an observation, after data is collected and/or after calculations are made.

#### 4.13.2.3 Corrections to Records

**Policy:**



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Changes to test data are made so as not to obscure or delete the previous data entry.

### Details:

Mistakes are lined out with a single stroke and the correct value entered alongside.

Mistakes are not erased, made illegible, or deleted. All alterations to records are signed or initialed and dated by the person making the correction. In the case of computer-collected data, similar measures are taken to avoid loss or change of original data.

## 4.14 Internal Audits

This section tells you that:

1. Trained internal auditors examine our internal operations for quality
2. Auditors report the results to those in charge
3. We must correct any areas that need fixing

## Key Words

Schedule  
Elements  
Independent  
Nonconformance  
CAR

## Cross-references

ISO 17025:2005 Section 4.14

ISO 9001:2000 Section 8.1, 8.2.2, 8.2.3



## 4.14.1 Internal Audit Program

### Policy:

The internal audit program involves periodic audits conducted according to a predetermined schedule for each year. This program is defined on an annual basis and conducted as outlined in this section with further details found in SOP# 4-14-1. All elements of this Quality Manual will be audited each year and all relevant laboratory records are available to personnel conducting the audit. These audits are performed to verify operations continue to comply with the requirements of this Quality Manual and are effective.

### Details:

The Quality Manual, test procedures, and laboratory results are verified for compliance. It is the responsibility of the Quality Assurance Director to plan and organize audits as required by the schedule and requested by management. Audits are carried out by trained and qualified personnel who are wherever resources permit independent of the activity to be audited. Personnel are not to audit their own activities except when it can be demonstrated that an effective audit will be carried out (see also 4.14.5). Audits are performed through the aid of a checklist prepared in advance to minimize the possibility of overlooking any details during the audit.

Generally, the types of audits include:

- Ø quality management system
- Ø processes and procedures
- Ø products, services, and reports

## 4.14.2 Corrective Action

### Policy:

When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of test or calibration results, timely corrective action is taken and clients are notified if investigations show that laboratory results may have been affected.

### Details:

Non-conformities that can be resolved easily are to be corrected immediately, ideally during the audit. Records are made on the audit checklist. Non-conformances that require a more involved resolution are recorded on a CAR and resolved as described in section 4.13.



Corrective actions and client modifications must be kept on record for each audit deviation that casts doubt as described in this section.

## 4.14.3 Records and Management

### Policy:

Records are made of the activity being audited, the audit findings, and corrective actions that arise. Management ensures that corrective actions are discharged within an appropriate and agreed timeline.

### Details:

A report is prepared by the auditors and distributed to those audited and/or the area manager/supervisor within an appropriate and agreed timeline. The audit report may include the following sections, as appropriate:

- Ø audit objective and scope
- Ø area or section audited
- Ø personnel involved – auditors and auditees
- Ø date of audit
- Ø reference documents
- Ø observations including non-conformance events and commendations
- Ø opening and closing meetings
- Ø recommendations
- Ø audit report distribution

The appropriate manager is responsible for ensuring that corrective actions are sufficiently recorded. Follow-up is performed by the auditor and recorded when corrective action is complete and deemed effective. The audit records are kept in the laboratory.

## 4.14.4 Follow-up Audits

### Policy:

Follow-up audits are performed to verify and record the implementation and effectiveness of the corrective action taken.

### Details:

The follow-up audit is performed at a mutually acceptable time between the area implementing corrective action and the auditor. This time is determined when the CAR is issued.



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### 4.15 Management Reviews

This section tells you that management must:

1. Periodically review technical competence and client satisfaction
2. Keep records of reviews
3. Ensure follow-up is executed
4. Measure progress

### Key Words

Supervisor Reports  
Audit Reports  
CAR / PAR  
Proficiency Results  
Client Satisfaction Survey  
Resources

### Cross-references

ISO 17025:2005 Section 4.15

ISO 9001:2000 Section 5.1, 5.4.2, 5.6, 6.2.1, 7.1, 8.5.1



## 4.15.1 Review of Quality System and Testing

### Policy:

Management periodically (at least annually) and in accordance with a predetermined schedule and SOP# 4-15-1, conduct a review of the laboratory's quality system and testing and/or calibration activities to ensure their continuing suitability and effectiveness and to introduce any necessary changes or improvements.

### Details:

The review takes account of:

- Ø suitability of policies and procedures
- Ø reports from managerial and supervisory personnel
- Ø the outcome of recent internal audits
- Ø corrective and preventive actions
- Ø assessments by external bodies
- Ø results of inter- laboratory comparisons or proficiency tests
- Ø changes in the volume and type of work undertaken
- Ø feedback from clients, including complaints and client satisfaction surveys
- Ø other relevant factors, such as quality control activities, resources and personnel training
- Ø recommendations for improvement

A minimum period for conducting a management review is once a year. Results of the review feed into the laboratory planning system and include goals, objectives and action plans for the coming year.

A management review can be supplemented by consideration of related subjects at regular management meetings.

## 4.15.2 Findings, Actions, and Records

### Policy and Details:

Findings from management reviews and the actions that arise are recorded in the minutes of the meeting. Management will ensure that the actions are discharged within an appropriate and agreed timeline.





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### 5.1 General

This section informs you that:

1. Many factors contribute to the correctness and reliability of tests and/or calibrations
2. The laboratory must account for these factors

### Key Words

Correctness  
Reliability  
Uncertainty

### Cross-references

ISO 17025:2005 Section 5.1  
ISO 9001:1994 Section N/A  
ISO 9001:2000 Section N/A



## 5.1.1 Correctness and Reliability

### Policy and Details:

Correctness and reliability of the tests and/or calibrations performed have many contributing factors including:

- Ø human factors (see section 5.2)
- Ø accommodation and environmental conditions (see section 5.3)
- Ø test and calibration methods and method validation (see section 5.4)
- Ø equipment (see section 5.5)
- Ø measurement traceability (see section 5.6)
- Ø sampling (see section 5.7)
- Ø handling of test and calibration items (see section 5.7)

## 5.1.2 Measurement Uncertainty

### Policy:

When developing test and calibration methods and procedures, total measurement uncertainty must be accounted for in the training and qualification of personnel, and in the selection and calibration of equipment.

### Details:

The extent to which the factors contribute to total measurement uncertainty differs between tests and between calibrations.

See section 5.4.6 for more details.



### 5.2 Personnel

This section tells you that management:

1. Analyzes training needs
2. Provides training to employees for them to do their jobs
3. Qualifies people performing specific tasks

### Key Words

Competence  
Qualification  
Authorize  
Training Needs  
Job Description  
Registry of Skills

### Cross-references

ISO 17025:2005 Section 5.2

ISO 9001:2000 Section 5.5.1, 6.2.1, 6.2.2, 7.5.1



### 5.2.1 Competence and Qualification

**Policy:**

Management ensures the competency of all specific equipment operators, those performing tests and/or calibrations, those evaluating results and sign test reports. Appropriate supervision is provided for employees undergoing training. Personnel performing specific tasks are qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

In addition, personnel responsible for the opinions and interpretations included in test reports also have:

- Ø relevant knowledge of the technology used for the manufacturing of the items, materials, products tested, or the way they are used or intended to be used and of the defects or degradation that may occur during or in service
- Ø knowledge of the general requirements expressed in the legislation and standards
- Ø an understanding of the significance of deviations found with regard to the normal use of the items, materials, or products concerned

**Details:**

Management defines the minimum levels of qualification and experience necessary for all posts within the laboratory.

Continued competence is monitored and where this is not achieved, the need to retrain personnel is considered. Where a method or technique is not in regular use, verification of personnel performance before they undertake tests, may be necessary.

### 5.2.2 Training Policies and Procedures

**Policy:**

Management will formulate the goals with respect to the education and the skills of the laboratory personnel. The training program is relevant to the present and anticipated tasks of the laboratory. SOP# 5-2-1 is utilized to identify training needs and providing the necessary training for personnel.

**Details:**

The skills and knowledge are defined in the job description for each job function as described in section 5.2.4. Management compares the job description to the skills and knowledge of the new incumbent to determine the training needs.



Training in the laboratory must include all methods or parts of methods and techniques that personnel are asked to perform. Minimally, the analyst must demonstrate competency by observation by management and verification using replicate and/or check samples. For technicians who perform only parts of the method, confirmation of competency may be verified by observation only. Re-verification of all personnel must be performed annually on all methods or techniques pertinent to their job description.

In some cases it may be appropriate to define competence related to a particular technique or instrument rather than methods. If so, it will be necessary to define for each method, the necessary technique-based competence required together with any additional requirements.

### 5.2.3 Employees

**Policy:**

Competent permanent or contractual employees are employed in the laboratory. The Laboratory Manager ensures that contractual, additional technical employees, and key support personnel are supervised and work in accordance to the policies and procedures of this Quality Manual.

**Details:**

Testing must be either performed or supervised by an experienced person qualified to degree level. Personnel have relevant practical work experience (at least 2 years) before being allowed to perform accredited work.

### 5.2.4 Job Descriptions

**Policy:**

Current job descriptions for managerial, technical and key support personnel involved in tests and/or calibrations are maintained centrally in the administration area of the laboratory.

**Details:**

Minimum contents of job descriptions include:

- Ø the duty of performing tests and/or calibrations
- Ø the act of planning tests and/or calibrations and evaluation of results
- Ø the responsibility of developing and validating new methods as / when requested
- Ø expertise and experience
- Ø qualifications and training programs
- Ø managerial duties



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Job descriptions are dated and signed to demonstrate that each incumbent has read it and is in agreement. They are maintained current in the personnel record.

### 5.2.5 Authorized Personnel

#### **Policy:**

Management authorizes specific personnel to perform particular types of sampling, test and/or calibration, to issue test reports, to give opinions and interpretations and to operate particular types of equipment. Records of the relevant competence, educational and professional qualifications, training, skills and experience of all technical personnel and contracted personnel are maintained. This information is readily available and includes the date on which authorization and/or competence was confirmed and the criteria on which the authorization is based and the confirming authority.

#### **Details:**

The purpose of these records is to provide evidence that personnel have been adequately trained and their competence to perform particular tests has been assessed. In some cases it may be pertinent to state any particular limitations to competence. The records are maintained in a registry of skills and include:

- Ø academic and professional qualifications
- Ø external and internal courses attended
- Ø relevant on-the-job training and retraining as necessary (i.e., demonstration of competence)
- Ø skills and experience (i.e., resume)
- Ø relevant authorizations

Records are held centrally in the administration area.



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### 5.3 Accommodation and Environmental Conditions

This section tells you:

1. That laboratory facilities are suitable for attaining correct performance of tests and calibrations
2. Critical environmental conditions are monitored, controlled and recorded
3. Incompatible activities are separated
4. Access to laboratories is controlled
5. Good housekeeping is practiced

#### Key Words

Incompatible activities  
Prevent cross-contamination  
Controlled access

#### Cross-references

ISO 17025:2005 Section 5.3

ISO 9001:2000 Section 6.3, 6.4, 7.1, 7.5.1, 7.5.2, 7.6, 8.2.3



## 5.3.1 Facility

### **Policy:**

Laboratory facilities are appropriate to attain correct performance of tests and/or calibrations. This may include, but not limited to, energy sources, lighting, heating, ventilation and any other environmental conditions.

Appropriate care is taken to ensure that the environment does not invalidate the results or adversely affect the required quality of any measurement. Particular care is taken when sampling, tests and/or calibrations are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of tests and calibrations are documented.

### **Details:**

This section deals with the test areas in the laboratory and premises for support such as sample receipt and storage. Central laboratory supplies and services, such as water purification systems, air supply, vacuum source, and sample storage, are appropriate to facilitate proper performance of tests.

## 5.3.2 Monitoring

### **Policy:**

Critical environmental conditions are monitored, controlled and recorded as required by the relevant specifications, methods, and procedures or where they may influence the quality of the results. Due attention is paid, for example, to dust and air quality as appropriate to the technical activities concerned. Tests and calibrations are stopped when the environmental conditions jeopardize the results of the tests and/or calibrations.

### **Details:**

Bench tops and floors are made of impervious, smooth, easily cleaned materials. There is at least two linear meters workspace per analyst while working. Walls and ceilings are made of materials that are smooth and easily cleaned.

All work surfaces where samples are handled for login, preparation, and analysis are cleaned daily by wet wiping. In addition, wipe samples are taken and analyzed on a quarterly basis.





### 5.3.3 Separation of Incompatible Activities

**Policy:**

Effective separation between neighboring areas is made when the activities are incompatible. Measures are taken to prevent cross-contamination.

**Details:**

Reference materials and certified reference materials must be kept separated from samples (log-in and storage). Sample log-in and storage must be segregated, ideally in a separate area from the testing laboratory, and include proper sanitation to exclude the possibility of cross-contamination. Segregation of activities is achieved through time and space allocations.

An example of space segregation would be for a trace analysis. Physical separation of the trace analysis from high-level analysis is achieved through the use of separate rooms.

An example of time segregation would be the coordination of activities at different times. It may be appropriate to perform work on “cleaner” samples first before starting “dirtier” type samples.

### 5.3.4 Controlled Access

**Policy:**

Access to and use of areas affecting quality of the tests and/or calibrations is defined and controlled.

**Details:**

Access to the laboratory is restricted to authorized personnel. The authorized personnel are made aware of the following items:

- Ø the intended use of the area
- Ø the restrictions imposed on working within such areas
- Ø the reasons for imposing the restrictions

### 5.3.5 Good Housekeeping

**Policy:**

Measures are taken to ensure good housekeeping in the laboratory. Special procedures are prepared when necessary.



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### Details:

Controlled use of cleaning and pest control materials is exercised. The laboratory complies with the local health and safety requirements.

## 5.4 Tests and Calibration Methods and Method Validation

This section tells you:

1. Preference is given to the use of a standard method when selecting procedures
2. All methods must be validated before use
3. Measurement uncertainty is estimated
4. Data is controlled

### Key Words

Standard Methods  
Laboratory-Developed Methods  
Non-standardized Methods  
Validation  
Uncertainty of Measurement  
Data Checks

### Cross-references

ISO 17025:2005 Section 5.4

ISO 9001:2000 Section 4.2.1, 4.2.3, 6.3, 6.4, 7.1, 7.2.1, 7.3, 7.4.3, 7.5.1, 7.5.2, 7.6, 8.1, 8.2.3, 8.2.4



### 5.4.1 General

#### Policy:

Methods and procedures used for all tests and/or calibrations are appropriate as per:

- Ø sampling, handling, receipt, logging in, transport, storage, and preparation of items to be tested and/or calibrated
- Ø an estimation of the measurement of uncertainty as well as statistical techniques for analysis of test and/or calibration data where appropriate

Instructions on the use and operation of all relevant equipment and on the handling and preparation of items for testing and/or calibration are available. All instructions, standards, manuals and reference data relevant to the work of the laboratory are maintained current and readily available to personnel. Deviation from test and calibration methods must be documented, technically justified, authorized, and accepted by the client.

#### Details:

There are SOPs for sampling, sample handling, transport, storage, preparation of test items, QA/QC procedures (media QC, incubation times and temperatures, equipment calibration and maintenance, process control QC), and standards for approving / rejecting results. These may be combined with or separate from the method. The content of a test method includes:

- Ø scope
- Ø description of test items
- Ø holding times
- Ø quantities to be tested
- Ø materials and equipment required
- Ø physical environmental conditions required (incubation times and temperatures, pH requirements)
- Ø description of procedures
- Ø sample identification
- Ø method of recording observations and results
- Ø safety measures
- Ø documentation
- Ø method for data analysis and presentation
- Ø sensitivity of method
- Ø quality control plan

International, national, or regional standards or other recognized specifications that contain sufficient and concise information on how to perform the tests and/or calibrations are not necessarily supplemented or rewritten as an internal procedure when they are written in a way that can be used as published by laboratory staff. Consideration may need to be given to providing additional documentation for optional steps in the method.



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### 5.4.2 Selection of Methods

**Policy:**

Test and/or calibration methods, including methods for sampling, meet the needs of the client and are appropriate for the tests and/or calibrations it undertakes. Preference is given to reference methods published as international, national, or regional standards. The laboratory ensures that the latest edition of a standard is used unless it is not appropriate or possible to do so. When necessary, the standard is supplemented with additional details to ensure consistent application.

**Details:**

Methods that have been published either in international, national, or regional standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer are selected when the client does not specify the method to be used. These methods may be adopted from the National Institute of Occupational Safety and Health (“NIOSH”), the Occupational Safety & Health Administration (“OSHA”), the Environmental Protection Agency (“EPA”), the California Air Resources Board (“CA ARB”), the American Society for Testing and Materials (“ASTM”), or other comparably recognized professional or regulatory bodies.

The ability of the laboratory to achieve satisfactory performance against documented performance characteristics is verified before samples are analyzed.

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. The client is informed as to the method chosen. The laboratory confirms that it can properly operate standardized methods before introducing the tests or calibrations. If the standardized method changes, the confirmation is repeated.

The client is informed when the method proposed by the client is considered to be inappropriate or out of date.

### 5.4.3 Laboratory-Developed Methods

**Policy:**

Introduction of test and calibration methods developed internally is a planned activity and is assigned to qualified personnel equipped with adequate resources. Plans are updated as



development proceeds and ensure effective communication amongst all personnel involved.

**Details:**

Methods developed in-house are validated and authorized before use. Where available, Certified Reference Materials (CRMs) are used to determine any systemic bias, or where possible results are compared with other techniques, preferably based on different principles of analysis. Determination of uncertainty must be part of this validation process and is essential for ongoing quality control.

## 5.4.4 Non-Standard Methods

**Policy:**

Utilization of non-standard methods is subject to agreement with the client and includes a clear specification of the client's requirements and the purpose of the test. The developed method is validated appropriately before use.

**Details:**

Discussion and agreement for the use of non-standard methods is recorded as part of contract review procedures (see section 4.4).

All non-standard and new tests are validated for their intended purpose. Qualitative test methods must be validated to demonstrate estimated sensitivity and specificity, relative accuracy to official methods (if appropriate), positive and negative deviation, limit of detection, matrix effect, repeatability, and reproducibility.

Quantitative test methods are validated to demonstrate specificity, sensitivity, relative accuracy, positive and negative deviation, repeatability, reproducibility, and limit of determination.

For new methods where procedures are developing rapidly, especially for emergency situations, it may be necessary to circumvent normal validation procedures. Minimally, this must be a demonstrated recovery in replicate.

New test and/or calibration methods are documented prior to providing test and/or calibration results to clients and contain at least the following information:

- Ø appropriate identification
- Ø scope
- Ø description of the type of item to be tested or calibrated
- Ø parameters or quantities to be determined



- Ø apparatus and equipment, including technical performance requirements
- Ø reference standards and reference materials required
- Ø environmental conditions required and any stabilization period needed
- Ø description of the procedure, including
- Ø affixing identification marks, handling, transporting, storing and preparing of items
- Ø ensuring checks are made before the work is started
- Ø checking that the equipment is working properly and, where required, calibrating and adjusting the equipment before each use
- Ø listing method of recording the observations and results
- Ø indicating any safety measures to be observed
- Ø criteria and/or requirements for approval/rejection (quality control plan)
- Ø data to be recorded and method of analysis and presentation
- Ø uncertainty or procedure for estimating uncertainty

## 5.4.5 Validation of Methods

### 5.4.5.1 Performance Characteristics

#### Policy:

Validation of a method establishes, by systematic laboratory studies, that the performance characteristics of the method meet the specifications related to the intended use of the test results.

#### Details:

The performance characteristics of a validation plan includes, as applicable:

- Ø selectivity and specificity
- Ø range
- Ø linearity
- Ø sensitivity
- Ø limit of detection
- Ø limit of quantitation
- Ø ruggedness
- Ø accuracy
- Ø precision
- Ø reporting limit
- Ø repeatability
- Ø reproducibility
- Ø recovery
- Ø confirmation techniques
- Ø criteria for the number of samples tested to validate method as per defined scope of method



- Ø action levels where defined by regulation
- Ø quality control incorporating statistics as applicable
- Ø interpretation of population results as applicable

Performance characteristics that are selected take into account the intended use of the method, whether for screening, confirmatory analysis, or quantitation.

The design, verification of the method and documentation procedures for validation are planned and conducted by qualified personnel, equipped with adequate resources.

This section lists a few acceptable validation procedures. The choice of the procedure depends on the extent of the deviation from the published method.

Validation of methodology is a value judgment in which the performance parameters of the method are compared with the requirements for the test data. A prerequisite for a valid method is that data produced by the method must attain a state of statistical control. Such a state is obtained when the mean value of a large number of individual values tends to approach a limiting value called the limiting mean.

Methods may be validated by one or more alternative procedures. Some of these procedures are described below. Apparent differences can be analyzed statistically to confirm their significance. In all cases, the reasons for choosing one or more alternatives must be documented.

- Ø analysis of standard reference materials (SRM) that are identical or almost identical to the test samples
- Ø in the absence of suitable SRMs, analysis of reference materials that are similar in all respect to the test samples; the use and validity of this reference material must be documented
- Ø using an alternative method to measure the same parameter provides a very high level of confidence if results are confirmed
- Ø recovery studies by the addition of a known concentration of the parameter of interest to some of the replicates being measured

The parameters to be determined include:

- Ø the scope of the method and any known interference
- Ø detection limit
- Ø the range of concentration where the method is valid
- Ø precision and bias
- Ø intra-laboratory variations
- Ø inter-laboratory variations

Judgment is required to determine if some or all of the above is required. Requirements will depend largely on the extent of deviation from the original method.



Developments in methodology and techniques require methods to be changed from time to time. The difference in performance between revised and obsolete methods is established so that it is possible to compare old and new data.

Where a change in method involves only minor adjustments, such as sample size, or different reagents, the amended method is validated and the changes brought to the attention of the accreditation body at the next accreditation audit. Where the proposed change involves technology or methodology, the laboratory seeks the approval of the accreditation body.

Records are kept on all validation activities. The records include any of the performance characteristics chosen, reference procedures or guidance documents followed to validate the method or custom validation procedure, and a final confirmation (memo to file) that the method validation results are acceptable for continued use of the method. An example statement would be "This memo serves as record that the validation of the XYZ Test Method has been approved for use by [name and title of approver]".

#### 5.4.5.2 Fit for Use

##### **Policy:**

The laboratory validates non-standardized methods, laboratory-designed/developed methods, standardized methods used outside their intended range, and amplifications of standard methods to confirm that the methods are fit for the intended use. The validation is as extensive as is necessary to meet the needs in the given application or field of application (may include procedures for sampling, handling, and transportation). The laboratory records the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

##### **Details and Procedure:**

Validation records are kept as in section 5.4.5.1. Included in these records is the validation procedure. The procedure used for the validation is likely to vary between different methods. Therefore, the procedures included in the laboratory records are not as detailed as a typical SOP, but are sufficient enough to re-create how the method was validated.

The techniques used for the determination of the performance of a method, are one of, or a combination of, the following:

- Ø calibration using reference standards or reference materials
- Ø comparison of results achieved with other methods
- Ø inter-laboratory comparisons
- Ø systematic assessment of the factors influencing the result





Ø assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.

When changes are made in the validated non-standard method, the influence of such changes carried out is documented and if appropriate a new validation is performed.

### 5.4.5.3 Client's Needs

#### **Policy:**

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 is relevant to the client's needs.

#### **Details:**

Validation includes the specification of the requirements, determination of the characteristics of the methods, the comparison of the requirements with the values of the characteristics of the method, and a statement on the validity.

As method development proceeds, regular review is required to verify that the needs of the client are still being fulfilled. Changing requirements requiring modifications to the development plan are approved and authorized.

Validation is always a balance between costs, risks, and technical possibilities.

## 5.4.6 Uncertainty of Measurement

### 5.4.6.1 Calibration

#### **Policy:**

Physical, chemical, and biological standards are calibrated or characterized by qualified subcontractors.

#### **Details and Procedures:**

Repeatability and reproducibility data are components of measurement uncertainty and are determined as a first step towards producing estimates of this parameter. The uncertainty of measurement is available on the certificate of analysis or calibration certificate from a subcontractor.

**Note** - in-house calibrations include procedures for uncertainty of measurement estimates where this is common practice.



## 5.4.6.2 Testing

### Policy:

The SOP# 5-4-6 is utilized to estimate uncertainties of measurement in testing, except when the test methods preclude such rigorous calculations. In certain cases it is not possible to undertake metrologically and statistically valid estimations of uncertainty of measurement. In these cases the laboratory attempts to identify all the components of uncertainty and make the best possible estimation, and ensure that the form of reporting does not give an exaggerated impression of accuracy. Reasonable estimation is based on knowledge of the performance of the method and on the measurement scope and makes use of previous experience and validation data.

### Details:

The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

- Ø requirement of the test method
- Ø requirement by the client
- Ø if there are narrow limits on which decisions on conformance to a specification are based

In 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 the estimation uncertainty of measurement by following the reporting instructions (see section 5.9).

## 5.4.6.3 Uncertainty Components

### Policy:

When estimating the uncertainty of measurement, all uncertainty components that are of importance in the given situation are taken into account using accepted methods of analysis.

### Details:

Sources contributing to the uncertainty include, but are not necessarily limited to, the reference standards and reference materials used, methods and equipment used, the environmental conditions, the item being tested or calibrated and the operator.

The predicted long-term behavior of the tested and/or calibrated item is normally not taken into account when estimating the measurement uncertainty.



For further information, see ISO 5725 and the Guide to Expression of Uncertainty in Measurement.

## 5.4.7 Control of Data

### 5.4.7.1 Calculations and Data Transfers

#### **Policy:**

Calculations and data transfers are subject to appropriate checks in a systematic manner.

#### **Details:**

Test data are validated through the following arrangements by the the Senior Analyst:

- Ø checks to determine accuracy of calculations, conversions, and data transfers
- Ø checks for transcription errors, omissions, and mistakes
- Ø checks to determine consistency with normal or expected values

For those analyses where manual data reduction is required, it is performed according to the instructions provided in the test method or SOP.

### 5.4.7.2 Computers and Automated Equipment

#### **Policy:**

When computers or automated equipment are used for the acquisition, processing, manipulation, recording, reporting, storage or retrieval of test or calibration data, the laboratory ensures that:

- Ø computer software developed by the user is documented in sufficient detail and suitably validated or otherwise checked as being adequate for use
- Ø procedures are established and implemented for protecting the integrity of data; such procedures include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission, and data processing (see section 4.12.1.4)
- Ø 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
- Ø data is securely maintained by preventing unauthorized access to, and unauthorized amendment of, computer records

#### **Details and Procedures:**

Data generated using computer software programs that are interfaced directly to instruments incorporates all dilutions and calculations, thereby eliminating the need for manual data reduction.



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Commercially developed software in general use within its designed application range may be considered sufficiently validated. Laboratory software configuration / modifications are validated as outlined in SOP# 5-5-1.

Electronic records, electronic signatures, and handwritten signatures executed to electronic records must be equivalent to proper records and handwritten signatures to paper and are validated by procedures in 21 CFR. Part II (Docket No. 92NO251) RIN0910-AA29; Federal Register: March 20, 1997, Volume 62, Number 54), Rules and Regulations, pages 13429-13466. For further details see:

<http://www.fda.gov/cder/esig/index.htm>

### **5.5 Equipment**

This section tells you to:

1. Identify information needs for accept / reject decisions
2. Install equipment capable of providing that information
3. Use the equipment in the proper environment
4. Periodically check the equipment calibration

### **Key Words**

Required Equipment and Accuracy  
Authorized Personnel  
Unique Identification  
Inventory  
Maintenance  
Procedures  
Out of Service  
Calibration Status  
Re-verification  
Checks



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Correction Factors  
Safeguards against Adjustment

### Cross-references

ISO 17025:2005 Section 5.5

ISO 9001:2000 Section 4.2.1, 4.2.3, 5.1, 7.1, 7.4, 7.5.1, 7.5.2, 7.5.3, 7.6, 8.1,  
8.2.3, 8.2.4



### 5.5.1 Required Equipment

**Policy:**

The laboratory is furnished with all items for 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). When equipment is used outside the laboratory's permanent control, it ensures that the requirements of this Quality Manual are met.

**Details:**

Equipment is used in an environment appropriate to its proper performance. All equipment required by a test is described in each method, including the equipment's tolerances.

### 5.5.2 Required Accuracy

**Policy:**

Equipment and software used for testing, calibration and sampling are capable of achieving the accuracy required and comply with specifications relevant to the tests and/or calibrations concerned. Calibration programs are established for key quantities or values of the instruments where these properties have a significant affect on the results. When received, equipment, including that used for sampling, is checked to establish that it meets the laboratory's specification requirements, complies with the relevant standard specifications, and is checked and/or calibrated in accordance with section 5.6 before use.

**Details:**

The procedures for checking newly received equipment are as determined by manufacturers' specification and/or those determined by the laboratory during procurement.

### 5.5.3 Authorized Personnel

**Policy:**

Equipment is 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) is readily available for use by the appropriate laboratory personnel.

**Details:**



Access to laboratory equipment is controlled to ensure that only authorized personnel use equipment.

### 5.5.4 Unique Identification

**Policy:**

Each item of equipment used for testing and calibration is uniquely identified as appropriate.

**Details:**

Measuring and testing equipment is uniquely identified through an asset or serial number. Measuring and testing equipment includes any instrument that could affect the quality of test results. Components that can be interchanged between various instruments are tracked in equipment logbooks, but are not assigned individual asset numbers.

### 5.5.5 Inventory and Maintenance Records

**Policy:**

Records are maintained of each item of equipment significant to the tests and/or calibrations performed. The records include the following:

- Ø identity of the item of equipment (and its software)
- Ø manufacturer's name, type identification, and serial number and/or other unique identification
- Ø checks that equipment complies with the specification (see section 5.5.2)
- Ø current location, where appropriate
- Ø the manufacturer's instructions, if available, or reference to their location
- Ø dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and due date of next calibration
- Ø maintenance carried out to date and the maintenance plan (includes calibration)
- Ø damage, malfunction, modification or repair to the equipment

**Details:**

A database is used to capture the above inventory information. The above information related to service and maintenance is kept in individual equipment files and/or binders. Other information kept in these files and/or binders may include:

- Ø date received and date placed in service
- Ø condition when received (e.g., new, used, refurbished)
- Ø dates and results of calibration and/or verification and date of next calibration and/or verification
- Ø performance history, where appropriate (e.g., response time, drift, noise level)



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### 5.5.6 Equipment Procedures

**Policy:**

The SOP# 5-5-1 is utilized as an established plan for safe handling, transport, storage, use and maintenance (including calibration) of measuring equipment, and appropriate use of correction factors 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 (currently not applicable at our laboratory).

**Details and Procedures:**

The procedures for each piece of measuring equipment are located in the appropriate room where the equipment is located. These procedures detail any information for safe handling, transport, storage, use, and maintenance of measuring equipment.

### 5.5.7 Out of Service Equipment

**Policy:**

Equipment that has either been subjected to overloading or mishandling, or gives suspect results, or has been shown to be defective or outside specified limits, is taken out of service, clearly marked, and appropriately stored until it has been repaired and shown by calibration or test to perform correctly.

**Details:**

Routine testing work is completely discontinued on equipment that even shows minor non-conformances. Not only do we do this for ethical reasons in support of our client, but minor non-conformances are often indicative of major breakdowns in expensive equipment. These breakdowns need to be avoided wherever possible.

Out of service equipment is clearly marked as outlined in section 5.5.7.

The laboratory examines the effect of the defect or departure from specified limits on previous test and/or calibrations and institutes the “Control of Nonconforming Work” procedure as outlined in section 4.9.





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### 5.5.7 Calibration Status

**Policy:**

Equipment requiring calibration is labeled to indicate the calibration status and/or operational status and the date when re-calibration is due when appropriate.

**Details:**

Calibration labels have a write-on surface and a pressure sensitive adhesive. The areas that are filled out include the person who performed the calibration, the date it was performed, the date it is due for re-calibration, and the equipment's identification number.

<b>CALIBRATION</b>	
BY _____	DATE _____
DUE _____	ID# _____

Measuring equipment that has failed calibration or is deemed out of service is labeled with one of the following labels:

<b>CALIBRATION VOID</b>
<b>DO NOT USE</b>

<b>OUT OF SERVICE</b>
<b>DO NOT USE</b>

A piece of equipment that is not calibrated or checked is labeled with the following label:

<b>FOR REFERENCE ONLY</b>
---------------------------

### 5.5.8 Return to Service

**Policy:**

When equipment goes outside the direct control of the laboratory for a period, the laboratory ensures that the function and calibration status of the equipment are checked and validated and shown to be satisfactory before the equipment is returned to service.

**Details and Procedures:**

The procedures used to check and ensure that the function and calibration status of the equipment are satisfactory before the equipment is returned to service are outlined in the manufacturer's equipment manual. Any additional quality control checks are outlined in the "Quality Control Plan" section of the appropriate test method.

## 5.5.9 Periodic Checks

**Policy:**

When intermediate checks are needed to maintain confidence in the calibration status of equipment, these checks are carried out periodically according to defined procedure.

**Details and Procedures:**

As stated in section 5.5.6, the procedures for each piece of measuring equipment are located in the appropriate room where the equipment is located. SOP# 5-5-1 outlines a general maintenance plan for equipment and includes various checks. Internal quality control checks are specified in individual test methods that are located in the appropriate laboratory areas thereby providing procedures for intermediate checks.

## 5.5.11 Correction Factors

**Policy**

Calibrations that give rise to a set of correction factors are updated along with all copies of this data (e.g., in computer software).

**Details and Procedures:**

The updating of correction factors including all copies is assured by following the appropriate test method or SOP. It is the responsibility of the Senior Analyst to ensure that all copies are updated.

## 5.5.12 Safeguards against Adjustments

**Policy:**



Test and calibration equipment, including hardware and software, are safeguarded from adjustments that invalidate test and/or calibration results/status.

## Details:

Safeguards against adjustment for laboratory equipment include:

- Ø detailed SOPs and manufacturer's manuals on the operation of the equipment
- Ø policies permitting only fully trained and competent personnel to operate equipment
- Ø access to the laboratory is restricted to authorized personnel

Safeguards against adjustment for software includes:

- Ø password protection for important files and packages
- Ø access to the laboratory is restricted to authorized personnel

## 5.6 Measurement Traceability

This section tells you:

1. Measurements are traceable to SI units (when applicable)
2. Reference Standards and Reference Materials are used

## Key Words

Système International  
Reference Standard  
Reference Material  
Traceability

## Cross-references

ISO 17025:2005 Section 5.6

ISO 9001:2000 Section 7.1, 7.6



## 5.6.1 General

### Policy:

Test and/or calibration 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 are calibrated before being put into service. All measurement and test equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before being put into service. As mentioned in section 5.5, the SOP# 5-5-1 outlines an established program for the maintenance of equipment and includes calibration.

### Details:

The program includes a system for selecting, using, calibrating, checking, controlling, and maintaining:

- Ø measurement standards
- Ø reference standards used as measurement standards
- Ø measuring and test equipment used to perform tests and calibrations

Procedures are documented where appropriate. All measurements that play a defining role in testing accuracy are based directly or indirectly on reference standards, reference materials, certified reference materials, or other standards or materials having appropriate traceability.

## 5.6.2 Specific Requirements

### 5.6.2.1 Calibration

### Policy:

The program for calibration equipment is designed and operated to ensure that calibration measurements are traceable to the Système International (SI) units of measurement.

### Details:

Traceability of measurement is assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories show that there is a link to a primary standard or to a natural constant realizing the SI unit by an unbroken chain of calibrations. The calibration certificates contain the measurement results including the measurement uncertainty and/or a statement of compliance with an identified metrological specification (see also section 5.9.4.2).



Calibration laboratories accredited to ISO 17025 are considered competent to provide the appropriate calibration services.

Traceability to SI units of measurement may be achieved by reference to an appropriate primary standard or by reference to a natural constant the value of which in terms of the relevant SI unit is known.

The term “identified metrological specification” means that it must be clear from the calibration certificate against which specification the measurements have been compared with, by including the specification or by giving an unambiguous reference to the specification.

When the terms “international standard” or “national standard” are used in connection with traceability, it is assumed that these standards fulfil the properties of primary standards for the realization of SI units.

Maintain certificates of all reference standards, measuring equipment, or certified reference material used in ensuring traceability. Where traceability to national standards of measurement is not applicable, the laboratory provides satisfactory evidence of correlation of results, for example by participation in a suitable program of inter-laboratory comparisons or proficiency testing.

Reference standards, such as thermometers and weights are traceable to a national or international standard (e.g., NIST).

## **5.6.2.2 Testing**

### **5.6.2.2.1**

#### **Policy:**

The requirements given in section 5.6.2.1 apply to measuring and test equipment with measuring functions used, unless it has been established that the associated calibration uncertainty contributes little to the total uncertainty of the test result. When this situation arises, the laboratory ensures that equipment used can provide the accuracy of measurement needed.

#### **Details:**

The extent to which the requirements in section 5.6.2.1 are followed depends on the relative contribution of calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements are strictly followed. If, however, calibration is not one of the major contributors to the total uncertainty, other ways for providing confidence may be used, as given in section 5.6.2.2.2.



### 5.6.2.2.2

**Policy:**

Where traceability to SI units of measurement is not possible and/or not relevant, other means for providing confidence in the results are applied such as:

- Ø the use of suitable reference materials certified to give a reliable characterization of the material
- Ø mutual-consent standards or methods which are clearly specified and agreed upon by all parties concerned
- Ø participation in a suitable program of inter-laboratory comparisons or proficiency testing

**Details:**

Reliable characterization involves an estimate of recovery.

The laboratory participates in proficiency testing and/or check sample programs. The list of programs is maintained by the Quality Manager.

## 5.6.3 Reference Standards and Reference Materials

### 5.6.3.1 Reference Standards

**Policy:**

The SOP# 5-6-1 outlines the program for the calibration of reference standards. Reference standards are obtained or calibrated by a body that can provide traceability as described in section 5.6.2.1. Such reference standards of measurement held by the laboratory are used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

**Details:**

Reference standards are obtained from the National Institute of Standards and Technology (NIST) or other established and recognized sources.

### 5.6.3.2 Reference Materials

**Policy:**

Where possible, reference materials are traceable to SI units of measurement, or to certified reference materials. Internal reference materials are checked as far as is technically and economically practicable.

**Details:**

Reference materials, including calibration standards, used in chemical measurement are prepared so that the point of measurement is similar or equivalent to that of the samples. The matrix, prior to the addition of the analyte does not have a detectable concentration of the analyte. Reagents used in the preparation of reference materials, including calibration standards are of certified purity.

### 5.6.3.3 Intermediate Checks

**Policy:**

Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials are carried out according to defined procedures and schedules.

**Details and Procedures:**

The control check standards used to verify the accuracy of all the other standards are prepared independently from all the other standards used to establish the original calibration. These control check standards are preferably prepared from a separate lot # or source. It is the responsibility of the Quality Assurance Director to establish and maintain the individual schedule for each SOP and/or test method.

### 5.6.3.4 Transport and Storage

**Policy:**

The SOP# 5-6-1 outlines 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.

**Details:**

Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations, or sampling.

Proper conditions are established for housing, handling, and care of reference materials and standards. All information needed to properly identify references appears on their housing or containers.



### 5.7 Handling of Test and Calibration Items

This section tells you to:

1. Keep samples in good condition.

#### Key Words

Identification

Receipt

Protection

#### Cross-references

ISO 17025:2005 Section 5.7

ISO 9001:2000 Section 6.3, 6.4, 7.1, 7.4.3, 7.5, 8.2.4





### 5.7.1 Procedures

**Policy:**

The SOP# 5-7-1 outlines the 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 the interests of the laboratory and the client.

**Details:**

Samples, reagents, and standards are stored so as to ensure their integrity by preventing against deterioration, contamination, and loss of identity. It is recognized that this is a general statement, but details are elaborated upon in SOP# 5-7-1.

### 5.7.2 Identification of Test and/or Calibration Items

**Policy:**

Test and/or calibration items are systematically identified as they arrive at the laboratory. The identification is retained throughout the life of the item in the laboratory. The system is designed and operated so as to ensure that items cannot be confused physically, or when referred to in records or other documents. The system accommodates a sub-division of groups of items and the transfer of items within and from the laboratory when appropriate.

**Details:**

Sample labelling indicates the unique identification and conforms to applicable legal requirements. Where conformity of possession of a test sample must be maintained for forensic or other purposes, the laboratory establishes and documents a system for appropriate chain-of-custody (forensic samples may be used in a court of law for evidentiary purposes).

### 5.7.3 Receipt

**Policy:**

Upon receipt of the test or calibration item, any abnormalities or departures from normal or specified conditions, as described in the relevant test or calibration method, are recorded. When there is any 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 consults the client for further instructions before proceeding and keeps a record of the discussion.

**Details:**

Conform to applicable regulations or contractual arrangements. The condition of sample may include or relate to damage, quantity, preparation, packaging, or temperature. Preparation may include addition of chemical preservative, removal of moisture, and isolation of portion of sample to be tested, homogenization, or sub-sampling.

Arrangements are in place to ensure that elapsed time between sampling and testing does not exceed test method specifications (holding time).

## 5.7.4 Protection

**Policy:**

The SOP# 5-7-1 outlines the procedures and appropriate facilities for avoiding deterioration, loss or damage to the test or calibration item during storage, handling and preparation and testing; instructions provided with the item are followed. When items have to be stored or conditioned under specified environmental conditions, these conditions are maintained, monitored, and recorded. Where a test item is to be held secure (e.g., for reasons of record, safety or value, or to enable complementary test and/or calibrations to be performed later), the laboratory has arrangements for storage and security that protect the condition and integrity of the secured item concerned.

**Details:**

Where test items are to be returned into service after testing (e.g., for non-destructive testing or human beings subject to medical tests), special care is required to ensure that they are not damaged or injured during the handling, testing or storing/waiting processes.

A sampling procedure and information on storage and transport of samples, including all information that may influence the test or calibration result, is provided to those responsible for taking and transporting the samples.

The laboratory establishes whether the sample has received all necessary preparation or whether the client requires preparation to be undertaken or arranged by the laboratory. Proper requirements for packaging, environmental conditions, and separation from incompatible materials are observed. Where samples have to be stored or conditioned under specific conditions, these conditions are maintained, monitored, and recorded, where necessary.

Where a sample, or portion of a sample, is to be held secure (e.g., for reasons of record, safety, or value, or to enable check tests to be performed later), the laboratory has storage and security arrangements that protect the condition and integrity of the sample.



### 5.8 Assuring the Quality of Test and Calibration Results

This section tells you:

1. That results are monitored
2. There is a plan for monitoring

#### Key Words

Internal Quality Control  
Statistical Techniques  
Inter-laboratory Comparisons  
Proficiency Testing  
Certified Reference Materials  
Secondary Reference Material  
Replicates  
Re-testing  
Correlation

#### Cross-references

ISO 17025:2005 Section 5.8

ISO 9001:2000 Section 6.3, 6.4, 7.1, 7.4.3, 7.5.1, 7.5.2, 7.5.3, 7.5.5, 8.1, 8.2.3, 8.2.4, 8.4



### 5.8 Assuring the Quality of Test and Calibration Results

#### Policy:

Quality control procedures are utilized to monitor the validity of test and/or calibration results. These procedures are for each test method utilized in the laboratory. The resulting data are recorded so that trends are detectable (and where practicable, statistical techniques are applied to the reviewing of the results. This monitoring is planned and reviewed and may include, but not limited to, the following:

- Ø regular use of certified reference materials and/or internal quality control using secondary reference materials
- Ø participation in inter-laboratory comparisons or proficiency testing programs
- Ø replicate tests or calibrations using the same or different methods
- Ø re-testing or re-calibration of retained items
- Ø correlation of results for different characteristics of an item

#### Details:

The methods utilized from the above list will be appropriate for the type and volume of the work undertaken. Records are maintained of assurance activities and any actions taken.

As a guide, for routine analyses the level of internal quality control is typically 5% of the sample throughput. For more complex procedures, 20% is not unusual and on occasions even 50% may be required. For analyses performed infrequently, a full system validation is performed on each occasion. This may typically involve the use of a reference material containing a certified or known concentration of analyte, followed by replicate analyses of the sample and spiked sample. For analyses undertaken more frequently, systematic quality control procedures incorporating the use of control charts and check samples are implemented. These procedures are documented in the "Quality Control Plan" of each test method.

Internal quality control schemes using statistics include:

- Ø design of experimental/factorial analysis
- Ø variation/regression analysis
- Ø safety evaluation/risk analysis
- Ø tests of significance
- Ø quality control charts
- Ø statistical sampling inspection

Proficiency testing helps to highlight not only repeatability and reproducibility performance between laboratories, but also systematic errors such as bias. It is important to monitor proficiency testing results as a means of checking quality assurance and take action as necessary.



The Quality Assurance Director maintains a list of all the current proficiency testing programs the laboratory participates in, monitors the results, and notifies the appropriate personnel of both problematic and successful results.

Technical personnel use certified reference materials and reference materials to evaluate test performance on a daily basis and include daily process control checks. These data are used to evaluate the validity of the test results.

Replicate tests may be used if suitable reference material is available. These materials and proficiency test materials are available for improving repeatability.

Re-testing of test items is performed occasionally at the discretion of the supervisor or when test results seem anomalous.

## **5.9 Reporting of Results**

This section tells you:

1. What needs to be on a report
2. How to handle amendments to reports

### **Key Words**

Specific Information  
Required Information  
Interpretation  
Opinion  
Subcontractor  
Electronic Transmission of Results  
Format  
Amendments

### **Cross-references**

ISO 17025:2005 Section 5.9

ISO 9001:2000 Section 7.1, 7.4.3, 7.5.1, 7.5.4, 8.2.4



### 5.9.1 General

**Policy:**

The results of each test, calibration, or series of tests or calibrations are reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test or calibration methods.

The results are reported, normally in a test report and include all the information requested by the client and necessary for the interpretation of the test results and all information required by the method used. This information may include what is outlined in section 5.9.2, 5.9.3 and 5.9.4.

In the case of tests or calibrations performed for internal clients, and in the case of a written agreement with the client, the results may be reported in a simplified way. The information listed in section 5.9.2 to 5.9.4, and not reported, is kept readily available.

**Details:**

Test reports and/or calibration reports are issued as either hard copy or by electronic data transfer.

### 5.9.2 Quality Control Data

**Policy:**

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.

### 5.9.3 Test reports and calibration certificates

**Policy:**

Test reports and/or calibration certificates include the following information, as appropriate:

- Ø a title (e.g., "Test Report" [or Calibration Certificate])
- Ø name and address of laboratory, and location where tests and/or calibrations were carried out if different from the address of the laboratory
- Ø unique identification of the test report [or calibration certificate] (such as a 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]
- Ø name and address of the client
- Ø identification of the method used



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- Ø description, condition, and unambiguous identification of the item(s) tested [or calibrated]
- Ø date of receipt of test [or calibration] items (where this is critical to the validity and application of the results) and date(s) of performance of the test [or calibration]
- Ø reference to sampling procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results
- Ø copy of count sheet for TEM analysis
- Ø test [or calibration] results with, where appropriate, units of measurement
- Ø the name(s), function(s) and signature(s) or equivalent of person(s) authorizing the test report [or calibration certificate]
- Ø where relevant, a statement to the effect that the results relate only to the items tested [or calibrated]

### Details:

Signing authority for test reports is the responsibility of the Laboratory Manager. Records for individuals with signing authority for test reports are approved and maintained by the Laboratory Manager.

Hard copies of test reports [or calibration certificates] include the page number and total number of pages.

A statement is included specifying that the test report [or calibration certificate] is not to be reproduced except in full, without written approval of the laboratory. Data reported to the client contains the appropriate significant digits for each test method. Low level data are identified as being below specified limits.

## 5.9.4 Test Reports

### 5.9.4.1

#### Policy and Details:

In addition to the requirements listed in section 5.9.2, test reports include the following, where necessary for the interpretation of results:

- Ø deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions
- Ø where relevant, a statement of compliance/non-compliance with requirements and/or specifications
- Ø where applicable, a statement on the estimated uncertainty of measurement of the test result; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a client's instruction so requires, or when uncertainty affects compliance to a specification limit
- Ø where appropriate and needed opinions and interpretations (see section 5.9.5)



- Ø additional information required by specific methods, clients, or groups of clients

## 5.9.4.2

### Policy and Details:

In addition to the requirements listed in sections 5.9.2 and 5.9.3.1, test reports containing the results of sampling include the following, where necessary for the interpretation of test results:

- Ø date of sampling
- Ø unambiguous identification of substance, matrix, material or product sampled (including name of manufacturer, model or type of designation and serial numbers as appropriate)
- Ø location of sampling, including any diagrams, sketches or photographs
- Ø reference to sampling plan and procedures used
- Ø details of any environmental condition during sampling that may affect the interpretation of the test results
- Ø any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned

## 5.9.5 Calibration Certificates

### 5.9.5.1

#### Policy:

The testing laboratory does not issue calibration certificates. However, the laboratory often receives calibration services from a calibration laboratory and needs to be familiar with the information on a calibration certificate.

#### Details:

In addition to the requirements listed in 5.9.2, the calibration certificate could include the following, where necessary for the interpretation of calibration results:

- Ø the conditions (e.g., environmental) under which the calibrations were made that have an influence on the measurement results
- Ø the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof
- Ø evidence that the measurements are traceable (see 5.6.2.1.1)

## 5.9.6 Opinions and Interpretations

#### Policy:





When opinions and interpretations are included in the test report, the basis upon which the opinions and interpretations have been made is documented. Opinions and interpretations are clearly marked as such in the test report.

**Note** - opinions and interpretations should not be mixed-up with inspections and product certifications as intended in ISO/IEC 17020 and ISO/IEC Guide 65.

**Details:**

Opinions and interpretations included in a test report may comprise, but not be limited to the following:

- Ø opinion on conformity of the results with requirements
- Ø fulfilment of contractual requirements
- Ø recommendations on how to use the results
- Ø guidance to be used for improvements

In many cases it is appropriate to communicate the opinions and interpretations by direct dialogue with the client. This dialogue is written down.

### 5.9.7 Testing and Calibration Results Obtained from Subcontractors

**Policy and Details:**

Test reports containing the results of tests performed by subcontractors are clearly identified for the subcontracted results. The subcontractor reports the results either in writing or electronically to our laboratory.

### 5.9.8 Electronic Transmission of Results

**Policy:**

In the case of transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of the policies and procedures of this Quality Manual continue to apply (see also 5.4.7).

**Details:**

Reports that are “published” electronically contain the statement that signatures are on file.

### 5.9.9 Format of Reports

**Policy:**



The format of reports is designed to accommodate each type of test carried out and to minimize the possibility of misunderstanding or misuse.

**Details:**

The layout of the test report is such that the presentation of the test data facilitates ease of assimilation by the reader.

The headings are standardized as far as possible.

### 5.9.10 Amendments to Reports

**Policy:**

Material amendments to a test report after issue are made only in the form of a further document, or data transfer, which includes the statement “Revised Analytical Report”, or an equivalent form of wording. Such amendments meet all the requirements in this Quality Manual.

**Details:**

When it is necessary to issue a complete new test report, it is uniquely identified and contains a reference to the original that it replaces.



## Appendix A – Policy on the Use of Accreditation Program Logos

### App. A.1

Asbestos TEM Laboratories, Inc conforms to the policy requirements for use of logos and advertised affiliation with accreditation bodies. ISO/IEC 17011 requires that accreditation bodies “have a policy governing the protection and use” of reference to its accreditation and logo. The various accreditation bodies have policy requirements which address the manner in which accredited laboratories may advertise, reference affiliation and display the accreditation body’s logos.

Accreditation may be referenced by; a statement of accreditation, using the Laboratory ID Number or by use of the accreditation body’s logo. These references can only be used for fields of testing, FOT(s), in which the laboratory is accredited. Asbestos TEM Laboratory uses both the accreditation body’s logo and the Laboratory ID Number to reference its accreditation on the laboratory report cover letters, on advertisement and on bid submittals. The Laboratory ID Number and the LOGO are only used within the scope, FOT(s), covered by the accreditation body. The Laboratory ID number and the Logo are displayed in a manner consistent with the accreditation body’s policies for size, placement and aspect ratios.

The accreditation bodies’ policy statements are attached herein for reference.

### Attachment 1 - AIHA



## AIHA LQAP Policy Document – Module 7

Effective Date: April 1, 2007

Revision 4: January 2, 2007

### MODULE 7 - REFERENCE TO ACCREDITATION AND ADVERTISING POLICY

#### 7.1 INTRODUCTION

All AIHA accredited laboratories are encouraged to advertise their accreditation by using prescribed language defined in this module and the approved AIHA accreditation logo. ISO/IEC Standard 17011 requires that accreditation bodies, such as the AIHA, “have a policy governing the protection and use” of reference to its accreditation and logo. The following policies govern a lab’s reference to its accreditation in all communication media, such as the Internet, documents, reports, brochures or advertising.

Failure to conform to these policies or the advertising/logo license agreement shall result in any or all of the following: request for corrective action, suspension or revocation of accreditation, publication of the transgression or possible initiation of legal actions.

Only accredited AIHA laboratories may use the AIHA accreditation logo for purposes of advertising their laboratory accreditation.

### Attachment 1 – AIHA continued

#### 7.1.1 Reference to AIHA Accredited Fields of Testing (FoTs)

AIHA accreditation or affiliation may be referenced by use of 1) a statement of AIHA accreditation, and/or 2) the AIHA Laboratory ID Number, and/or 3) the AIHA accreditation logo.

Any of these references may not be used or implied for a FOT(s) for which lab is not accredited by AIHA.

A laboratory shall not advertise that it is accredited by AIHA until the laboratory has actually received written notification from AIHA that it has been accredited. Also, an AIHA accredited laboratory that adds an additional FoT to its existing scope of accreditation (see Module 3,

Section 3.9) shall not advertise that it is accredited for that scope of testing until it receives written notification of approval from AIHA.

#### 7.1.2 Reference to AIHA Accreditation for Suspended-Status FoTs

An accredited laboratory whose accreditation has been suspended or withdrawn shall not reference AIHA accreditation for the FoT/Method(s) for which it is suspended or



withdrawn for the duration of the suspension period. Upon suspension or withdrawal, the laboratory shall discontinue the use of all communication media that contains any reference to the suspended or withdrawn accreditation.

### 7.2 STATEMENT OF AIHA ACCREDITATION

An AIHA accredited laboratory may use the following statements, or the equivalent, in communication media, subject to the limitations listed in 7.6, below.

**7.2.1** "\_\_\_\_ Laboratory is accredited by the American Industrial Hygiene Association (AIHA) in the \_\_\_\_ accreditation program for \_\_\_\_ Fields of Testing as documented by the Scope of Accreditation Certificate." (Blanks are to be filled with the applicable terms, as listed on the accreditation certificate.)

AIHA accredited laboratories may also use the following statement in their communication media, in conjunction with 7.2.1.

**7.2.2** "AIHA accreditation complies with the ISO/IEC Standard 17025 requirements, but this does not imply ISO certification or registration."

**7.2.3** Laboratories with multiple locations must clearly identify the location of the accredited laboratory(s) and their applicable accreditation programs in their communication media.

### 7.3 LABORATORY ID NUMBER

An AIHA accredited laboratory may use its AIHA assigned Laboratory ID Number in its media communications subject to the limitations listed in Section 7.6.

## Attachment 1 – AIHA continued

### 7.4 PROFICIENCY TESTING ONLY ORGANIZATIONS

A laboratory may use its Laboratory ID Number to indicate participation in one or more AIHA PT programs, as long as accreditation is not implied, for example: "\_\_\_\_ Laboratory participates in the AIHA \_\_\_\_ PAT Program, Laboratory ID Number \_\_\_\_". (Blanks are to be filled with the applicable terms.)

### 7.5 AIHA ACCREDITATION LOGO

The AIHA accreditation logo may be used by accredited laboratories, subject to the limitations listed in Section 7.6. An AIHA accredited laboratory shall only use the AIHA accreditation logo after signing the appropriate advertising/logo licensing agreement, detailing the permissible usage. The AIHA accreditation advertising/logo licensing agreement is provided by the AIHA at the time the accreditation certificate is issued. The laboratory shall sign and return the advertising/logo licensing agreement to AIHA before the AIHA will release the copy ready artwork of the logo to the laboratory.



### 7.6 LIMITATIONS TO REFERENCING AIHA ACCREDITATION

**7.6.1** A statement of AIHA accreditation or the AIHA accreditation logo shall only be displayed by laboratories that hold AIHA accreditation, using the organization name as stated on the accreditation certificate.

**7.6.2** A statement of AIHA accreditation or the AIHA accreditation logo shall only be used by the laboratory on its Internet web site, letterhead documents, reports, brochures or advertising ("communication media"). The laboratory shall not use a statement of AIHA accreditation or AIHA accreditation logo on communication media when such testing is outside the scope of accreditation, unless the laboratory provides a clear disclaimer and/or identifies the testing that is outside the scope of AIHA accreditation.

**7.6.3** A statement of AIHA accreditation and/or the AIHA accreditation logo signify that a laboratory meets certain standards. The laboratory shall not display a statement of AIHA accreditation or the AIHA accreditation logo on products, product catalogs product packaging or inserts or otherwise on any item not specifically outlined as communication media, above.

Furthermore, a statement of AIHA accreditation or the AIHA accreditation logo may not be displayed on communication media or any other laboratory materials that are outside the scope of accreditation for which the laboratory is accredited by the AIHA.

### Attachment 1 – AIHA continued

**7.6.4** The laboratory shall only display a statement of AIHA accreditation or the AIHA accreditation logo on the internet or on other segmented materials one those web pages or those areas of materials that are relevant to the scope of accreditation for which the laboratory is accredited by AIHA.

**7.6.5** The laboratory shall not make any statement regarding its AIHA accreditation or AIHA PT participation that AIHA may consider to be misleading or unauthorized.

**7.6.6** The laboratory shall take care that no report or certificate nor any part thereof referencing AIHA accreditation or AIHA PT participation is used in a misleading manner.



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**7.6.7** Accreditation by AIHA does not imply that a product, process, system, or person is approved by AIHA. Accordingly, a statement of accreditation or an AIHA logo may not be used in manner suggesting or implying that a product, process, system, or person is approved or certified by AIHA or that AIHA is otherwise certifying something other than the laboratory itself.

### **Attachment 2 – NVLAP**



## 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 is 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 000000-00" (Fig. 1).
  - "NVLAP Lab Code 000000-00" (Fig. 2).
- f) When the NVLAP symbol is used, 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 (width to height) shall be 2:25 to 1 (Fig. 3).





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— 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.

g) The name of at least one Approved Signatory shall appear on a test or calibration report that displays the NVLAP symbol or effluence 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) 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.

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."

i) 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.

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 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*.



## A.2 Joint ISO-ILAC-IAF Communiqué

On June 18, 2005, a Joint ISO-ILAC-IAF Communiqué on the Management Systems Requirements of ISO/IEC 17025:2005 was issued. The text of this communiqué reads as follows:

“A laboratory’s fulfillment of the requirements of ISO/IEC 17025:2005 means the laboratory meets both the technical competence requirements and management system requirements that are necessary for it to consistently deliver technically valid test results and calibrations. The management system requirements in ISO/IEC 17025 (Section 4) are written in language relevant to laboratory operations and meet the principles of ISO 9001:2000 *Quality Management Systems—Requirements* and are aligned with its pertinent requirements.”

Laboratories may find this language useful when discussing the issue of ISO 9001 certification versus ISO/IEC 17025 accreditation with its customers. A copy of the Communiqué may be found on the NVLAP web site.

## A.3 Approved symbols



FOR THE SCOPE OF ACCREDITATION UNDER NVLAP LAB CODE 000000-0

Figure 1. NVLAP logo and caption 1.



NVLAP LAB CODE 000000-0

Figure 2. NVLAP logo and caption 2.



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Height = 1

Width = 2.25 (does not include registration mark)

Aspect ratio of 2.25:1

Figure 3. Aspect ratio of the NVLAP logo (width to height).



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### Approval

A handwritten signature in black ink, appearing to read "Robert E. Butler", written over a horizontal line.

**Robert E. Butler**  
Quality Assurance Director

12 / 10 / 2009

Date

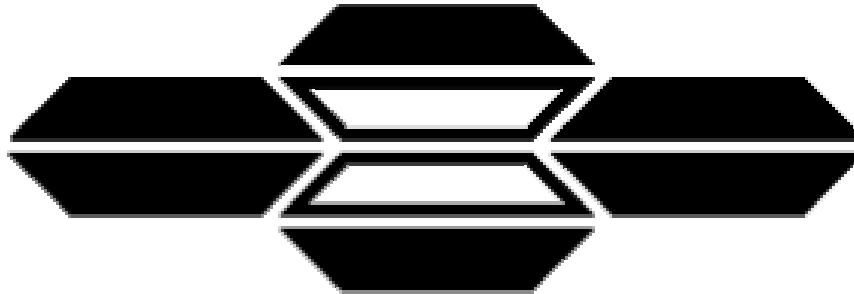
A handwritten signature in black ink, appearing to read "R. Mark Bailey", written over a horizontal line.

**R. Mark Bailey**  
General Manager, President

12 / 10 / 2009

Date

**Asbestos TEM Labs, Inc.**  
**630 Bancroft Way**  
**Berkeley, CA 94706**



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# **Standard Operation Procedures**

## **Bulk Asbestos Analysis By Polarized Light Microscopy**

Quality Manager: Yanxia Xie

General Manager: Mark R. Bailey, R.G., President

Date of Issue: 08/29/2007

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

Polarized Light Microscopy Division

Issue Date: **2007/08/29**

Revision: **1**

SOP#: **0-0**

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## **Table of Contents**

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- SOP 4-04-1 Contract Review
- SOP 4-06-1 Purchasing
- SOP 4-08-1 Complaints
- SOP 4-09-1 Control of Nonconforming Work
- SOP 4-11-1 Corrective Action Request
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- SOP 4-14-1 Internal Quality Audits
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### Technical Requirements Standard Operation Procedures

- SOP 5-02-1 Training
  
- SOP 5-04-2-PLM-01 PLM Analysis – Standard Method
- SOP 5-04-2-PLM-02 PLM Analysis – CAL ARB 435 Method
- SOP 5-04-2-PLM-03 PLM Analysis – Gravimetric Point Count Method
  
- SOP 5-04-6-01 Uncertainty
- SOP 5-04-6-02 Analyst Proficiency Control
- SOP 5-04-6-03 Laboratory Proficiency Control
- SOP 5-04-6-04 Contamination Control
  
- SOP 5-05-1-01 Equipment (Including Computers)
- SOP 5-05-1-02 HEPA Hood/Microscope Maintenance
  
- SOP 5-06-1-01 Reference Standards and Reference Materials
- SOP 5-06-1-02 Refractive Index Liquid Calibration
  
- SOP 5-07-1 Handling of Test Items



Asbestos TEM Labs, Inc.

# Standard Operating Procedure

Polarized Light Microscopy Division

## Table of Contents

Issue Date: **2007/08/29**

Revision: **1**

SOP#: **0-0**

### Revision History

Revision	Date	Revision Notes
0	Feb. 09, 2005	Initial Publication
1	Aug. 29, 2007	Re-numbered Section 4

Approval

*Yanxia Xie*

Author

*Yanxia Xie* Init:

Quality Assurance Manager

*08/29/2007*

Date

*08/29/2007*

Date





Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Document Control**

Issue Date: **2005/02/09**

Revision: **0**

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#### **Purpose**

To control all quality system documents (internally generated and from external sources).

To identify changes made to documents.

To control amendments to documents by hand [if applicable].

To control documents maintained in computerized systems.

Document control ensures that:

- authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed
- documents are periodically reviewed and where necessary revised to ensure continuing suitability and compliance with applicable requirements
- invalid or obsolete documents are promptly removed from all points of issue or use to assure against unintended use
- obsolete documents retained for either legal or knowledge preservation purposes are suitably marked

#### **Scope / Field of Application**

This procedure applies to the Quality Manual, test methods, and standard operating procedures. Also applies to externally generated quality system documents.

**Note** – While there are standard formats for writing test methods and SOPs, documents may take on different formats. These formats may consist of signs, flowcharts, pictures, drawings, sketches, forms, and bulletized lists. Regardless of the format, all quality system documents must be controlled through this procedure.

#### **Definitions and Acronyms**

**Quality manual** – a document stating the quality policy, quality system, and quality practices of an organization (ISO 8402).

**Standard operating procedure (SOP)** – a document that specifies or describes how an activity is to be performed. It may include methods to be used and sequence of operations.

#### **Responsibilities**

Laboratory management ensures that this document control procedure is established, implemented and maintained. The Quality Manager oversees the day-to-day operations of document control. Laboratory personnel are responsible for following this procedure in its entirety.



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# ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

## **Document Control**

Issue Date: **2005/02/09**

Revision: **0**

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### **Materials Required**

Master list

### **Procedure**

The Quality Manager maintains a master list of all controlled documents.

#### **Quality Manual:**

1. The Quality Manual is approved by the President and the signature of this approval is kept on file by the Quality Manager.
2. Controlled hard copies are indicated by the “Controlled Copy” colored ink stamp at the bottom of each page.

#### **Procedures:**

1. Test methods and standard operating procedures are controlled documents with limited distribution. The Table of Contents includes all current and dormant procedures assigned to a given work area and the following information:
  - the code and title of each procedure
  - the revision number of each procedure
  - the effective date of each procedure
  - review date – the date each procedure was last reviewed
  - the status of each procedure (current or dormant)
2. Unique codes are assigned for each procedure.
3. Test methods are written with the following headings:
  - Scope
  - Description of Test Items
  - Holding Times
  - Quantities to be Tested
  - Materials and Equipment Required
  - Physical Environmental Conditions Required
  - Procedure
  - Sample Identification
  - Documentation
  - Safety Measures
  - Method for Data Analysis and Presentation
  - Sensitivity
  - Quality Control Plan



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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Document Control**

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- Reference Procedures
- References
- Revision History
- Appendix

4. SOPs are written with the following headings:

- Purpose
- Scope / Field of Application
- Definitions and Acronyms
- Responsibility
- Materials Required
- Procedure
- Documentation
- Reference Procedures
- References
- Revision History
- Appendix

5. Procedures are peer-reviewed, authorized by management, copies uniquely identified, and distributed to users. Signatures indicate the approval of the procedure.

**Note** – the issue date (see header) is the date the document was issued to the user for review. The effective date (see footer) is the date the document is effectively in place and followed.

6. Obsolete master copies are clearly marked obsolete and archived for at least seven years. All other draft copies and obsolete versions are destroyed to prevent inadvertent use.

7. The Quality Manager maintains the master copies of the most current procedures.

8. The master list of procedures includes:

- code
- title
- revision number
- review date
- status (current, obsolete, dormant, or assigned)
- distribution list (copy number)

#### **Document Review:**

1. Quality manual is reviewed annually by the Quality Manager. Records are kept of this review.
2. Written procedures are reviewed on a biennial basis. The reviewer makes a record of this review, any required changes, and forwards to the Quality Manager.



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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Document Control**

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#### **Document Changes:**

1. Changes to the Quality Manual or a procedure require the same review and approval that performed the original review and approval.
2. Changes are indicated by a vertical line in the left-hand margin adjacent to the revised area. The master index is updated to reflect the changes in revision status and revision release date.
3. Personnel affected by the issue of a revised procedure are informed in writing. Holders of obsolete copies return them to the Quality Manager and place the replacement into the manual.
4. An index of obsolete quality manual sections and procedures is maintained.

#### **Amendments by Hand:**

1. Where permitted, hand written amendments are clearly marked, initialed, and dated on all copies.

#### **Computerized Documents:**

1. Electronic copies of documents are maintained under the authority of the Quality Manager. "Source" documents are standard word-processing documents which can be edited and modified in the course of the evolution of the document. "Published" documents are in Adobe .pdf format, but are not editable by viewers of the documents.
2. Access to electronic copies is password protected.
3. Documents that are revised are maintained in a folder identified as current; the obsolete version is moved to a folder identified as obsolete.

#### **Documentation**

Revisions to Quality Manual sections and procedures are accompanied by a transmittal notice, which details the appropriate additions, removals, and replacements.

<b>Required Record</b>	<b>Custodian</b>
Approved master copies of Quality Manual and procedures	Quality Manager
Controlled hard copies of Quality Manual and procedures	As assigned
Master list	Quality Manager



Asbestos TEM Labs, Inc.

# Standard Operating Procedure

Polarized Light Microscopy Division

## Document Control

Issue Date: 2005/02/09

Revision: 0

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Required Record	Custodian
Document reviews	Quality Manager
Transmittal Notices	Quality Manager

### Reference Procedures

Templates for writing SOPs and test methods.

### References

Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.

### Revision History, Authorship and Approval

Revision	Date	Revision Notes
0	Feb. 09, 2005	Initial Publication

Approval

Yanxia Xie

Author

02/09/2005

Date

Yanxia Xie Init: \_\_\_\_\_

Quality Assurance Manager

02/09/2005

Date



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Contract Review**

Issue Date: **2005/02/09**

Revision: **0**

SOP#: **4-04-1**

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#### **Purpose**

To review requests, tenders, or contracts.

This procedure ensures that:

- the client's requirements including the methods to be used are adequately defined, documented, and understood
- the laboratory has the capability and resources to meet the requirements
- the appropriate test method is selected and capable of meeting the client's requirements

This procedure also describes the activities that take place should there be subsequent amendment to a client's order.

#### **Scope / Field of Application**

This procedure is to be performed by all employees who supply quotations to clients for laboratory services.

#### **Definitions and Acronyms**

**Contract Review** – systematic activities carried out by the supplier, before signing the contract to ensure that requirements for quality are adequately defined, free of ambiguity, documented, and can be realized.

**Requirements for Quality** – expression of the needs or their translation into a set of quantitatively or qualitatively stated requirements for the characteristics of an entity to enable its realization and examination.

#### **Responsibilities**

All employees involved in contract review are to follow this procedure.

#### **Materials Required**

Client information

Price list

Formal service quotations

#### **Procedure**



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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Contract Review**

Issue Date: **2005/02/09**

Revision: **0**

SOP#: **4-04-1**

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### ***Preparation and Review***

1. Upon client inquiry, the following information needs to be obtained:

A. Client

Contact person:

Tel: / Fax:

Address:

B. Objective / goals / required information

Requested analysis:

☐ Qualitative / semi-quantitative, limit of detection:

☐ Quantitative, range of concentration:

C. Costs

Expected costs:

Cost limits:

D. Date of completion / schedule

Date of intermediate results / reports:

Deadline for final results / report:

E. Description of sample(s)

Identification:

Approximate composition:

Main component:

Minor constituent:

Intended use:

Packaging / stability:

Special care for storage / transport / stabilization:

Pretreatment / preconditioning:

Reference materials / reference samples:

F. Methodology

Description of methods used for sampling, sample preparation, measurement

Standard method:

Generic method:

New / adapted method:

Validation required for method:

2. This information is reviewed to determine the laboratory's ability to perform the work requested by the client. If the laboratory has the technical capabilities and resources, continue



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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Contract Review**

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this procedure. Otherwise inform the client that the laboratory is unable to fulfill their request.

3. Quotations for routine requests can be quickly provided through the standard price list.
4. Quotations for non-routine requests take more time to cost out and are formally issued as a service quotation document. This is typically faxed to the client. Each new service quotation is assigned a unique number.

#### ***Acceptance of a Contract***

1. The client sends back the service quotation with their signature within the acceptance period and/or a purchase order referring to the service quotation.
2. When a client submits samples without written authorization and the submission form requests the previously discussed services, a confirmation fax is sent to the client informing them that the work will be performed as proposed.
3. When a client submits samples without written authorization and the submission form requests services that are different from the original service quotation, the client is contacted to clarify the required testing and to obtain written authorization to proceed.
4. When a client submits samples without any prior discussion (re: required testing and pricing), the client is contacted, requirements are reviewed, a service quotation is issued to clarify request and provide pricing.

#### ***Contract Amendment***

Amendments to a contract are reviewed in the same manner as previously outlined.

#### **Documentation**

The manager/supervisor maintains copies of all service quotations issued (and fax confirmation sheets), client authorizations, and any amendment records.

#### **References**

Euarchem / CITAC Guide 2 – Quality Assurance for Research and Development and Non-Routine Analysis.





Asbestos TEM Labs, Inc.

# ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

## **Contract Review**

Issue Date: **2005/02/09**

Revision: **0**

SOP#: **4-04-1**

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### **Revision History**

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0	Feb. 09, 2005	Initial Publication

Approval

***Yanxia Xie***

Author

***02 / 09 / 2005***

Date

***Yanxia Xie*** Init: \_\_\_\_\_

Quality Assurance Manager

***02 / 09 / 2005***

Date



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Purchasing**

Issue Date: **2005/02/16**

Revision: **0**

SOP#: **4-06-1**

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#### **Purpose**

This procedure defines the process for the selection and purchase of services and supplies. This procedure is also used for reception and storage of supplies.

#### **Scope / Field of Application**

This procedure applies to all purchases/acquisitions of services and supplies made by the laboratory.

#### **Definitions and Acronyms**

**Request for Purchase** – Document initiating a procurement of equipment, supplies, or services.

**Qualification Process** – Process of demonstrating whether an entity is capable of fulfilling specified requirements.

**Grade** – Category or rank given to entity having the same functional use but different requirements for quality.

#### **Responsibilities**

The responsibilities of individuals who will perform the process described in this procedure are detailed in the following Procedure section.

#### **Materials Required**

Request for Purchase form

#### **Procedure**

1. The information on the Request for Purchase instructs suppliers to identify packaging supplies and packing slips with the following information, as applicable:
  - name of material
  - vendor's name and address
  - lot number, if appropriate
  - quantity
  - material specification number and date, if appropriate
  - certification documentation, if appropriate



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In addition to the information sent to the vendor, the Request for Purchase also contains the following information for use internally at Asbestos TEM Laboratories, Inc.

- Name of the division initiating the Request for Purchase
- Initials of the Senior Analyst in the division
- A unique Request for Purchase number of the form Request for Purchase-<Division Initials>-<Date as MMDDYY>-<Optional sequence number>, e.g. "RFP-AA-060704-1"
- Applicable SOP #'s for all analytical methods for which the material is being purchased

#### 2) Generation and routing of Request for Purchase forms:

- a) Request for Purchase forms are initiated and filled out by Senior Analysts in the laboratory division initiating the purchase. The Senior Analyst is responsible for ensuring that the any ordered supplies meet the minimum quality requirements for all methods for which the material will be used. One copy of the Request for Purchase is retained by the Senior Analyst. Two copies of the Request for Purchase are forwarded to the Technical Quality Manager
- b) The Technical Quality Manager reviews the Request for Purchase to ensure that the quality standards for chemicals or other supplies meet the requirements of the appropriate test methods, and to ensure that the laboratory maintains appropriate Materials Safety Data Sheets for all chemicals ordered. The Technical Quality Manager retains one copy of the Request for Purchase and forwards one copy to the Purchasing Officer.
- c) The Purchasing Officer places the order with the appropriate vendor.

- 3) Upon receipt in the laboratory, the ordered materials are delivered to the initiating Senior Analyst who checks the shipments of materials received for the correct quantities, for certification, if required, to match the packing slip against the Request for Purchase, and to verify that the supplied materials meet the order specifications for minimum quality standards. If a discrepancy is found that could affect the quality of laboratory output, the material is replaced and a disposition record is kept. If the material is accepted, The Senior Analyst marks his or her copy of the Request for Purchase and also the supplier packing slip with the date of receipt and his or her initials.

- 4) The container is labeled by the Senior Analyst with the date of receipt and the shelf- life expiration date. No reagents, chemicals, standard solutions, or other time-sensitive materials should be used after the expiration of the assigned shelf- life date.

**Note** – if no shelf- life expiration date is available, the laboratory assigns an expiration date of 2 years.

- 5) The dated and initialed supplier's packing slip is delivered by the Senior Analyst to the Quality Manager.
- 6) The Technical Quality Manager marks his or her copy of the Request for Purchase with the date received and forwards the packing slip to the Purchasing Officer for payment.



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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Purchasing**

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- 7) The Requests for Purchase record, receiving documents, and any certifications are used as control over the material being received.
- 8) The Technical Quality Manager periodically requires checks on the validity of a grade or certification of a purchased material. The check can be performed by the laboratory's own capabilities or through a subcontractor. If materials do not meet their specified grade or certification, the vendor is notified and the material is replaced.
- 9) The Senior Analyst monitors the inventory at least twice a year to identify material approaching the expiration date.
- 10) Senior Analysts are responsible for monitoring the rate of usage for consumable materials and for placing an order to replenish stock when appropriate. Typically, a minimum of 90 days stock of consumables is maintained on site at all times.
- 11) The user of an in stock material or supply checks to ensure the material is properly identified and has a current shelf- life expiration date. When more than one container of a material is in stock, the oldest is used first.
- 12) Disposition records are reviewed for trends in vendor performance and to ensure high quality materials and supplies are accepted.
- 13) When the quality of media, reagents, chemicals, solutions or solvents are checked against standards as part of the test method they are used in, they are not checked prior to placing them in storage, other than to validate the identity, shelf- life, or certification, as covered in the steps above.

### **Documentation**

Records for purchasing include:

<b>Required Record</b>	<b>Custodian</b>
Request for Purchase	Senior Analyst
Performance data of contractor performance	Quality Manager
Material and supply inventory	Quality Manager
Packing slips	Office Manager



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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Purchasing**

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### **Reference Procedures**

Test methods specifying the requirements or grade of supplies.

### **References**

Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.

### **Revision History**

Revision	Date	Revision Notes
0	Feb. 16, 2005	Initial Publication

Approval

*Yanxia Xie*

Author

*02 / 16 / 2005*

Date

*Yanxia Xie* Init: \_\_\_\_\_

Quality Assurance Manager

*02 / 16 / 2005*

Date



Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

**Polarized Light Microscopy Division**

### **Complaints**

Issue Date: **2005/02/16**

Revision: **0**

SOP#: **4-08-1**

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#### **Purpose**

To define how to handle and resolve client complaints.

#### **Scope / Field of Application**

A special corrective action request where the problem has been discovered by the client rather than identified and rectified within the laboratory.

#### **Responsibility**

Managers and supervisors continually solicit client feedback.

Employees receiving complaints are responsible for recording the details of the client complaint, do what they can to resolve the immediate problem or assure the client that their complaint will receive immediate attention, inform the client that the laboratory will contact them by a certain time or date, and pass the details of the complaint on to their supervisor and advising them if the nature of the complaint is serious or might lead to legal action.

Managers/supervisors analyse the nature of the complaint (contacting the client for further information if necessary), initiate action to resolve the complaint (keeping records of these actions), contact the client to determine whether the solution is sufficient, implement long-term solutions to prevent the recurrence of this type of complaint (keeping records of these solutions), and monitor the effectiveness of the long-term solution (keeping records of follow-up verifications). Circulate information as to the nature of the complaint to all interested personnel within the laboratory.

The Quality Manager follows up with all appropriate personnel to assure that corrective action has been implemented and demonstrated.

#### **Materials Required**

Corrective Action Request form

#### **Procedure**

##### ***Record-Keeping***

1. Record complaint on a Corrective Action Request form and identify it as a client complaint by checking off the client complaint check-box.
2. Perform whatever immediate corrections can be made and record these details.



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## ***Standard Operating Procedure***

### **Polarized Light Microscopy Division**

#### **Complaints**

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3. Forward to the supervisor's attention.
4. Supervisor determines whether a corrective action needs to be taken to prevent recurrence.
5. A root cause analysis is performed to identify what the true cause of the complaint was and records are kept.
6. Take corrective action to prevent recurrence and record actions.
7. Perform follow-up verification to ensure the appropriate corrective action was taken and effective and record verification.

**Note** - all records are kept on the Corrective Action Request form.

#### ***Resolving Difficult Situations***

1. Listen actively while the client is venting and don't interrupt.
2. Control your tone of voice (calm and low) and body language.
3. Empathize first with a phrase that genuinely shows concern (e.g., "I appreciate that must have been very annoying for you", "I understand how frustrating that is", "I am sorry to hear that"). Do not start off by saying "May I have your name please". Finally, if your laboratory did make an error, don't forget to say sorry - it's surprising how much this actually does mean.
4. Show you are willing to help. Take responsibility for the situation and give your name to establish a personal bridge.
5. Question the client to get the facts (i.e., who, what, where, when, how).
6. Summarize what you've heard by writing it down. If the complaint is over the phone, the employee needs to let the client know that they are recording the complaint so that there is some assurance that the situation is being taken seriously.
7. Say what you can do.
8. Give the client a choice to make the client feel empowered and respected.
9. Follow the action through to completion.



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## Standard Operating Procedure

Polarized Light Microscopy Division

### Complaints

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10. Check that the client is now satisfied by following up.

#### *Written Responses*

1. Any required written responses to clients are authored by the manager / supervisor.
2. Copies of the written response are attached to the Corrective Action Request.
3. Final review is conducted by the Quality Manager.

#### **Documentation**

All records are kept on the Corrective Action Request form.

#### **Reference Procedures**

SOP 4-10-1 – Corrective Action

#### **Revision History**

Revision	Date	Revision Notes
0	Feb. 16, 2005	Initial Publication

Approval

Yanxia Xie

Author

02 / 16 / 2005

Date

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Quality Assurance Manager

02 / 16 / 2005

Date





Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Control of Nonconforming Work**

Issue Date: **2005/02/16**

Revision: **0**

SOP#: **4-09-1**

Page #: **1 of 3**

#### **Purpose**

To control any aspect of testing work or the results of this work that do not conform with its own procedures or the agreed requirements of the client.

#### **Scope**

Nonconforming work or any part thereof.

#### **Definitions and Acronyms**

**Nonconformity** – nonfulfillment of a specified requirement.

**Concession** – written authorization to use or release a product which does not conform to the specified requirements.

**Disposition of Nonconformity** – action to be taken to deal with an existing nonconforming entity in order to resolve the nonconformity.

**Repair** – action taken on a nonconforming product so that it will fulfill the intended usage requirements although it may not conform to the originally specified requirements.

**Rework** – action taken on a nonconforming product so that it will fulfill the specified requirements.

#### **Responsibilities**

This procedure applies to all employees. Any employee can halt work when nonconformances are identified.

Analysts and management personnel that are fully trained in trouble-shooting equipment problems are authorized to resume work that has been corrected before incorrect results have been reported to clients.

The laboratory supervisor is responsible for authorizing the resumption of work after effective corrective action has been taken to prevent the release of unacceptable test results in the future. This requires the use of the corrective action procedure described in SOP# 4-10-1 to find the root cause of the nonconformance and eliminate future occurrences.



Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Control of Nonconforming Work**

Issue Date: **2005/02/16**

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#### **Materials Required**

Corrective Action Request form

Equipment logbook

#### **Procedure**

1. Record the occurrence of a nonconforming event in the Corrective Action Request form / equipment maintenance logbook.
2. Report the nonconforming event to the lab supervisor.
3. Suspend further work and begin an investigation to correct the nonconformance.
4. Evaluate the significance of nonconforming work.
5. Notify the client of all incorrectly reported results, if applicable.
6. Correct nonconformance, if possible.
7. Initiate corrective action to prevent recurrence of the nonconformance.
8. Repeat the test or analysis, if possible.

#### **Documentation**

Nonconforming samples are recorded on accompanying submission forms. Equipment problems are recorded in the appropriate equipment maintenance log. All nonconformances are recorded in the Corrective Action Request form.

Root cause analysis and closed loop corrective actions taken to prevent recurrence of nonconformances are recorded as described in SOP# 4-10-1.

<b>Required Record</b>	<b>Custodian</b>
Signed Waiver or Deviation when required by client agreement	Laboratory Manager
Nonconformance Report (first section of CAR)	Laboratory Manager
"Calibration Void – Do Not Use" label	Laboratory Manager



Asbestos TEM Labs, Inc.

# Standard Operating Procedure

Polarized Light Microscopy Division

## Control of Nonconforming Work

Issue Date: **2005/02/16**

Revision: **0**

SOP#: **4-09-1**

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Required Record	Custodian
"Out of Service – Do Not Use" label	Laboratory Manager

### Reference Procedures

SOP 4-10-1 - Corrective Action

### References

Quality Manual – Sections 5.5.7 and 5.5.8

### Revision History

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0	Feb. 16, 2005	Initial Publication

Approval

Yanxia Xie

Author

02 / 16 / 2005

Date

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Quality Assurance Manager

02 / 16 / 2005

Date



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Corrective Action Request**

Issue Date: **2007/07/29**

Revision: **1**

SOP#: **4-11-1**

Page #: **1 of 4**

#### **Purpose**

To detail the systematic approach for closed loop corrective action to find and eliminate the actual root cause(s) of nonconforming work or departures from policies and procedures in the quality system or technical operations.

#### **Scope / Field of Application**

This procedure is applicable to all organizations providing products and services governed by the requirements specified within the Quality System.

#### **Definitions and Acronyms**

**Correct** – action taken to fix a mistake or problem.

**Corrective Action** – action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

**Corrective Action Request (CAR)** – request to initiate corrective action.

**Root Cause** – fundamental deficiency that results in a nonconformance and must be corrected to prevent recurrence of the same or similar nonconformance.

#### **Responsibilities**

The appropriate authorities for the implementation of a corrective action include:

##### **CAR Originator shall:**

- initiate CAR when a need for corrective or preventive action is identified
- Any member of the laboratory staff may initiate a CAR when he or she believes there may be an issue with the quality of analytical results or laboratory service provided to clients

##### **Quality Assurance Manager shall:**

- assign CAR number and update master CAR log
- review and assign CAR to the appropriate Senior Analyst
- monitor CAR status to ensure complete and timely response
- approve proposed corrective action
- approve close-out of corrective action
- track CAR completion dates
- file completed CARs and update log
- train and support users of the CAR system, if needed



Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Corrective Action Request**

Issue Date: **2007/07/29**

Revision: **1**

SOP#: **4-11-1**

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- ensure that appropriate records are created and preserved and that any needed changes in quality procedures, the Quality Manual, Standard Operating Procedures, or other laboratory documents, practices, or procedures are modified as appropriate to reflect improvements revealed by the process.

#### **Senior Analyst shall:**

- review CAR and determine if corrective action is warranted
- determine who will participate in needed action(s)
- review corrective action to verify implementation
- review and evaluate client comments and generate CARs if appropriate

#### **Senior Analyst or subordinate Analysts at the direction of the Senior Analyst shall:**

- investigate and determine root cause(s) of nonconformance
- identify and implement timely corrective or preventive action

### **Materials Required**

Corrective Action Request form

CAR Log

### **Procedure**

#### **General**

1. Each test method and/or standard operating procedure specifies the required quality control (i.e., blanks, spikes, positives, negatives, reference values, workmanship standards).
2. Compare actual quality control results with expected specified results.
3. Report any nonconformances as outlined in SOP# 4-9-1.
4. The investigator initiates research into the root cause of the nonconformance and designs a plan.
5. Take corrective action to implement plan and record on CAR form.
6. Initiator, manager or auditor follows up on the effectiveness of the action taken and record on CAR form.
7. Act on any necessary actions found during the follow-up.



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Corrective Action Request**

Issue Date: **2007/07/29**

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#### **Detail**

When a QC analysis gives a contradictory result, the QA Officer is notified. The source of the discrepancy is determined immediately (quantification errors are not as critical and are usually dealt with on a longer-term basis as consistent biases are discovered). Equipment and materials are generally checked for defects and contamination, and additional slides are prepared and analyzed. The type of corrective action that is taken to correct discrepancies in analytical results depends on the type and frequency of error. Examples of such actions include:

- 1) It may be determined that the sample contains very close to 1% asbestos and that the discrepancy was caused by normal sample heterogeneity. No corrective action is taken.
- 2) The discrepancy may be caused by a high degree of heterogeneity in the sample. If the additional slides analyzed indicate that this is the case, an average asbestos concentration is determined through homogenization efforts or more thorough picking of material from the sample. If the original analysis was in error and the results were reported to the client, the client is notified and an amended report is prepared. It is emphasized to the client that sample heterogeneity caused the change in the result and that such changes are unavoidable on occasion.
- 3) If it is determined that an analyst mis-identified a fiber type, the properties of the fibers in question and the procedures for proper identification are reviewed by the analyst with the guidance of the QA Manager. Usually, the problem is quickly identified and corrected. If consistent deficiencies are found via QC checks, more thorough discussion and examination of internal and external standard materials by the herring analyst are also conducted. If the original analysis was in error and was reported to the client, the client is contacted and an amended report is prepared.
- 4) If it is determined that an analyst's procedure was in error (e.g. overloading of sample material), the QA Manager suggests corrections and checks on their implementation in the following days.

An entry is made in monthly PLM report for each corrective action. The QC Officer keeps track of errors by each analyst through a monthly compilation of corrective actions and conducts discussions with analysts who appear to have consistent problems.

Quantification Errors. Acceptable limits for quantification errors vary somewhat with the nature of the sample, but for general purposes, the "factor of two" guideline is used. For example, a 10-20% vs. 20-30% discrepancy is acceptable whereas a 1-5% vs. 20-30% discrepancy is not.

#### **Documentation**

Root cause analysis and corrective action taken is recorded on the CAR form.



Asbestos TEM Labs, Inc.

# Standard Operating Procedure

Polarized Light Microscopy Division

## Corrective Action Request

Issue Date: 2007/07/29

Revision: 1

SOP#: 4-11-1

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Required Record	Custodian
Completed CAR form	Quality Manager
CAR Log	Quality Manager

### Reference Procedure

SOP 4-09-1 – Control of Nonconforming Work

SOP 4-08-1 – Complaints

### References

Quality Manual Section 4.8.1 and 4.9.2

### Revision History

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0	Feb. 16, 2005	Initial Publication
1	Aug. 29, 2007	Renumbered this section

Approval

Yanxia Xie

Author

08 / 29 / 2007

Date

Yanxia Xie Init: \_\_\_\_\_

Quality Assurance Manager

08 / 29 / 2007

Date



Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Preventive Action**

Issue Date: **2007/08/29**

Revision: **1**

SOP#: **4-12-1**

Page #: **1 of 3**

#### **Purpose**

To implement opportunities for needed improvement and prevent potential sources of nonconformance.

#### **Scope / Field of Application**

Any procedure or process relating to the quality system or of a technical nature.

#### **Definitions and Acronyms**

**Preventive Action** – action taken to eliminate the causes of potential nonconformity, defect, or other undesirable situation in order to prevent occurrence.

**Preventive Action Request (PAR)** – request to initiate preventive action.

#### **Responsibilities**

**Facilitator** - initiator of the preventive action or the Senior Analyst of a laboratory division.

**Team members** - those involved in preventing potential nonconformances or improving processes. Members may all be working in a similar area if the issue is isolated or from multiple areas if the solution requires input from various experts.

#### **Materials (and Skills) Required**

1. Quality tools to identify the root cause of a potential problem rather than just a superficial cause.
2. The ability to take a process focus rather than an organizational focus (e.g., the process is the problem rather than employees are the problem).
3. PAR form

#### **Procedure**

1. Select the potential problem (or process to be improved).

**Note** - when a team is being utilized, the team members must fundamentally agree that a potential problem exists. They must review the background and what drives the





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## Standard Operating Procedure

Polarized Light Microscopy Division

### Preventive Action

Issue Date: 2007/08/29

Revision: 1

SOP#: 4-12-1

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process and then clarify their thoughts by writing a problem statement. The quality tools that may be useful for this process include “Brainstorming”, “Affinity Diagrams”, “Interrelationship Diagram”, “Voice of the Customer”, and “Gap Analysis”.

2. Describe the current situation by collecting baseline data, describing the process, confirming the problem statement, and setting improvement targets.

**Note** - the quality tools that may be useful for this process include “Affinity Diagrams”, “Bar Charts”, “Control Charts”, “Histograms”, “Voice of the Customer”, “Flowcharts”, “Pareto Analysis”, “Gap Analysis”, and “Benchmarking/Competitive Analysis”.

3. Choose the most likely cause by conducting a root-cause analysis, prioritizing root causes, and using a cost/benefit analysis.

**Note** - the quality tools that may be useful for this process include “Cause and Effect Diagram/Fishbone”, “Pareto Analysis”, “Prioritization Matrix”, “Spreadsheet”, and “Value Analysis”.

4. Develop a solution/action plan by identifying solutions, mapping solutions to root causes, conducting cost/benefit analysis, assess barriers to implementation, identifying countermeasures to barriers, and writing an action plan.

**Note**- the quality tools that may be useful for this process include “Benchmarking/Competitive Analysis”, “Value Analysis”, “Force-Field Analysis”, “Brainstorming”, and “Gantt Chart/Action Plan”.

5. Obtain management approval to implement improvements.
6. Implement the solution/action plan by conducting a test pilot study, evaluating test results, revising the action plan, implementing and monitoring the revised plan, and summarizing the results.

**Note** - the quality tools that may be useful for this process include “Checksheets”, “Control Charts”, “Line Chart/Run Chart”, “Histograms”, “Pie Charts”, “Scatter Diagram”, “Gantt Chart/Action Plan”, and “Work Breakdown Structure”.

7. Review and evaluate results to determine whether solution did work through the completion of the action plan, if the best solution was selected, and if the correct cause was selected.



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**Note** - the quality tools that may be useful for this process include “Line Chart/Run Chart”, “Gannt Chart/Action Plan”, “Planning Matrix”, and “Cause and Effect Diagram”.

8. Reflect and act on learning.

**Note** - the quality tools that may be useful for this process include “Lessons Learned”, “Plan-Do-Check-Act (PDCA)”, and “Planning Matrix”.

### Documentation

Records are logged, identified and kept on file as laboratory improvements.

### Reference Procedures

SOP 4-11-1 Corrective Action

PAR form

### References

Nancey R. Tague. 1995. The Quality Toolbox. ASQ Press.

### Revision History

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Author

08 / 29 / 2007

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Quality Assurance Manager

08 / 29 / 2007

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Asbestos TEM Labs, Inc.

# **Standard Operating Procedure**

Polarized Light Microscopy Division

## **Control of Records**

Issue Date: **2007/08/29**

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### **Purpose**

To identify, collect, index, access, file, store, maintain, protect, backup, and dispose quality and technical records. To outline procedures for the protection and backup of data/records held on computers.

### **Scope / Field of Application**

This procedure applies to all quality and technical records. Quality records include audit reports, management review, corrective action requests, and preventive action requests. Technical records include observations, calculations, derived data, calibration records, personnel records, and test reports.

**Note** - forms are not records until they are completed.

### **Responsibilities**

**Senior Management (President or Laboratory Manager) shall:**

- specify any alternative retention period

**Quality Manager shall:**

- ensure that records specified in the Quality System are handled in accordance with the requirements of this document
- ensure the unique indexing of records

**Records custodian (all personnel) shall:**

- collect, store, and maintain records for the minimum retention period or the period designated by management
- ensure that records retained are legible
- for any technical information, analytical data, or quality control records or data, when an alteration to an original entry becomes necessary, no attempt shall be made to obliterate, erase, black out, or otherwise destroy or make unreadable the original entry. Corrections are made in such a manner as to clearly reveal that the original entry has been stricken out and a new entry made.

### **Materials Required**

Master index of records

### **Procedure**

#### **General**



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#### **Identification**

Records are appropriately identified by a descriptive title clearly labeling the record.

#### **Collection**

The personnel/user identified for each record is responsible for collecting the record.

#### **Indexing**

Each record is assigned a unique name, number or alphanumeric identification, and date to distinguish it from other records with the same identification. This is referred to as the master index of records.

#### **Accessing**

Records are readily accessible to individuals requiring information contained in the record.

Records are available to clients for the period agreed to per client agreements. Subcontractor's records, as specified by contract, are made available upon request.

#### **Filing**

Filing is considered the location where active records are kept. All records are physically or electronically filed by a method which enhances accessibility and retrieval by a user.

If electronic files are used, a backup system or other suitable measures to prevent record loss is implemented.

#### **Retention**

A record's "retention" time refers to how long it is kept before it is either discarded or destroyed, or sent to off-site for long-term storage. Records are retained on-site for the minimum retention time of 7 years. Records may be retained longer than the minimum retention time for the convenience of the laboratory. Retention times for work area-specific records are determined by the laboratory and stated in their Quality System documents.

***Note on past practices and inconsistent record retention times:*** Effective December 16, 2004, it became the Policy of Asbestos TEM Laboratories, Inc., to retain all documents for a minimum of ten years (See Section 4.13 of the Quality Manual.) Prior to this date, records management and retention practices were inconsistent. Older documents are available for varying periods but all records are now being retained for ten years.



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#### **Maintenance**

All records are filed and stored in an office or laboratory environment unless specific media and/or special environmental control is specified to prevent damage, deterioration, or loss.

#### **Disposition**

Records are disposed when the retention time has been exceeded. Disposal of all records shall be including shredding to ensure that confidential information cannot be retrieved from the disposed materials. Records may be retained for longer than the minimum retention period, at the discretion of senior management.

#### **Storage**

After the minimum on-site retention time, records may be moved to long-term storage at the discretion of senior management.

### **Control of PLM Testing Report**

Inaccuracies in Reporting are avoided by repetitive checks of calculated results (for verbal transmission) and of printed reports against the data entered on the count sheets and log sheets.

#### **A) Verbal Reports.**

While it is urgent that results be transmitted verbally as soon as possible after the analysis has been completed, careful checks of the accuracy of the data and calculations are more important. A record track of the verbal report should be entered to the LIMS database along with the date and time of the verbal report.

#### **B) Written Reports.**

The data from the PLM analysis count sheets is entered into the report data sheet form on the computer and an initial hard copy of the report and accompanying cover letter are generated. Any deviations from standard procedures are described in the cover letter. This is returned to the analyst, who checks all of the information on the data sheet and cover letter for accuracy. When any revisions are completed, a final copy is generated on the company letterhead, is signed by the analyst, and is submitted to the laboratory manager for a final review.

#### **C) FAX Reports.**

Fax copies of written reports may be sent to the client upon completion of the written report. All fax copies must be followed up by mailed hard copy reports.

#### **D) Archival of Reports**



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The archival procedures are designed to make records and materials readily accessible to laboratory employees (only), to maintain and to maintain security and ensure survival of the following items for a minimum of three years:

#### Hard Copies

- i). Analytical Data - Hard copies of log sheets, custody records, reports, and count/data sheets are stapled together by lot and are filed until payment is received by login number in the green fireproof filing cabinet in the office area, or after payment, are archived in file boxes and stored in the storage or office areas. These files are kept confidential: Only company employees may access them and the files of each client are shown only to a confirmed member of that client's staff. Hard copies of QA procedures documents are stored in the head PLM analyst's desk.
- ii). QA/QC Data - Hard copies of contamination monitoring data, calibration and verification data, quality control activities and results, and equipment maintenance are kept in log books, or in QC data files for a minimum of three years.

#### Computer Archives

Log sheets and reports are generated using the computers, and periodic archival of these documents is performed by both disk and tape backup. A quick backup of the pertinent PLM database files on the computer network server is made daily onto a client computers hard-drive. A comprehensive backup of the computer server is made weekly via a tape backup system and stored in the fire-proof filing cabinet.

### **Documentation**

#### Master list of records

Typical records maintained in the laboratory include:

- instrument and equipment maintenance logbooks
- calibration record of instruments and analytical processes
- records associated with test method quality control plans (e.g., control charts)
- records associated with approved SOPs
- spreadsheets used to calculate accuracy and precision of instruments
- standard logbooks
- sample logbooks
- analyst notebooks
- sample preparation notebooks

### **Reference Procedures**

All Quality System procedures resulting in the production of records.



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### **Control of Records**

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#### **References**

Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.

ISO 9001. 1994. Quality Management Systems – Requirements.

#### **Revision History**

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Author

***08 / 28 / 2007***

Date

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Quality Assurance Manager

***08 / 28 / 2007***

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Asbestos TEM Labs, Inc.

# **Standard Operating Procedure**

Polarized Light Microscopy Division

## **Management Review**

Issue Date: 2007/08/29

Revision: 1

SOP#: 4-15-1

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### **Purpose**

To regularly review the Quality System by the laboratory's management team

To ensure that:

- the Quality System continues to be effective and suitable - fulfilling the changing and future needs of the laboratory and its clients
- the Quality Management System is updated as necessary
- the results of Internal Audits are reviewed
- the defined Quality System is being implemented and followed

### **Scope / Field of Application**

Laboratory Quality System

### **Definitions and Acronyms**

**Management Review** – formal evaluation by top management of the status and adequacy of the quality system in relation to quality policy and objectives.

### **Responsibility**

#### **The President shall:**

- call Management Reviews at regular intervals - not greater than twelve months, or more frequently at his/her discretion
- decide who should attend
- allocate follow-up actions and timelines to specific personnel

#### **The Quality Manager shall:**

- provide a summary of the Internal Audit reports that have been completed since the last Management Review meeting
- archive the Minutes of the meeting as Quality Records
- provide a summary of the Corrective Action reports and Client Complaints raised since the last Management Review meeting - paying particular attention to those which remain unresolved
- provide a summary of supplier/subcontractor performance reports since the last Management review meeting

### **Materials Required**

Internal audit reports

Management review meeting minutes

Corrective Action Requests

Client complaints





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#### **Procedure**

The minutes taken reflect the following items:

1. Date, attendees, and the individual completing the minutes.
2. A review of the minutes from the previous management review and any action items.
3. New items for review may include:
  - Business planning – forecasting
  - Internal audit status
  - Corrective actions
  - Preventive actions
  - Process improvements
  - Training (issues and needs)
  - Equipment (calibration and maintenance program)
  - Client complaints
  - Resources - personnel (absence, increasing work load, surprises from clients)
  - Resources - equipment (time, capacity, failure)
  - External audits
  - Quality policy statement
  - Client satisfaction
  - Quality goals
  - Proficiency results
4. Discussion may identify trends.
5. Action items that are identified should be assigned to individuals and an appropriate completion date agreed upon. Any communication plan is noted.
6. Any other business discussed is recorded with action items as appropriate.

#### **Documentation**

Minutes from the Management Review meeting are signed by the President and the Quality Manager. They are maintained by the President.

#### **References**

Quality Manual



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Quality Assurance Manager

08 / 29 / 2007

Date

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Asbestos TEM Labs, Inc.

# ***Standard Operating Procedure***

Polarized Light Microscopy Division

Issue Date: **2005/02/09**

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Asbestos TEM Labs, Inc.

# **Standard Operating Procedure**

**Polarized Light Microscopy Division**

## **Training**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-02-1**

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### **Purpose**

This procedure describes:

- the assessment of employees' training requirements needs to assure satisfactory performance
- the provision of specific training and general quality awareness training
- the maintenance of Training Records

### **Scope / Field of Application**

This procedure applies to all employees.

### **Definitions and Acronyms**

**Competence** – ability consisting of theoretical knowledge, practical skills, and attitudes.

**Proficiency testing** – determination of the laboratory calibration or testing performance by means of inter-laboratory comparison.

**Skills** – ability to apply knowledge effectively and readily in performance.

**Training** – a process to provide and control competence to meet requirements.

### **Responsibilities**

#### **Laboratory Manager shall:**

- Ensure new hires possess qualifications, such as education, experience, and professional credentials, which meet the requirements specified by job descriptions.
- Ensure that the employees are trained for the assigned jobs.
- Retain records of these assessments and training along with any supporting documentation (certificates, diplomas etc.).
- Prepare job descriptions when a new position is established or duties of the position change.
- Identify continuing training requirements such as additional formal training, on-the-job training (OJT), and certification.
- Develop training plans that address any gaps between current and required knowledge, skills, and competencies needed to perform assigned tasks
- Identify positions needing certification or special process qualification
- Ensure only qualified personnel perform work affecting quality or ensure that tasks performed by employees who have not yet received appropriate training are reviewed by an appropriately qualified individual.



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# **Standard Operating Procedure**

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## **Training**

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### **All Employees shall:**

- Bring to the attention of the manager their needs for specific or general training
- Attend and participate in training sessions provided, whether in- house or external

### **Senior Analysts shall:**

- Perform specific training (test methods, quality controls used...)
- Record the training performed with the training record log.

## **Materials Required**

Training Record form

## **Procedure**

The Laboratory Manager uses the job description to identify and document education, experience, and professional credentials of each position.

The Training Review is an informal discussion between the Laboratory Manager and each employee - in which the employee's job description, current skills and knowledge and current/future responsibilities are compared in order to identify areas where additional training is necessary or beneficial.

### ***Introductory Training***

Upon date of hire, the Laboratory Manager introduces the new employee to a general set of training requirements that must be completed prior to any further activities in the laboratory.

This training includes:

- an overview of the company's history and its business philosophy
- a walk-through of the facility
- a review of the company's personnel/orientation manual
- a review of the safety and chemical hygiene plan and a safety equipment walk-through/usage review

### ***QA/QC Training***

It is essential that the employee have a thorough understanding of the general quality assurance/quality control principles and objectives that are the underlying foundation of all work performed. Accordingly, a detailed review of the Quality System is performed with the Quality Manager.



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#### ***Laboratory Specific Training (Test Methods and SOPs)***

Laboratory specific training includes:

- detailed training on the instrument(s) which the employee will use
- thorough coverage of the methods and SOPs to be employed
- Training about data interpretation, calculating, and reporting

#### **The trainee:**

- reads the test method / SOP
- reads the Material Safety Data Sheets (MSDS) of required materials
- observes the trainer perform the procedure
- practices the procedure
- performs the procedure, under the direction of the trainer, using split samples, spiked samples or proficiency samples
- performs the procedure, under direction of the trainer, using client samples
- reads and then reports results to the trainer

Unsatisfactory results require re-training. The trainee is deemed competent by the trainer when they produce satisfactory results. The trainer performs a follow-up evaluation to determine the effectiveness of training within one month after training.

The Laboratory Manager is responsible for ensuring effective training. Upon completion of initial training, all analysts are required to prove proficiency prior to analyzing client samples.

#### ***Change Control***

All employees are to be advised that they perform their jobs as instructed or as covered by standard operating procedures (SOP's). They are **NOT** allowed to change tasks covered by SOP's until the change is approved according to the document control SOP (SOP # QSP 4-3-1).

Further procedures are defined on an *ad hoc* basis at the time training needs are identified for each employee.

#### ***Performance Appraisal***

The Supervisor shall use the employee performance appraisal to address the following:

- performance planning discussions with each employee
- employee's knowledge of current Quality System procedures and work instructions affecting the performance of their duties
- preparation of a performance plan to be signed by the employee
- a mid-term review of the current performance plan
- a year-end discussion with the employee and assessment of the year's performance

The Supervisor shall ensure that formal, informal, or OJT training is provided to fill the gap(s) identified in the performance appraisal.



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At each scheduled employee performance review, the Supervisor shall assess the effectiveness of training provided. Ineffective training will be addressed and a remedy will be identified in the next performance appraisal.

Refer to Appendix on the specific training requirement and procedure for all the PLM analysts.

### Documentation

All activities relative to training will be thoroughly documented and maintained in each employee's personnel training file. Included in this file are copies of academic transcripts and/or degrees, resumes, job descriptions, all in-house training records and all documents pertaining to external training (e.g. seminars, instrument manufacturer training courses, etc.).

All classroom and on-the-job training are recorded by the trainer on a Training Record Sheet. Records should include orientation, Quality Manual review, and appropriate SOPs and test methods.

Required Record	Custodian
Employee Performance Appraisal	Laboratory Manager
Position Description	Laboratory Manager
Additional training records	Laboratory Manager

### Reference Procedures

All procedures requiring training.

### References

Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.

ISO 10015 – Quality Management Guidelines for Training



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### **APPENDIX PLM Analyst Training Manual**

#### **I. Introduction**

Personnel hired at Asbestos TEM Laboratories for work in the PLM laboratory should have a background in optical mineralogy or a related field. All new analysts shall be required to read all Asbestos TEM Laboratories manuals documenting standard operating procedures, as well as standard references on the nature, hazards, occurrence, and identification of asbestos. The length of the training period will be no less than five working days. Said period will depend on the analyst's ability to identify and quantify asbestos and to follow all laboratory procedures, and will be determined by the Lab Director.

Training will include preparation and examination of a wide variety of standards and common bulk materials, including analyses of samples previously reported to clients. The new analyst's results during training will be recorded, but not reported to clients. During the first 5 working days following training, at least 20% of reportable analyses done by each new analyst will be repeated by a supervisor, regardless of the new analyst's experience. During the first few weeks following training, the progress of the new analyst will be closely monitored by senior personnel.

The purpose of the intensive training program at Asbestos TEM Laboratories is to give all analysts the capability to generate accurate and reproducible results by learning and applying the principles of optical mineralogy. Special emphasis is given to the safe handling of hazardous sample materials and reagents. Each analyst shall proceed through three levels of competence: 1) trainee; 2) supervised analyst; and 3) experienced analyst. Only Level 3 analysts shall be judged as qualified to train new analysts.

A file is kept which contains records of or references to records of the training of each analyst. This file also contains records of corrective actions and reviews after training.

#### **II. Level I - Trainee**

##### **A. Theory**

All new analysts undergo an initial assessment of their background, mineralogical knowledge, and specific knowledge of asbestos. This information is recorded by a supervisor (a qualified Level 3 analyst). Trainees must then read the laboratory manual and review basic references C-3 on optical mineralogy, as needed. Subsequently, they shall become familiar with federal, state, and local regulations of bulk asbestos analyses.

##### **B. Analysis of Standards**

A part of the Asbestos TEM Laboratories collection of reference samples is used to familiarize new analysts with the characteristic optical properties of all asbestos minerals.



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and other common substances found in bulk samples. The trainees will be supervised as they carefully study the samples, characterize their component substances, and record all of the optical properties used in the identification of the components. The trainees will also be required to show proficiency on asbestos look-alike materials (e.g. wollastonite). The trainees shall also be tested using prior NIST NVLAP PAT round test samples, especially those which are difficult and potentially misleading.

#### **C. Handling of Samples and Laboratory Equipment**

Trainees are introduced to Asbestos TEM Laboratories sample log- in procedures, criteria for acceptance and rejection of samples, the diversity and characteristics of sample containers, and safe handling of samples and analytical equipment before, during, and after analysis.

#### **D. Sample Preparation and Analysis**

Trainees are instructed in the basic techniques of slide preparation. Emphasis is given to proper selection of representative samples and to even distribution of material. A wide range of building materials is examined. The prepared slides are then analyzed and evaluated by both the trainee and the supervisor. Samples requiring special attention, such as floor tiles and soil samples, are treated in detail. The trainee's ability to quantify percentages of the various components is discussed and tested. The numbers of hours spent by the supervisor and analyst in each of the above activities are recorded and placed in the appropriate personnel files.

During Level 1 activities, the trainee must show proficiency in recognizing common asbestiform and other materials, determining the optical properties of unknown minerals, and verifying identification by using common references on optical mineralogy. Once this has been accomplished, the trainee is admitted to the comprehensive post-trainee exam (PTE), consisting of written, oral, and practical parts and designed to meet the Lab requirement of mineralogical competence. Satisfactory performance on the PTE is a prerequisite for admission to Level 2.

### **III. Level 2 - Supervised Analyst**

Supervision of a Level 2 analyst is required during the first several weeks of work. During this period, the analyst shall be tested frequently for accuracy and precision, utilizing the advance set of internal reference samples. The number of "replicated" and "duplicates" of samples originally done by Level 2 analysts should be no less than 20% of all analyses.

As in Level 1, the time spent in Level 2 activities by both the analyst and the supervisor is recorded and entered in their personnel files. Satisfactory performance at Level 2, as judged by supervisors, is a prerequisite for admission to the Advanced Proficiency Exam (APE). The second requirement is completion of a minimum of 100 original analyses. The APE is similar in organization to the PTE, but more difficult. Passage of the APE results in advancement to Level 3.



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#### **IV. Level 3 - Experienced Analyst**

A Level 3 analyst should perform analyses with minimum supervision aside from routine QA analyses (including internal proficiency testing) and discussions with the Laboratory Director. All corrective actions are recorded and entered into the analyst's and supervisor's personnel files.

#### **PLM Post-Training Exam**

- 1) What is the sign of elongation of Wallastonite?
- 2) What color is Crocidolite?
- 3) What is the refractive index of Chrysotile in the perpendicular direction (give a range, if necessary)?
- 4) What common fiber found in many materials usually has a ribbon-like morphology?
- 5) Is the birefringence of calcite high, medium, or low?
- 6) What is the maximum extinction angle of Tremolite?
- 7) Which is the best oil for identifying Amosite (1.550, 1.604, 1.640, 1.680)?
- 8) Does the refractive index of chrysotile change when the sample is burned?
- 9) What type of asbestos is found in many floor tiles?
- 10) What is the refractive index of Talc likely to be in the parallel direction?
- 11) What are the refractive indices of gypsum (which oil are they closest to, and are they less than or greater than that oil)?
- 12) How are blanks prepared?
- 13) How are the refractive indices of the oils checked?
- 14) What is the sign of elongation of Crocidolite?
- 15) What is the extinction angle of anthophyllite?
- 16) What is the refractive index of cellulose in the parallel direction?
- 17) You are looking at a fiber with a refractive index that is lower than the oil's. As you move the stage down, do the bright Becke lines move into or out from the fiber?
- 18) Which of the asbestos minerals is usually pleochroic?
- 19) Which common fibrous building material is isotropic?
- 20) How can one reliably distinguish polyethylene from chrysotile?



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**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

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#### **Purpose**

To outline the equipment and procedures used by Asbestos TEM Laboratories in the analysis of bulk asbestos by the technique of polarized light microscopy which is based upon the EPA "Interim Method for the Determination of Asbestos in Bulk Insulation Samples", and the EPA document, "Asbestos-Containing Materials in Schools; Final Rule and Notice", 40 CFR Part 763.

#### **Scope / Field of Application**

Any regular PLM analysis requested by a client.

#### **Definitions and Acronyms**

##### **What is Asbestos?**

Asbestos is a family of minerals with a set of unique properties, including high tensile strength, resistance to heat and corrosion, and excellent binding characteristics. These properties have been found to be very useful to mankind with the result that they have been widely used in a variety of construction and fire-proofing materials. Unfortunately, voluminous medical evidence has been gathered indicating that exposure to asbestos has the potential to cause cancer in humans. This has led to a widespread effort to document the location, amount, and condition of asbestos-materials in buildings where exposure to humans could lead to adverse health effects. It is the purpose of Asbestos TEM Laboratories to assist in this effort to document the presence of asbestos in buildings and other materials through the application of proven scientific asbestos analytical techniques.

The minerals which have been designated as asbestos by the EPA (Chrysotile serpentine and the amphiboles - amosite, anthophyllite, tremolite, actinolite, and crocidolite) are those minerals which Asbestos TEM Laboratories calls asbestos when found in laboratory samples. Other minerals exist which also have asbestiform habits and similar physical characteristics, but these are not covered by current regulatory requirements and are not considered asbestos, i.e. attapulgite, sepiolite, palygorskite.

#### **Analytical Hardware requirements**

##### **A. Apparatus for Gross Examination**

1. Magnifying Lens, 10X
2. Light Source: fluorescent and incandescent lights
3. Hand Tools: tweezers, scalpel, razor blades, probes, etc.
4. Petri Dish: clean glass plate



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#### **B. Apparatus for Sample Preparation**

1. Filtered Ventilation System
2. Microscope Slides
3. Cover Slips
4. Disposable Gloves
5. Hand Tools
6. Mortar and Pestle
7. Hot Plate
8. Cigarette Lighter

#### **C. Apparatus for Identification and Quantification**

1. Polarized Light Microscope
  - a. Polarizer
  - b. Analyzer
  - c. Port for Wave Retardation Plate
  - d. 360° Graduated Rotating Stage
  - e. Substage Condenser
  - f. Lamp
  - g. Lamp Iris
  - h. Condenser Diaphragm
  - i. Objective Lenses - 4X, 10X, 10X Central Stop Dispersion Staining, 20X, 40X,
  - j. Ocular Reticule (10X) w/ Cross Hair
  - k. Retardation Plate - First Order Red, 550 nm

#### **D. Reagents for Sample Preparation**

1. Refractive Index Liquids:  
1.490-1.570 and 1.590-1.720 in 0.002 or 0.004 Step Increments
2. Distilled Water
3. Mineral Oil
4. Dilute HCl acid

#### **E. Analytical Standard Reference Materials**

1. U.C. Berkeley Mineral Collection Reference Standards
2. NIST Asbestos PAT Round Reference Sample Sets

## **Analytical Procedures**

#### **A. Sampling**

The collection of bulk material that will be analyzed for asbestos content is beyond the scope of this method. The analyst must assume that the samples were taken according to the prescribed guidelines for sample collection (U.S. Environmental Protection Agency, 1979,



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1980, 1985, 1987). In the case of doubt as to the sampling procedures, the analyst will not proceed with the analysis until the client has been notified and the problem resolved.

#### **B. Data Review and Transcription**

Prior to sample analysis, all paperwork sent to the analyst after sample log-in (Covered in the QA/QC Manual) is reviewed by the analyst for accuracy. A sample is then chosen from the sample lot, and all relevant data concerning that sample is transcribed to the PLM Data Sheet (See Appendix A) to be used during the analysis. This information includes Laboratory Sample ID#, Client Sample ID#, Job Site, Location, and Description.

#### **C. Gross Examination**

The sample chosen for analysis is closely reviewed with a low power magnifying glass to aid the examination process. Notes are made concerning the following:

1. Homogeneity - If sample is heterogeneous, briefly describe the different materials.
2. Texture - e.g. fibrous, matted, rubber, clotted, etc.
3. Color
4. Friability
5. Gross estimated percentage of asbestos.

The distinction between homogeneous and inhomogeneous material is subjective. A sample is considered to be inhomogeneous if discontinuities between material types are visually significant in hand specimen. Material with obvious layers is the most common case, examples are: 1) roofing samples with backing paper, roofing felt, tar paper, 2) cored samples of insulation material containing various wraps and insulation, and 3) floor tiles and mastic. If such material is found, the different materials are prepared separately with note made of the relative spatial relationships of the layers. Soil and rock also are commonly inhomogeneous.


Other factors to be noted include moisture content and hardness of the material. Samples which are moist must be dried prior to analysis. Wet fibers will stick together in clumps and inhibit a homogeneous distribution of the material.

#### **D. Sample Preparation**

##### **1. General Cautionary Guidelines**

Sample preparation should be carried out within general guidelines with the understanding that each analyst will develop working habits which are suited to him/her. The general guidelines should ensure that the individual's habits promote good



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workmanship and not detract from the work quality. Any sample preparation style should conform to the following basic rules:

- a. All samples shall be opened within the confines of a filtered ventilation system.
- b. All spills shall be cleaned immediately and the work area cleaned periodically, regardless of the absence of visible debris.
- c. The slides, coverslips and refractive index liquids shall be placed under the fume hood and covered at all times when not in use.
- d. All solutions used for cleaning and analysis should be covered and clearly labeled.
- e. A sealable bag should be used for all debris generated during preparation. This bag should be considered as hazardous waste and should be handled and disposed of as such. The debris bag should remain in hood prior to disposal.
- f. All preparation tools should be kept exceptionally clean. Tools should be wiped thoroughly between sample preparations. Due to constant cleaning of the tools, be aware of cellulose contamination in the samples, especially when using the mortar and pestle.
- g. Refractive index liquids are toxic and should be used with extreme caution. Any spills should be cleaned immediately. All trash generated should be placed in the fume hood debris bag. Skin contamination from the liquids should be cleansed thoroughly. The laboratory equipment, such as the microscopes, tools, telephone and doorknobs, should not be contaminated with soiled hands. The work area should not smell of refractive index liquids. Disposable gloves should be available to the analyst. The index liquid dispenser should be kept clean and free of residue.

## 2. Sample Preparation Procedures

With application of the above guidelines, a basic outline for sample preparation is as follows:

- a. After a thorough gross examination of the material (see III.C.) select several homogeneous tweezerfuls of the sample and place a few drops of the desired refractive index liquid on a microscope slide labeled with the laboratory sample ID# and, if necessary, the refractive index of the mounting oil. For non-homogeneous samples a variety of steps may need to be taken to obtain a homogeneous sample to obtain an accurate result:
  - i. Mix, crush and/or grind in a mortar and pestle.



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- ii. For layered or grossly in-homogeneous materials, separate layers and analyze each unique material type. Report results for each layer separately, and a composite for the entire material, if client requests it.
- iii. For irregularly inhomogeneous materials, make multiple preps of the various different materials and report a composite the result.
- b. Cut or mash the sample, blending the material evenly throughout the liquid. The material should be distributed so that the liquid is not clouded. Light should be capable of passing through all sections of the liquid equally.
- c. For non-friable materials such as floor tiles, roofing tars, and other such materials, standard techniques of chopping, cutting or grinding may not work.
  - i. For floor tiles, use a sharp scalpel blade to slice very fine shavings of material off of the floor tile. When placed in 1.550 high dispersion oil, the oil acts as a solvent for the vinyl in most instances and after repeated chopping of the material on the slide, asbestos fibers, if present, can be observed quite well.
  - ii. For tarry roofing materials, chop off a small piece of the material and place it into 1.55 high dispersion oil, which usually acts as a solvent for the tar. Chop the material up as much as possible. Often the tar forms a gooey mass, and it is necessary to put the slide aside for several minutes while the solvent action of the oil works on the tar. (Be careful in analyzing fibers found in the sample as the dissolved tar can change the refractive index of the oil).
  - iii. Other materials - Sometimes placing samples in a flame from an alcohol lamp or a cigarette lighter works well as a test by burning off coatings and binders. Use a small 1 cm X 1 cm brass screen to support the sample in the flame so the residue does not disintegrate onto the prep bench. Be careful in analyzing the residue as it is often soot covered and the refractive index of any contained asbestos may have been altered by the high temperature associated with the burning process.
- d. Place a coverslip over the sample. Remove the air pockets with applied pressure to the coverslip. The refractive index liquid and the sample should be evenly distributed to the edges of the coverslip.

The analysis of the material on the slide is a representation of the entire homogeneous portion of the material. The relationship between the prepared slide and the submitted sample is known only by the microscopist and should be kept clearly in mind during the analysis. Each microscopist should prepare the slides which he/she intends to analyze. The amount of samples prepared will not exceed the amount in which the analyst can clearly remember, i.e. No more than five at a time.

There are many types of materials submitted for asbestos analysis. Some of these materials may require different types of preparation. New analysts should be taught the



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different types of preparation by a qualified PLM microscopist, and with experience should become proficient in a short period of time.

#### **3. Special Prep Procedures for Rock & Soil Samples**

There are some sample types which demand special attention during preparation. In the case of soil or rock samples, the material is rarely homogeneously distributed. Extra care should be taken in selecting a combination of all the materials present.

- a. A small portion of material should be taken from the various size ranges present, i.e. clay, silt, sand, pebbles, cobbles, etc.
- b. The soil samples should always be dried even when moisture is not visible.
- c. The material selected from step #1 above should be ground with a mortar and pestle until thoroughly mixed and pulverized.
- d. The slide should be prepared as previously specified in this section.

#### **4. Special Prep Procedures for Sediment Samples**

In the case of sediment samples, distinguishing separate materials is usually difficult. The preparation should go along these lines:

- a. Core into the center of the container of sediment material. Extract a core sample representing the layers of sediment.
- b. Dry the sample completely.
- c. Mix the sample thoroughly. Pulverize the sample using the mortar and pestle. Be aware that sludge samples may contain other hazardous wastes.
- d. Prepare the slide as previously specified in this section.

#### **E. Fiber Identification**

Fiber identification will be performed utilizing all of the available optical properties. As described in the "Interim Method for the Determination of Asbestos in Bulk Insulation Samples", all materials identified as an asbestiform mineral will be distinguished by the following optical properties (See V. below for detailed description of these properties and how they are measured):

1. Morphology
2. Color
3. Pleochroism
4. Refractive index parallel and perpendicular to the fiber elongation direction
5. Birefringence & interference colors
6. Extinction characteristics
7. Sign of elongation
8. Other properties used to characterize a material



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For the purpose of identifying non-asbestos fibrous materials, at least one optical property will be identified and recorded that serves to distinguish them from asbestos.

Personnel utilized for training in asbestos microscopy, must have Bachelor's or higher degree in the geological sciences. All data collected by the analyst will be entered onto the designated PLM Data Sheet (See Appendix A).

#### **F. Quantification of Sample Contents**

The quantification of asbestos in bulk material samples has been the subject of much debate. The EPA stated in the Federal Register on May 27, 1982 in the "Interim Method for the Determination of Asbestos in Bulk Insulation Samples" found in Appendix A to subpart F of the rule, section 1.7.2.4, page 23382, "Quantitation of Asbestos Content" that "Asbestos quantitation is performed by a point-counting procedure." Also in the first paragraph of section 1.7.2.4 is the following: "Point counting provides a determination of the area per asbestos. Reliable conversion of area percent to percent dry weight is not currently feasible unless the specific gravity's and relative volumes of the material are known". Published in the Federal Register on Wednesday, September 1, 1982, is the notice "Asbestos; Friable Asbestos-containing Material in Schools: Identification and Notification; Correction". On page 38536 of the publication is the following correction, "Paragraph 1.7.2.4 of Appendix A of the rule was intended to provide for a point counting procedure or an equivalent estimation method for determining the amount of asbestos in bulk samples. The phrase 'or an equivalent estimation method' was inadvertently omitted from the first sentence of paragraph 1.7.2.4 of the Appendix".

It is the decision of Asbestos TEM Laboratories, Inc. to utilize a "calibrated analyst" estimation method equivalent to the point counting method unless the client specifically requests a point count analysis. In both instances, the area percent asbestos will be reported.

##### **1. Area Estimation Quantification Method**

Reference diagrams are used as standard practice for the quantification of the percent composition of minerals in a thin section during routine petrographic analysis (e.g., Kerr 1977). The same diagrams are applied for the quantification of minerals and other materials during the analysis of bulk material by the oil immersion method. The microscope provides a two-dimensional view of the sample, so that the estimated percent represents an area percentage. The oil immersion method allows for empty space between particles, which is not counted in the estimation of percentages. Practice and experience develop the skills required to determine a realistic visual estimation of the area percent value for all components of the sample.

The area percent value is an estimated value, not an exact percentage. All results will be listed in a range between the lowest and the highest possible values present as judged by the analyst. The upper detection limit for the asbestos content is 95% to 100%. The lower detection limit for the asbestos content is "less than 1%, none detected". The



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possible ranges available within the scope of the procedure are: <1%, None Detected; 1-5%; 5-10%; 10-20%; 20-30%; 30-40%; 40-50%; 50-60%; 60-70%; 70-80%; 80-90%; 90-95%; 95-100%. However, these ranges do not apply to the Non-Fibrous Material category. The ranges given in the results column for this category are simply the remaining percentages of material needed to make the data sum to 100%.

#### **2. Point Count Method**

A 25 point Chalkley ocular reticle is used to visually superimpose semi-randomly scattered points onto the microscope field of view. Using a standard two key laboratory counter, record the number of points positioned directly above each of particle or fiber of interest. Score only points directly over asbestos fibers or non-asbestos 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. For the purposes of this method, "asbestos fibers" are defined as having an aspect ratio of greater than 3:1 and being positively identified as one of the six regulated asbestos minerals described in greater detail in Section VII below.

A total of 400 points superimposed on either asbestos fibers or non-asbestos matrix material must be counted over at least eight different preparations of representative sub-samples. Take eight fine-pointed forceps samples and mount each separately with the appropriate refractive index liquid. The sample should be well dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 non-empty points on each preparation, using a reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

Quantitation should be performed at the lowest magnification of the polarized light microscope which can effectively distinguish the sample components (typically 100X). The percentage asbestos is calculated as follows:

$$\% \text{ asbestos} = A / N$$

where A = number of asbestos counts


where N = total number of non empty points counted (400).

If A=0, report "No Asbestos Detected"

If  $0 < A \leq 3$ , report <1% asbestos.

The value reported should be rounded to the nearest percent.

It has been the decision of Asbestos TEM Laboratories, Inc. that all analysts will be trained to recognize and identify common building materials, in addition to the six asbestos minerals. The reporting of the data will include three categories: "Asbestos Fibers", "Non Asbestos Fibers", and "Non Fibrous Material." As a sub-category under Non Fibrous Material the term "miscellaneous particles" is used. Miscellaneous particles refers to any non fibrous material which the analyst does not have the time, knowledge or magnification power available with

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which to base a decision. Any material that would qualify as miscellaneous particles would be of a non fibrous nature and therefore not relevant to the objective of the analyses.

## Report Generation & Data Review

Upon completion of the analysis, the analyst is required to review his material closely for errors, and then enter data to the laboratory database system. Any deviations from the standard method are described in a statement to be added to the cover letter.

Data to be entered into all PLM reports includes, at a minimum, the following:

1. Client name and contact address
2. Date samples submitted
3. Date report completed
4. Total samples submitted
5. Total samples analyzed
6. Client sample ID number for each sample
7. Lab ID number for each sample
8. Percentage and name of each asbestos type found in each sample
9. Percentage of all non-asbestos fibrous material
10. Name of the two major non-asbestos fibrous materials
11. Types of non-fibrous materials present
12. Description &/or location of sample material (if given by client)
13. Sample color
14. Notation is made if sample is inhomogeneous and if it was split.

If a heterogeneous sample was split and contained homogeneous materials analyzed separately, the analytical quantification data are recombined in proportion to the amount present in the original bulk material with a single result reported to the client. Note is also made in the report stating that the sample was split. However, the client may specify that sample splits are to be reported separately.

When a draft of the report is complete, the analyst reviews the preliminary report and if no errors are found by the analyst, the preliminary report is faxed or verbally reported to the client according to the clients' request on their COC form. The preliminary report is reviewed by a laboratory QC reviewer and if no errors are found, the final report is sent to the client by mail.

Several notations are included in the cover letter to accompany each report given to the client. These include the following statements:

- 1) "This report must not be used by the client to claim product endorsement by the NVLAP or any other government agency of the U.S. government."



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- 2) "The data in this report applies only to the samples tested."
- 3) "The PLM analytical method for which the laboratory is accredited does not extend to non-friable materials."

### **Common Diagnostic Optical Tests and Related Principles Regarding PLM Analysis for Asbestos**

#### **A. Preamble**

The following is an excerpt from "Elements of Mineralogy" by Mason and Berry (1968): "Determinative mineralogy can be defined as the science (and art) of identifying a mineral from its physical and chemical properties. The recognition of an unknown mineral may be instantaneous or may require careful and time consuming tests, depending upon the identity of the mineral, the quality of the specimen, and the knowledge, experience, and skill of the observer. Many schemes of mineral identification have been devised, but it should be emphasized that any scheme is valuable only if it is applied with experience and common sense. ....Logical schemes of mineral identification are useful guides, but experience and intelligence will often suggest a short cut to the procedure. Even if a rapid examination does not serve to identify the specimen, it should limit the possibilities to comparatively few minerals, and the next step is to select the most suitable diagnostic test...."

In accordance with section 2.7.2.3, Fiber Identification, from the "Interim Method for the Determination of Asbestos Minerals in Bulk Insulation Samples" the positive identification of asbestos requires the determination of the following optical properties:

1. Morphology
2. Color
3. Pleochroism
4. Refractive indices
5. Birefringence & Interference Colors
6. Extinction characteristics
7. Sign of elongation
8. Other optical qualities of minerals

The following text is an introduction to the optical properties listed above. The purpose of Section Three is to familiarize the reader with the optical properties which are most significant to the identification of the asbestos minerals. The material in Section Three is an overview of the most important optical properties of minerals and is not meant to be a substitution for formal training in the principles of optical mineralogy.





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#### **B. Introduction**

Minerals, by definition, are naturally occurring crystalline materials found in or upon the earth. Asbestos is a family of minerals (Chrysotile serpentine and a variety of amphiboles) with specific characteristics, including a high degree of fibrosity, tensile strength, chemical inertness, and the ability to withstand extreme temperatures. Therefore, to understand and identify the various types of asbestos, one must turn to the study of mineralogy and crystallography, and the various techniques of analysis employed by such fields. A summary of this information as it applies to asbestos is presented in this and the following sections.

#### **C. Crystal Morphology**

The characterization of the morphology exhibited by crystalline materials is the study of their shape, structure and form. There are thousands of crystals, both natural, man-made, and extra-terrestrial, each identified by distinct morphological characteristics. The basic property characteristic to all crystals is symmetry and the characteristic feature of symmetry is repetition. A crystal may show repetition with respect to a point in which case it has a center of symmetry, and with respect to a line in which case it has an axis of symmetry, and with respect to a plane in which case it has a plane of symmetry. Every crystal is characterized by a specific combination of symmetry elements.

##### **1. Crystal Shape and Mineralogical Structure**

Based on studies of the external form of crystals and the angular relationships between crystal faces, mathematical reasoning has been used to establish thirty-two different classes of crystals. All crystals are characterized by a 3-dimensional periodically repeating array of the atoms within the crystal structure giving rise to specific combinations of symmetry elements. These elements, and the stacking arrangements of the atoms in the crystals, give rise to what are called crystal classes. Thirty-two classes of crystals are seen to exist in nature which have been further grouped into seven crystal systems listed below:

- a. Triclinic
- b. Monoclinic
- c. Orthorhombic
- d. Trigonal
- e. Hexagonal
- f. Tetragonal
- g. Isometric

Asbestos minerals, in particular, belong to either the orthorhombic or monoclinic crystal classification systems giving rise to a variety of mineral characteristics described below. Other asbestos look-alike minerals & materials may belong to any of the seven crystal





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systems. (For a complete understanding of crystal morphology, instruction in Crystallography and Mineralogy is strongly recommended)

## **2. Crystal Form**

"When a mineral occurs in crystals, their form may be sufficient to identify the mineral without any further tests" (Mason and Berry, 1968).

The shape into which the crystal characteristically grows and envelops is known as the "form" or "habit" of the crystal. In the case of the asbestos minerals, the distinctive form the crystals take on is termed "fibrous" or "asbestiform". Fibrous/asbestiform crystals are characterized by parallel to subparallel sides and a high aspect (length/width) ratio. The EPA Interim Method (1981) defines a material to as having a fibrous or asbestiform habit when it has an aspect ratio of 3:1 or greater. However, just because a particle is observed to have a fibrous or asbestiform habit does not mean that it is asbestos. Numerous minerals and materials exhibit such habits and are not considered to be hazardous asbestos.

Fortunately, there are differences in form, habit, and other identifying characteristics between the six asbestos minerals, and between the non-asbestos fibrous materials which, in most cases, allow them to be differentiated quite easily. For instance, in asbestiform material viewed in plane polarized light will become virtually invisible in a refractive index liquid which matches the index of refraction of the mineral in question. Under the same conditions, while using crossed polarizers, the form of the mineral is often obscured and distorted, especially when the interference colors of the mineral are low.

When performing a mineralogical analysis for asbestos, there are two obvious analytical procedures which can be utilized. One method emphasizes the use of the form or habit of the material as a means of identification, with one specific commonly indicative refractive index liquid used for all analyses. (This method commonly requires only one sample prep). The other method excludes form as an indication of the presence of an asbestos mineral, and focuses upon pinning down the refractive indices of the material alone. (This method commonly requires the use of several refractive index liquids and multiple sample preps). As asbestos minerals commonly have very distinctive forms, the first method is often completely adequate. However, on occasion it is necessary to use the latter method. The significance of form to the analyst will generally determine the mode of analysis.

## **D. Color and Pleochroism**

Color, when present, is a distinctive feature. The presence of color in a material is due to preferential absorption and/or transmission of certain wavelengths of light. For instance, deep blue minerals preferentially absorb red & yellow wavelengths of light, and transmit only the blue. Materials that are deeply colored during gross examination, are commonly



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colored when prepared and analyzed under the PLM microscope in plane polarized light. More lightly colored appearing materials under gross analysis, however, commonly show no color at all under PLM analysis.

When viewing a material under plane polarized light, most materials yield no color change as the mineral is rotated. Some anisotropic minerals, however, may exhibit a change in colors to varying degrees as the stage is rotated. The change produced is known as pleochroism and is due to preferential absorption or transmission of certain wavelengths of light in a certain specific orientations of the mineral.

The variety of pleochroism which an anisotropic mineral exhibits is a function of the symmetry system to which it belongs. Hexagonal or tetragonal colored minerals are dichroic - i.e., the pleochroic coloring of minerals in these two crystal systems, as exhibited with the polarizer, is twofold. In pleochroic uniaxial minerals, light vibrating parallel to the optic axis is one color, whereas light at right angles is another. Orthorhombic, monoclinic, and triclinic minerals, when colored in thin section, exhibit three different colors and are termed trichroic. However, for any given single crystal orientation, only two pleochroic colors will be observed which may be a combination of the three trichroic colors.

#### **E. Refractive Index**

A ray of light passing through a transparent substance will be broken into two separate rays upon contact. The portion of light which is reflected back into the air is called the reflected ray. The portion of light which enters the substance is called the refracted ray. The original ray of light, prior to contact with the substance, is known as the incident ray.

##### **1. Snell's Law**

The relationship between the incident and refracted rays is the ratio of the sine of the angle of incidence to the sine of the angle of refraction and is a constant (Snell's Law). The constant is known as the index of refraction. The index of refraction can also be stated as the ratio of the velocity of light in air to the velocity of light in a solid.

Every transparent substance has an index of refraction, or a range of indices, which can be used to identify the material. In terms of minerals, the crystalline structure is closely related to the optical properties. Amorphous and isometric minerals pass light equally in all directions, therefore having one index of refraction. The other crystal systems are more complex and the refracted ray is split into two rays which are at right angles to one another (generally) and are moving at different velocities. Due to the difference in velocities there will be two different indices of refraction.

Optical properties, and especially the refractive indices, are among the most valuable determinative properties for mineral identification. From refractive indices and other



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optical properties, an unknown mineral generally can be easily identified by referring to tables of the properties (as given in VII & VIII below).

#### **2. Measuring Refractive Indices**

The measurement of the refractive indices of a transparent solid is made by immersing the substance in question into a liquid of known refractive index. Using a polarizing microscope with a parallel light source, a comparison can be made between the refractive index of the solid and the liquid. Two methods for determining the refractive index of asbestos and other materials are given below. They are 1) the Dispersion Staining method using a specialized “Central Stop” objective lens and, 2) the Becke Line technique. The Dispersion Staining method is the recommended procedure for refractive index determination of asbestos minerals at Asbestos TEM Laboratories. Both methods, however, are very useful and by being able to use both, the trained analyst is able to determine the refractive index of asbestos and other materials with a high degree of accuracy.

#### **3. The Dispersion Staining Test**

The Dispersion Staining test is very useful in determining the refractive index of any transparent material for which dispersion staining charts are available. Fortunately, the asbestos minerals and other fibrous building materials have been thoroughly studied by dispersion staining researchers (Brown, McCrone, Su) and a large amount of easy to use data is available. Unlike the Becke Line Method, accurate Dispersion Staining refractive index analysis requires a high degree of initial knowledge concerning the material being analyzed. In most cases asbestos minerals can be tentatively identified based on other characteristics (i.e. morphology, color, pleochroism, extinction). With this prior knowledge, by using just one immersion oil recommended for the suspected material (See Su, Table 1 - Appendix C), Dispersion Staining is able to accurately determine the refractive index of the material over its entire range of refractive indices, if the material is what it was suspected to be. If the material is not the suspected material, then the determined refractive indices will be wrong. Knowledge of the range of refractive indices allowed for a given asbestos type is important to guard against this error. Assuming the initial guess was accurate, the Dispersion Staining technique is much more efficient than the Becke Line Method in determining refractive index with the latter sometimes requiring many successive mounts in a series of immersion oils to obtain an accurate reading.

(The following is summarized from Brown, Kenneth et al., 1963 - See McCrone Dispersion Staining Reference Manual). To analyze a particle using dispersion staining, the particle must first be immersed in a liquid of known refractive index. The liquid is selected so that at some visible wavelength  $\lambda_0$ , the refractive indices of the particle and the immersion liquid are identical. At all other wavelengths in the visible spectrum, however, the refractive indices differ. As a result, at all wavelengths except  $\lambda_0$  the edges



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of the particle act as prisms, producing a spectrum. This spectrum is displayed at the back focal plane of the microscope objective. Here, the central stop of the dispersion staining objective is applied.

The central stop of the dispersion staining objective blocks out axial light which has been transmitted without deviation throughout the particle. If it has been transmitted without deviation, the axial light must be of wavelength  $\lambda_0$ . Light of all other wavelengths is deviated. The axial light, therefore, has the color corresponding to  $\lambda_0$ . The non-axial light or annular light seen when the central stop is in place, has a color caused by a deficiency of  $\lambda_0$  light; that is, its color complements the axial color. Most of the transmitted light is axial and is blocked by the central stop. Therefore, the central stop produces dark field illumination. Against the darkfield, particles show the color complimentary to  $\lambda_0$ . By comparison with special color charts (Su, Table 2- Appendix C) the color of the observed complementary light can be used to accurately assess  $\lambda_0$ . Knowing the mineral or material being observed, the refractive index of the immersion oil, and the temperature of the immersion oil, the refractive index of the material can be determined by consulting a series of other charts (Su, Tables 4 thru 23 - See Appendix C).

#### Detailed Dispersion Staining Procedure (Summarized from Su, 1994 - Appendix C)

- a. Select a proper immersion oil using your preliminary information as to asbestos fiber type. (Su, Table 1 - Appendix C). Use high dispersion Cargille Series E oil if possible.
  - 1.550 - Chrysotile
  - 1.620 - Tremolite, Actinolite, Anthophyllite
  - 1.700 - Crocidolite, Amosite

Be careful to watch for surface coatings on the observed particles. Coatings will drastically altered the dispersion staining colors observed and the resulting refractive index determinations. Chopping or grinding asbestos fiber bundles to break through the coating material and reveal interior uncoated fibers is often helpful in allowing observation of clean fibers. If this fails to work (i.e. on the basis of morphology and other characteristics you believe that asbestos is present, but anomalous dispersion staining colors are present, resulting in accurate refractive index results. In

- b. Measure the temperature of the dispersion oil.

Measure and record the temperature of the dispersion oil on the microscope slide. To simplify, assume that the oil and the room are in thermal equilibrium and measure the ambient room temperature. This data is needed for making temperature correction.
- c. Be sure the microscope is aligned as described in the QA Manual.



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- d. Observe the Dispersion Staining color for the  $\alpha$  direction of the asbestos fibers.  
With the polarizer oriented E-W, orient the asbestos fibers so that they are  $\perp$  to the E-W direction. Be careful to check crocidolite fibers as they may be length fast and hence will require re-orienting the fiber  $\parallel$  to the E-W direction to obtain the correct dispersion staining color for determination of the refractive index. Although the  $\alpha$  direction of the monoclinic amphiboles (tremolite, actinolite) is not exactly  $\perp$  to the fiber elongation, for standard day-to-day asbestos analysis, the refractive index at this orientation can be assumed to be reasonably close to  $\alpha$ . If more precise refractive index determination is required, the substage iris can be opened, the polarizer inserted, and the fiber in question to determine its closest extinction orientation. Returning to the dispersion staining mode the fiber can now be exactly analyzed for  $\alpha$ .
- e. Convert the observed dispersion staining color into the corresponding matching wavelength,  $\lambda_o$  between the asbestos fiber and the immersion oil used.  
Refer to the “Dispersion Staining Colors” Chart (Su, Table 2 - Appendix C).
- f. Determine the refractive index for  $\alpha$  corresponding to the observed  $\lambda_o$  and temperature,  $t$ . Refer to the Dispersion Staining Refractive Index Tables (Su, Tables 4 thru 23 - Appendix C).
- g. Observe the Dispersion Staining color associated with  $\gamma$  of the asbestos fibers.  
Rotate the stage  $90^\circ$  and then repeat steps 4 - 6 above to determine  $\gamma$ .

In all, the Dispersion Staining method is an excellent, fast and efficient way to measure the refractive index of asbestos fibers.

#### 4. The Becke Line Test

The Becke Line Test is another technique used to determine the refractive index of a substance. In this method, an immersion oil of known refractive index is determined to be of higher, lower or the same refractive index than that of any transparent material or mineral. While it is more widely applicable to unknown materials than Dispersion Staining, it is much slower and less efficient in determining the refractive index of the asbestos minerals which are generally easily identified, without the knowledge of the refractive index. With experience, the amount the refractive indices of the material are higher or lower than the liquid can be closely estimated, although over a much narrower range than the dispersion staining method allows.

There is a fine line of light which surrounds each particle submerged in the refractive index liquid. The line of light is most easily visible with the substage diaphragm partly closed and under a moderate to high magnification. The migration of the Becke Line, as the objective is raised slowly, will indicate the material with the higher index of



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refraction. That is to say, the Becke Line will migrate towards the higher refractive index. The amount of migration and the intensity of the Becke Line indicate the degree of difference between the refractive indices of the solid and the liquid. A Becke Line which is bright and well defined, and migrates long distances while retaining clarity, indicates that the indices of the materials are different by a large margin. A pale Becke Line with weakly defined edges which moves only slightly, when the objective is raised, indicates that the refractive indices of the two materials are close.

When the refractive index of a material is equal or very close to the refractive index of the liquid, a phenomenon known as dispersion is commonly observed which is helpful in precisely identifying the refractive index of the solid. Dispersion occurs when the refractive index of the material is different for different wavelengths of light. This results in the Becke line being split into a rainbow-like band of colors at the edge of the crystal when the refractive index of the oil is very close or equal to that of the crystal. Using the correlation of the Becke line colors & their relative movements to the refractive index difference between the grain & the oil (F.D.Bloss, 1961, p.59) the following approximate refractive indices for the grain can be deduced (for sodium light, microscope racking upward\stage downward). (See the table below). For example, if the grain is immersed in an oil of index 1.550, the following colors of two Becke lines moving toward the grain & the oil, respectively, will be observed: whitish-yellow moving in & dark blue-violet moving out, indicating that the grain's index exceeds the oil's by 0.007 to 0.014 & is equal approximately  $1.561 \pm 0.004$ ; lemon-yellow & violet-blue, indicating that the grain's index exceeds the oil's by 0.001 to 0.007 & is approximate to  $1.554 \pm 0.003$ ; orange-yellow & sky-blue, confirming match of their indices, that is  $1.550 \pm 0.001$ ; reddish-orange & whitish-blue, indicating that the grain's index is lower than oil's by 0.001 to 0.003 & is approximate to  $1.548 \pm 0.002$ ; dark reddish-brown & blue-white, indicating that the grain's index is lower than oil's by 0.003 to 0.005 & is equal approximately  $1.546 \pm 0.004$ . In the case an increment for the refractive index is higher than  $\pm 0.004$  (these numbers in the table below are shaded) use other oils.





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Color of Becke Line	Refractive indices of asbestos in oil.			
grain\liquid	1.55	1.604	1.64	1.68
White\				
White	>1.564	>1.625	>1.666	>1.714
Whitish-yellow\				
Dark blue violet	1.561	1.62	1.66	1.707
Lemon-yellow\				
Violet-blue	1.554	1.609	1.646	1.687
Orange-yellow\				
Sky blue	1.55	1.606	1.642	1.682
Reddish-orange\				
Whitish-blue	1.548	1.602	1.638	1.676
Dark reddish-brown\				
Blue-white	1.546	1.599	1.634	1.672
White\				
White	<1.546	<1.598	<1.633	<1.672



--- For shaded regions, Becke Line colors do not predict refractive indices of tested material to  $\pm 0.004$ . Therefore, use a different R.I. oil if these colors are observed or use the Dispersion Staining Method with the recommended R.I. oil for the asbestos type observed.

### 5. Refractive Index Measurement of Asbestos in Difficult Samples

Sometimes, problems arise in the measurement of refractive index due to the presence of surface coatings that cover and alter the apparent refractive index of asbestos fibers. Materials such as joint compounds and muds, fiberglass ceiling tiles, and a variety of others, can exhibit these characteristics. Several alternate procedures, in order of increasing analytical effort are listed below:

- Spend extra time chopping or grinding your sample materials to break up asbestos fiber bundles to expose uncoated fibers in the interior of coated fiber bundles. Sometimes a significant amount of time must be spent looking for that one uncoated asbestos fiber from which you can measure accurate refractive indices (for single fibrils, the Becke line method has been found to work better in determining refractive index as compared to the dispersion staining method which works well with bundles). If, or when you find a fiber you can accurately measure, it is necessary to extrapolate your positive finding to the other coated fibrous materials for which refractive index cannot be accurately determined, but which you are confident are the same as the fiber which you obtained a positive result. This is the type of material that requires experience to accurately



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examine, and newly trained analysts should confer with an experienced analyst before releasing final results.

- b) If the above technique fails to produce acceptable results but all the other data indicate asbestos, the analyst shall recommend to the client that a modified gravimetric ashing and acid washing preparation procedure be performed using either PLM and/or TEM to analyze the residue for asbestos. If the residue gives good PLM refractive indices, PLM is sufficient. However, it is not uncommon that problems remain and that TEM analysis is required (See TEM Bulk Asbestos Manual).

#### **F. Birefringence & Interference Colors**

The velocity of light passing through an amorphous material or a material with an isometric crystal structure will be the same in all directions. Such substances are termed "optically isotropic" and do not display the property of birefringence.

All other substances in which the velocity of light differs with the direction in which it is passing through the material are termed "optically anisotropic". A ray of light will be split into two rays, moving at different rates of speed and at right angles (generally) to one another, when passed through an anisotropic substance. Due to the difference in velocity, the two directions will exhibit different indices of refraction. The difference in these refractive indices is the birefringence. The visible manifestation of birefringence is revealed by the colors, called interference colors, which a crystal appears to exhibit when viewed under crossed polarizers. These colors range from a very weak gray (Called first order gray) to a pearly white (High order white in the fourth or fifth order color range). If one knows the thickness of the material, the value of the birefringence can be obtained from reference to a Michel-Levy Interference Color Chart. Commonly, for asbestos and other minerals, there are characteristic interference colors as described in VII. & VIII. below.

#### **G. Extinction**

Isotropic minerals and materials, when viewed under crossed polarizers, become completely invisible because they do not preferentially retard or transmit light in any specific direction. Anisotropic minerals, which do preferentially retard and transmit light in different directions, appear to wink in and out as the stage is rotated. An anisotropic material which becomes darkened during rotation under crossed polarizers is said to exhibit the property of extinction. The vibration directions of the two rays are parallel to the vibration directions of the optical components of the microscope when the particle becomes dark under crossed polarizers (polars). An accessory plate is used to determine the fast and slow ray. Turn the sample from the point of extinction to the point of the maximum interference colors and insert the compensator plate. If the order of color increases, the parallel ray is the slow vibrating direction of the mineral, while decrease would represent the fast ray. Note that the slow ray direction on the compensator plate is fixed.





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The term addition refers to the alignment of the slow ray of the plate and the slow ray of the mineral, producing higher color. The term subtraction refers to the alignment of the slow ray of the plate and the fast ray of the mineral, producing a decrease in the order of color. Note that the index of refraction of the slow ray is always higher than the index of refraction of the fast ray.

#### **1. Parallel Extinction**

Parallel extinction occurs when the mineral becomes darkened parallel to the directions of the optical components of the microscope. The components, the analyzer and the polarizer, are aligned in the north-south and east-west directions. Note that the cross-hairs are also oriented parallel to the optical components.

#### **2. Inclined or Oblique Extinction**

When the mineral becomes darkened (extinct), at an angle which is inclined, or oblique, to the optical components of the microscope, the mineral is said to exhibit inclined or oblique extinction. In such a case, it is desirable to determine the angle of extinction. The extinction angle is usually measured from the orientation of the slow ray. The stage is rotated to extinction from a position parallel to the vibration plane of the polars.

The value is calculated by degrees, read from the graduated stage of the microscope. a series of readings should be taken on several crystals to obtain an average value. The values of the extinction for minerals can be obtained from an optical mineralogy text.

#### **3. Symmetrical Extinction**

Many crystals exhibit cleavage or rhombic cross sections, giving a striated or banded appearance to the specimen. During symmetrical extinction every other "band" in the crystal becomes extinct simultaneously. During continuous rotation of the stage these bands pass out of extinction as their counterparts enter extinction.

#### **4. Mottled Extinction**

Materials such as micas, strained quartz, cellulose, and various synthetics often exhibit a characteristic called mottled extinction. Mottled extinction is the property of a material which, when at its greatest extinction position under the polarizers, is irregularly and incompletely extinct. This is different than undulatory extinction which sweeps across a material as it is rotated under crossed polarizers under the microscope. It is one primary observation which can differentiate cellulose from chrysotile which has straight extinction.

## **H. SIGN OF ELONGATION**



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A beam of light passed through an anisotropic material will be split into two rays of light. When the anisotropic material is in an elongated form, as are the asbestos minerals, then the speed of the two rays of light relative to each other and the direction each ray is passing through the elongated crystal can be determined. These properties are summarized as being a positive or a negative sign of elongation. In the case of positive elongation, the slow ray is vibrating parallel to the length of the crystal. In the case of negative elongation, the fast ray is vibrating parallel to the length of the crystal. The terms length-slow and length-fast are commonly used.

To determine the sign of elongation, a 550nm first order red retardation plate is inserted into the microscope accessory port while viewing the crystals under crossed polars. The length-slow, or positively elongated, fibers will be blue when oriented on a diagonal from the 2 o'clock position to the 8 o'clock position. The length-fast, or negatively elongated, fibers will be yellow in a 2 o'clock to 8 o'clock position.

Typically the sign of elongation is quickly and easily identified for the asbestos minerals. However, this is not always true for asbestos look-alike minerals. One mineral in particular, wollastonite, has the characteristic of exhibiting both length fast and length slow behavior. One method of testing a suspect material is to look around to see if there are fibers of similar morphology and refractive index with some exhibiting length slow and others length fast behavior. Another is to tap the coverslip while looking at the sample under the microscope in such a manner that the fibers roll onto a different cleavage face to see if they change their sign of elongation.

#### **I. OPTIC SIGN; INTERFERENCE COLORS; 2V ANGLES**

The optic sign and 2V interference figure value are two other commonly used indicator qualities of minerals. These qualities, and numbers quantifying their value are given in VII. & VIII. below, but will not be described here as they are outside the scope of this method and of little utility for asbestos identification. The information was included for the use of those with the proper background to completely understand the principles of optical mineralogy. In the opinion of the author, geologists are the scientists most highly qualified to conduct polarized light analyses for asbestos.

This section has summarized a few basic principles of optical mineralogy and applied these principles specifically to asbestos analysis. The actual complexity of mineralogical analysis can be studied at a senior college offering a four year program of study in the geological sciences.



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### **Common Diagnostic Physical and Chemical Tests Used as an Aid in PLM Analysis For Asbestos**

Several common physical and chemical tests that can be of aid in identifying asbestos from non-asbestos look-alike minerals, or which can be used to remove interfering particulate are described below.

#### **A. Subjecting Sample Material to Intense Heat**

Subjecting sample material to intense heat or burning is often helpful in removing substances such as fibrous polyethylene. Asbestos minerals are unaffected during heating while low melting temperature and combustible materials are removed or altered sufficiently to make identification of asbestos minerals easy. A flame source such as a cigarette lighter or alcohol vapor lamp may be sufficient. However, in many cases, the use of a muffle furnace is preferable due to the minimization of soot left on the material after heating.

#### **B. Subjecting Sample Material to Caustic Reagents & Solvents**

Subjecting sample materials to caustic reagents (i.e. dilute acids such as HCl) and is often helpful in identifying carbonate-containing materials through the observation of vigorous fizzing. It is also helpful in while removing fine obscuring carbonate particulate, and as a test for fibrous aragonite needles, which are also dissolved.

Subjecting sample materials such as floor tiles and rubbery compounds to solvents (i.e. methylene chloride) can also be useful in removing interfering materials and allowing easier visualization of asbestos materials.



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#### Characteristic Optical Properties of the Six Asbestos Minerals

The following information is taken largely from the EPA Interim Method (1981) and from "An Introduction to the Rock Forming Minerals" Deer, Howie & Zussman (1986)

##### A) Sheet Silicates Group

###### CHRYSOTILE (Serpentine)

Refractive Indices:  $\alpha = 1.493 - 1.560$

$\beta = \text{-----}$

$\gamma = 1.517 - 1.562$

Crystal System: Monoclinic

Birefringence: Moderate, 0.013 to 0.017

Color: Colorless to pale green with increasing thickness

Pleochroism: None

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-)

Chemistry:  $\text{Mg}_3[\text{Si}_2\text{O}_5](\text{OH})_4$

Recommended D.S. R.I. Liquid: Series E (High Dispersion) - 1.550

Chrysotile is the most common type of asbestos used in building materials. Chrysotile popularity comes from its many uses, due to the fibers unique properties. The most obvious characteristic is its ability to withstand extreme temperatures, making it a very beneficial element in fire-proofing. Chrysotile is also used as a binder, for it has great tensile strength. Strength and flexibility make the mineral excellent for weaving into a fireproof cloth. Chrysotile is also chemically resistant and can be used to make



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laboratory bench tops that prevent chemical stains. Chrysotile has been referred to as the "Wonder Fiber", for its almost unlimited industrial uses.

Chrysotile is the fibrous form of serpentine, a magnesium-rich mineral typically found in metamorphic rocks. Chrysotile is unique in that fibers are formed by sheetlike crystals that roll into tight tubes. There are several forms which chrysotile fibers may exhibit. Generally speaking, chrysotile fibers can be compared to strands of fine textured hair. Bundles of long, thin fibers can bend at angles greater than 90 degrees without breaking. Long, thin fibers of chrysotile are usually not brittle, whether the material has been manufactured or is derived directly from a serpentine rock. Shorter fibers can be moderately brittle, and are generally thicker. There is an uncommon variety of chrysotile which has the form of thick, straight sticks. This type of chrysotile has bundles of short fibers that can be considerably different in appearance than that of common chrysotile that has been shortened during a milling process.

Chrysotile fibers are often obscured by other materials in the sample, such as fine clay-size calcite and gypsum in spray-on ceilings, leaving only the fiber ends exposed. Recognizing the fiber ends of chrysotile is an important tool in the identification of this common type of asbestos. The ends of chrysotile bundles and fibers are distinctive. Chrysotile bundles most often separate into individual fibers, that can frequently form split ends. Samples containing low percentages of chrysotile can be very difficult to identify, due to their sparseness. If a PLM analyst finds small pieces that cannot be identified, and suspects the sample of containing asbestos, the analyst should continue searching through the sample for a larger fiber so that a positive identification can be made. If only small pieces of fibers are detected, the analyst should re-prepare the sample until satisfied that the material has been properly identified.

The analyst should be aware of other fibrous materials that resemble chrysotile: among these is polyethylene, morphologically and optically very similar to chrysotile, except for the slightly higher birefringence of the former. If an analyst suspects a sample of containing polyethylene, the best way to determine this is to heat the sample with flame or hot plate. Polyethylene will incinerate at 136 degrees C, chrysotile at well over 1100 C. Another fiber that can resemble chrysotile in minute portions is cellulose. Analysts should look for a larger fiber that will show the much higher birefringence. Other substances that may be confused with asbestos are treated in McCrone (1987).

The crystal system of chrysotile is monoclinic and the optic sign is negative. Under plane light, chrysotile appears colorless to pale green and is non-pleochroic. The birefringence is 0.013 - 0.017. Under polarized light, chrysotile typically exhibits interference colors ranging from very dark grays to light grays and which increase with increasing thickness.

B) Chain Silicates, Amphibole Group

AMOSITE (CUMMINGTONITE-GRUNERITE)

Refractive Indices:	$\alpha = 1.635 - 1.696$ $\beta = 1.684 - 1.697$ $\gamma = 1.655 - 1.729$
Crystal System:	Monoclinic
Birefringence:	Strong, 0.020 - 0.045
Color:	Colorless to yellow/green to green/brown
Pleochroism:	Colored varieties are weakly pleochroic.
Extinction:	Mostly parallel; thin fibrils may show oblique extinction
Elongation:	Length - slow (+)
Optic Sign:	(-); $2V = 79^{\circ} - 86^{\circ}$
Chemistry:	$(\text{Fe}^{+2}, \text{Mg})_7[\text{Si}_8\text{O}_{22}](\text{OH})_2$

Recommended D.S. R.I. Liquid: Series E (High Dispersion) - 1.680

Grunerite is the name used for minerals of the cummingtonite - grunerite series in which iron is predominant over magnesium. Within the asbestos industry, asbestiform grunerite is commonly called amosite (a trade name derived from the "Asbestos Mines of South Africa", where large deposits of the material are mined).

Fibers of amosite range in morphology from relatively thick flat prisms to very long thin fibers, a characteristic that is a function of where their chemical composition falls within the cummingtonite -grunerite series. The thick, flat fibers are brittle and needle-like and are typically the fibers used in industry, these fibers are usually the iron-rich end member. The long thin fibers are moderately brittle and often bend in wide arches, these fibers are typically the softer, more flexible magnesium-rich end member called montasite.



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Bundles of amosite tend to separate into smaller groups of fibers. As the bundles break apart into single fibers they become very needle-like. Because amosite fibers do not normally fray or have split ends, the needle-like morphology is one way to distinguish this type of asbestos from chrysotile. Small thin fragments of the mineral are common in some building materials, and samples of this type as well as all other samples should be analyzed.

Amosite is monoclinic and typically colorless in plane light. The magnesium-rich end member of amosite is colorless and non-pleochroic, but as magnesium is replaced with iron, the fiber becomes pleochroic. Iron-rich grunerite fibers may have colors ranging from very pale yellows to light browns. It should be noted that, although non-fibrous grunerite is monoclinic, amosite fibers show parallel extinction. This is attributed to the overall increase in symmetry, caused by the random crystallographic orientation of fibers normal to the fiber axes. Apparently even narrow fibers will show this property, as they are actually bundles of very thin fibers.

The refractive index of amosite ranges from 1.635 to 1.729, depending on the orientation of the optical axes. Under plane light, a typical amosite fiber mounted in a refractive index liquid of 1.680 is faint or not visible at all. Under crossed polars amosite is readily recognizable. The thickness of the fiber and the high birefringence make the bright amosite fiber protrude dramatically against the dark background. The birefringence of amosite ranges from 0.020 - 0.045. Amosite under polarized light exhibits interference colors from bright gray to white and yellow along the margins, and even higher order colors with increasing thickness.



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#### CROCIDOLITE (RIEBECKITE)

Refractive Indices:	$\alpha = 1.654 - 1.701$
	$\beta = 1.662 - 1.711$
	$\gamma = 1.668 - 1.717$
Crystal System:	Monoclinic
Birefringence:	Weak, 0.006 - 0.016
Color:	Prussian blue to indigo-blue to yellow green
Pleochroism:	Very strong
Extinction:	Parallel
Elongation:	Length - fast/slow ( $\pm$ )
Optic Sign:	(-); $2V = 40^\circ - 90^\circ$
Chemistry:	$\text{Na}_2\text{Fe}^{+2}_3\text{Fe}^{+3}_2[\text{Si}_8\text{O}_{22}](\text{OH})_2$

Recommended D.S. R.I. Liquid: Series E (High Dispersion) - 1.680

Crocidolite is the asbestiform variety of riebeckite.

Riebeckite is the name used for minerals of the glaucophane-riebeckite series in which ferrous and ferric iron is predominant over magnesium and aluminum. Crocidolite is called the "Blue Asbestos", because it is strongly pleochroic, ranging from Prussian blue and indigo blue to yellow green. This distinctive pleochroism, and a negative sign of elongation, contrast with other types of asbestos. Under crossed polars, crocidolite shows shades of grays to whites and yellows, and even higher order colors with increasing thickness.

Riebeckite is monoclinic, but crocidolite fibers often show parallel extinction, similar to amosite (see above), although it is not uncommon to find extinction angles of up to  $20^\circ$ . The characteristic form of crocidolite is very similar to that of chrysotile. The fibers generally resemble strands of fine textured hair and are slightly more brittle than chrysotile, but can usually bend beyond  $90^\circ$  before breaking. Crocidolite is harder than the other varieties of asbestos in that it resist cutting by a knife blade.





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#### ANTHOPHYLLITE

Refractive Indices:  $\alpha = 1.596 - 1.652$

$\beta = 1.605 - 1.662$

$\gamma = 1.615 - 1.676$

Crystal System: Orthorhombic

Birefringence: 0.013 - 0.028

Color and Pleochroism: Colorless to pale green or yellow; weak gray-brown to brown pleochroism may be observed

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = 70^\circ$  to  $90^\circ$

Chemistry:  $(\text{Mg,Fe})_7[\text{Si}_8\text{O}_{22}](\text{OH,F})_2$

Recommended D.S. R.I. Liquid: Series E (High Dispersion) - 1.620

Anthophyllite is the magnesian end-member of the anthophyllite-gedrite series and is optically negative. The accepted refractive index liquid used when analyzing for anthophyllite is 1.605. Under plane light, anthophyllite is distinguishable from the liquid because the refractive index of the mineral is most often between 1.623 to 1.676. Under polarized light, in 1.605, anthophyllite shows colors ranging from off-white, to yellow, to orange/yellow. Some of the colored varieties show faint pleochroism.

Anthophyllite is an orthorhombic amphibole. Fibers of anthophyllite always appear to be extremely two-dimensional, appearing to be extremely flat and thin. The characteristic shape resembles that of a knife blade, coming to a point at one end. The fibers from a particular sample tend to be relatively uniform in size. The parallel extinction of the mineral should distinguish it from tremolite or actinolite.



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#### TREMOLITE-ACTINOLITE

Refractive Indices:  $\alpha = 1.599 - 1.668$   
 $\beta = 1.612 - 1.655$   
 $\gamma = 1.622 - 1.688$

Crystal System: Monoclinic

Birefringence: 0.017 - 0.027

Color and pleochroism: Colorless to yellow green, green and blue green; colored varieties are pleochroic

Extinction: Commonly oblique,  $0^\circ - 20^\circ$ , though may show parallel extinction

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = 65^\circ - 86^\circ$

Chemistry:  $\text{Ca}_2(\text{Mg,Fe})_5[\text{Si}_8\text{O}_{22}](\text{OH,F})_2$

Recommended D.S. R.I. Liquid: Series E (High Dispersion) - 1.620

Tremolite and actinolite form a continuous isomorphous series. The division between tremolite and actinolite is defined by the relative amounts of magnesium and iron. Tremolite is the magnesian end-member of the series and is white in the hand sample; manganiferous varieties are pink or pale violet. Actinolite is higher in iron content and is green in the hand sample. The accepted refractive index to use when analyzing for tremolite is 1.605. The refractive indices increase with increasing iron content. Under plane light, the fibers appear outlined and hollow in the center. Under polarized light, the fibers show colors ranging from white to off-white to yellow. The morphology of tremolite and actinolite ranges from prismatic or blade-like to acicular (needle-like). The blades are often pointed at both ends. Bundles commonly exhibit a radial arrangement.

**NOTE** - Wollastonite, a pyroxenoid mineral, appears very similar to tremolite/actinolite in its morphology, refractive indices, and extinction angle. The major difference between tremolite/actinolite and wollastonite is that the former are always length slow, while the latter is both length slow and length fast. To differentiate tremolite/actinolite from wollastonite be sure to look for this optic effect as it is not always immediately obvious. One method of testing a suspect material is to tap the coverslip while looking at the



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sample under the microscope in such a manner that the fibers roll onto a different cleavage face. Look to see if they change their sign of elongation.



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#### **Characteristic Optical, Physical, & Chemical Properties of Common Asbestos Look-Alike Minerals & Materials**

##### **A. Non-Mineral Materials**

###### **CELLULOSE**

Refractive Indices: Perpendicular to fiber axis ( $>1.552$ )

Parallel to fiber axis ( $<1.552$ )

Birefringence: Moderate to High

Color and pleochroism: Lt. brown, lt. yellow to colorless in thin section.

Extinction: Parallel, often mottled

Elongation: Length - slow (+)

Chemistry: Organic

Cellulose is found quite commonly in many building materials. It displays characteristic highly elongate fibers which are virtually always bent in an irregular wavy fashion. This fact allows for it to be confused on occasion with chrysotile. The fibers, however, are usually much fatter than chrysotile and appear ribbon-like, thinning dramatically where folded over on themselves. Other distinguishing characteristics of cellulose from chrysotile include 1) the much more variable refractive indices both higher and lower, 2) the much higher birefringence with first order colors common, 3) the presence of mottled incomplete extinction.

Subjecting sample to flame and burning away of the cellulose can be of help in discerning cellulose from chrysotile when both are present.



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#### **POLYETHYLENE**

Refractive Indices: Perpendicular to fiber axis (~1.552)

Parallel to fiber axis (~1.552)

Birefringence: ~0.010 - 0.020

Color and pleochroism: Colorless in thin section.

Extinction: Parallel

Elongation: Length - fast or length - slow, depending on how

Chemistry: Organic

Polyethylene and other similar plastic materials are commonly found in ceiling tile materials and can easily be confused with chrysotile. Several differentiating characteristics are present however and include: 1) A subtle difference in morphology is seen with polyethylene exhibiting more individual fibers and where split ends of fiber bundles, common in chrysotile, are not seen, 2) Where plastic sheeting is present, it commonly exhibits both length fast and length slow characteristics, and really doesn't exhibit a fibrous morphology upon close examination.

Subjecting sample to flame and burning away of the polyethylene can be of great help in discerning polyethylene and other plastic materials from chrysotile.



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#### **FIBERGLASS**

Refractive Indices: Uniform in all directions (~1.50 - 1.70)

Birefringence: None, extinguished under crossed polars

Color and pleochroism: Colorless in thin section.

Extinction: Isotropic

Elongation: Not applicable

Chemistry: Silicate glass

Fiberglass is very common in ceiling tiles and insulation materials. It commonly occurs as long, very straight rods. Occasionally, in mineral wool samples, it will be curved and bent, often with irregular blobs of glass attached, due to irregular forming procedures. The distinguishing characteristic of fiberglass is its isotropic behavior where it disappears under crossed polars. To see the fibers better, it is often useful to insert the retardation plate where the fibers maintain a constant first order red color throughout rotation.



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#### B. Sulfates

##### GYP SUM

Refractive Indices:  $\alpha = 1.519 - 1.521$

$\beta = 1.523 - 1.526$

$\gamma = 1.529 - 1.531$

Crystal System: Monoclinic

Birefringence:  $\sim 0.01$

Color and pleochroism: Colorless in thin section.

Extinction: Oblique,  $\sim 38^\circ$  on 010 faces, though may show lesser extinction angles in other sections.

Elongation: Length - fast, although may be very close to length slow

Optic Sign: (+);  $2V = \sim 58^\circ$

Chemistry:  $\text{CaSO}_4 \cdot \text{H}_2\text{O}$

Gypsum commonly occurs in building materials, and is most often found in sheet rock, plaster, and other cementitious materials. It characteristically forms in both fibrous needles and non-fibrous habits. The needles are usually quite small and often very close to the edge of the 3:1 EPA fiber definition, unlike most amphiboles. The gypsum needles are also commonly extremely numerous and are found in a fine grained groundmass of poorly resolvable gypsum. The most distinguishing characteristic of gypsum from the amphiboles is its extremely high extinction angle, much higher than any of the asbestiform amphiboles.



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#### ANHYDRITE

Refractive Indices:  $\alpha = 1.569 - 1.574$

$\beta = 1.574 - 1.579$

$\gamma = 1.609 - 1.618$

Crystal System: Orthorhombic

Birefringence:  $\sim 0.040$

Color and pleochroism: Colorless in thin section.

Extinction: Parallel

Elongation: Length - fast or length slow depending on section.  
i.e. when rolled it may flip it sign of elongation.

Optic Sign: (+);  $2V = 42^\circ - 44^\circ$

Chemistry: CaSO4

Anhydrite is not commonly found in building materials, but does occasionally occur. It's most common form is not as fibers and usually is not differentiated on those occasions. However, it can appear in fibrous habit. It's most differentiating characteristics are its lower refractive indices than the amphibole asbestos minerals.





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#### C. Carbonates

##### ARAGONITE

Refractive Indices:  $\alpha = 1.530 - 1.531$

$\beta = 1.680 - 1.681$

$\gamma = 1.685 - 1.686$

Crystal System: Orthorhombic

Birefringence: 0.155 - 0.156

Color and pleochroism: Colorless in thin section.

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = \sim 18^\circ$

Chemistry:  $\text{CaCO}_3$

Aragonite occasionally occurs in building materials, and is most often found in plaster, and other cementitious materials. It characteristically forms in both fibrous needles and non-fibrous habits. The needles are usually quite small and often very close to the edge of the 3:1 EPA fiber definition, unlike most amphiboles. Aragonite commonly occurs in samples that also contain calcite. The most distinguishing characteristics of aragonite needles from amphibole asbestos are 1) their length slow sign of elongation & 2) their very high birefringence.

The presence of can sometimes be detected, if it is in large enough quantities, by placing a portion of sample material into a drop of dilute acid, where it will vigorously fizz and dissolve.



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#### D. Silicate Minerals

##### ANTIGORITE

Refractive Indices:  $\alpha = 1.558 - 1.567$

$\beta = \simeq 1.566$

$\gamma = 1.562 - 1.574$

Crystal System: Monoclinic

Birefringence: 0.004 - 0.007

Color and pleochroism: Green, green-blue, white. Colorless to pale green in thin section.

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = 37^\circ - 61^\circ$

Chemistry:  $\text{Mg}_3[\text{Si}_2\text{O}_5](\text{OH})_4$

Antigorite is a polymorph of the serpentine family of minerals, of which chrysotile is a member. Antigorite can be mistaken for asbestos if the analyst is not careful. Typically, antigorite is not seen in building materials. However, in soil and rock samples, particularly from the California coast ranges or foothills which often contain abundant serpentine minerals, it is quite common.

Distinguishing antigorite from chrysotile can best be done on the basis of two criteria, 1) fiber morphology and, 2) refractive index. While antigorite often occurs in a fibrous habit, it is not as well developed as in chrysotile which is often hairlike in form. However, when chrysotile is present in small fiber bundles and does not manifest the fine fibrous morphology, it can be difficult to distinguish between the two. The other technique for differentiating the two minerals is by refractive index. While the two are quite close in refractive index, antigorite is consistently higher than 1.55, whereas chrysotile is equal to or less than 1.55.



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#### TALC

Refractive Indices:  $\alpha = 1.539 - 1.550$

$\beta = 1.589 - 1.594$

$\gamma = 1.589 - 1.600$

Crystal System: Monoclinic

Birefringence: 0.05

Color and pleochroism: Colorless, white, pale green, dark green, brown.  
Colorless in thin section.

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = 0^\circ - 30^\circ$

Chemistry:  $\text{Mg}_6[\text{Si}_8\text{O}_{20}](\text{OH})_4$

Talc is an asbestos look-alike mineral when it occurs in a fibrous morphology, which is not uncommon. Talc is a hydrous magnesium silicate mineral with composition and optical properties which can lead to its mis-identification as chrysotile or anthophyllite if care is not taken. However, talc has a variety of features by which it can be uniquely identified. These properties include: 1) Morphology, 2) Refractive Index and, 3) Birefringence.

The morphology of talc can be highly fibrous. However, it usually appears stiffer than chrysotile, and more flexible than amphiboles. The refractive index of talc, can mimic chrysotile or amphibole if the analyst does not look at both the parallel and perpendicular vibration directions. The refractive indices of talc can be identical to chrysotile in the perpendicular direction, and approximately equal to anthophyllite the parallel direction. However, looking at talc in both 1.55 and 1.604 refractive index oil, and by looking at the fibers in both the parallel and perpendicular directions, can lead to its quick and easy identification. The birefringence of talc is typically higher than chrysotile and closer to amphiboles.

Talc, when seen in building materials, is most commonly observed in flooring mastics and glues.



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#### COMMON HORNBLENDE

Refractive Indices:  $\alpha = 1.615 - 1.705$

$\beta = 1.618 - 1.714$

$\gamma = 1.632 - 1.730$

Crystal System: Monoclinic

Birefringence: 0.014 - 0.026

Color and pleochroism: Pale green, green, lt. yellow-brown to brown. Pleochroism variable in greens, yellow-green, bluish-green and brown.

Extinction: Commonly oblique,  $130^\circ - 340^\circ$ , though may show parallel extinction in certain sections.

Elongation: Length - slow

Optic Sign: (-);  $2V = 950^\circ - 270^\circ$

Chemistry:  $(\text{Na},\text{K})_{0-1}\text{Ca}_2(\text{Mg},\text{Fe}^{+2},\text{Fe}^{+3},\text{Al})_5$   
 $[\text{Si}_{6-7}\text{Al}_{2-1}\text{O}_{22}](\text{OH},\text{F})_2$

Common hornblende and other related non-asbestos amphibole minerals can, on occasion, be difficult to differentiate from asbestiform amphiboles when they occur with aspect ratios of 3:1 or greater. However, usually their aspect ratio is very close to 3:1, unlike the asbestiform amphiboles. The most easily recognizable differences are 1) the stronger pleochroism of the iron rich hornblende varieties, 2) the higher refractive indices of the iron rich varieties. Differentiation of the iron-poor hornblende minerals, if they occur with aspect ratios of  $>3:1$ , is difficult and best performed through a combination of refractive index tests with consultation of refractive index vs. composition charts found in Deer, Howie & Zussman, as well as notation of the subtle pleochroism differences.



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#### WOLLASTONITE

Refractive Indices:  $\alpha = 1.616 - 1.640$

$\beta = 1.628 - 1.650$

$\gamma = 1.631 - 1.653$

Crystal System: Triclinic

Birefringence: 0.013 - 0.014

Color and pleochroism: Colorless in thin section.

Extinction: Commonly oblique,  $39^\circ$ , though may show parallel extinction in certain sections.

Elongation: Length - slow & Length - fast

Optic Sign: (-);  $2V = 95^\circ - 27^\circ$

Chemistry:  $\text{Ca}[\text{SiO}_3]$

Wollastonite is not commonly found in building materials, though it is occasionally present. Its morphology, when fibrous, is only weakly asbestiform (near 3:1 aspect ratio) however, on occasion it closely mimics tremolite/actinolite. It is distinguished from the amphibole forms of asbestos by its weaker birefringence, and its variable sign of elongation. To test the variable sign of elongation, view the sample with the retardation plate inserted and under crossed polars, and with a pair of tweezers, push on the coverslip and get the fiber to roll over. If it changes its sign of elongation, it is wollastonite and not tremolite/actinolite.



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#### **Characteristic Optical, Physical, & Chemical Properties of Other Common Minerals & Materials Found in Building Materials**

##### QUARTZ

Refractive Indices:  $\omega = 1.544$

$\varepsilon = 1.553$

Crystal System: TRIGONAL

Birefringence: 0.009

Color and pleochroism: Colorless in thin section.

Extinction: Irregular to conchoidal fracture.

Elongation: Not applicable

Optic Sign: (+)

Chemistry:  $\text{SiO}_2$

Quartz is commonly found in plasters, cementitious materials, and soils. It has no cleavage and commonly occurs as rounded grains. It is extremely hard and usually impossible to reduce in size. It has low birefringence and very constant refractive indices.



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#### ALKALI FELDSPARS

Refractive Indices:  $\alpha = 1.518 - 1.529$

$\beta = 1.518 - 1.533$

$\gamma = 1.521 - 1.539$

Crystal System: Monoclinic & Triclinic

Birefringence: 0.006 - 0.010 (Low)

Color and pleochroism: Colorless in thin section.

Extinction: Commonly oblique to twinning and exsolution lamellae, though may be parallel in certain sections.

Elongation: Not Applicable

Optic Sign: (+ or -);  $2V = 5^\circ - 20^\circ$

Chemistry:  $(K,Na)[AlSi_3O_8]$

Alkali feldspars rarely appear as fibers (though sanidine may). They are generally present in building materials as rounded grains in cementitious materials with quartz. They are commonly distinguished from quartz by the presence of twinning and its lower refractive indices. It is not necessary to differentiate between the alkali and the plagioclase feldspars and the term feldspar may be used.



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#### PLAGIOCLASE FELDSPARS

Refractive Indices:  $\alpha = 1.527 - 1.577$

$\beta = 1.532 - 1.585$

$\gamma = 1.534 - 1.590$

Crystal System: Triclinic

Birefringence: 0.007 - 0.013 (Low)

Color and pleochroism: Colorless in thin section.

Extinction: Commonly oblique to twinning and exsolution lamellae, though may be parallel in certain sections.

Elongation: Not Applicable

Optic Sign: (+ or -);  $2V = 45^\circ - 78^\circ$

Chemistry:  $\text{Na}[\text{AlSi}_3\text{O}_8] - \text{Ca}[\text{Al}_2\text{Si}_2\text{O}_8]$

Plagioclase feldspars do not appear as fibers. They are generally present in building materials as rounded grains in cementitious materials with quartz. They can commonly be distinguished from quartz by the presence of twinning and their lower refractive indices. It is not necessary to differentiate between the alkali and the plagioclase feldspars and the term feldspar may be used.





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#### CALCITE

Refractive Indices:  $\omega = 1.486 - 1.550$

$\varepsilon = 1.658 - 1.740$

Crystal System: TRIGONAL

Birefringence: 0.172 - 0.190

Color and pleochroism: Colorless in thin section.

Extinction: Symmetrical

Elongation: Not applicable

Optic Sign: (-)

Chemistry:  $\text{CaCO}_3$

Calcite is extremely common in building materials and may occur in floor tiles, ceiling tiles, spray-on ceilings, and in plasters and other cementitious materials. It does not occur as fibers, but often as rhombic sections. It is identified by its extremely high birefringence, and the common occurrence of twinning.

The presence of calcite can be easily tested by placing a portion of sample material into a drop of dilute acid, where it will vigorously fizz and dissolve.



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#### Documentation

The following documents are managed:

Required documents	Custodian
EPA: Method for the Determination of Asbestos in Bulk Building Materials	Laboratory Manager
EPA: Interim Method for the Determination of Asbestos in Bulk Insulation Samples	Laboratory Manager
EPA: Asbestos-Containing Materials in Schools; Final Rule and Notice	Laboratory Manager

#### Revision History

Revision	Date	Revision Notes
1	October, 1996	
2	Feburary,2005	Format, Wording and Reference Update

Approval

**Yanxia Xie**

Reviewer

**02 / 16 / 2005**

Date

**Yanxia Xie** Init:\_\_\_\_\_

Quality Assurance Manager

**02 / 16 / 2005**

Date



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**Appendix A: Sample PLM Analysis Data Sheets**

**Appendix B: Sample PLM Report**

**Appendix C: Refractive Index Measurement**



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- APPENDIX A -

SAMPLE PLM ANALYSIS DATA SHEETS



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(Replace with data analysis sheet page)



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(Replace with Analysis Data Sheet)



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(Replace with Analysis Data Sheet)





Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

Revision: **0**

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- APPENDIX B -

SAMPLE PLM REPORT



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

Revision: **0**

SOP#: **5-04-2-PLM-01**

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

Revision: **0**

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

Revision: **0**

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

Revision: **0**

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis – Standard Method**

Issue Date: **2005/02/16**

Revision: **0**

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## **- APPENDIX C -**

### **REFRACTIVE INDEX MEASUREMENT**

<b>1005</b>	<b>00075</b>	<b>001</b>	<b>B</b>	<b>UNLV-279-2004</b>						
<b>DESCRIPTION</b>		<b>COLOR</b>		<b>HOMC</b>	<b>FRIAE</b>	<b>TEXTURE</b>		<b>EST. % ASB.</b>		
Mastic		Yellow		Good	on-Friat	Gummy		ND		

Morpholog	Color/Ple		Ref Index		Birel	Sign	Eln	Ext	Pct		FiberTyp
			Par	Perp							
Ribbons	Clear	None	>1.55	<1.55	H	+		P	1	5	Cellulose

<b>NON-FIBROUS</b> <span style="border: 1px solid black; padding: 2px;">Glue, Calc, Other m.p.</span>	<div style="border: 1px solid black; padding: 2px; text-align: center;">ND ASBESTOS</div> <div style="border: 1px solid black; padding: 2px; text-align: center;">1-5% NON-ASBESTOS</div> <div style="border: 1px solid black; padding: 2px; text-align: center;">95-99% NON-FIBROUS</div>
---	--

<b>NOTES</b> <span style="border: 1px solid black; display: inline-block; width: 250px; height: 20px;"></span> <div style="display: flex; justify-content: space-between; margin-top: 5px;"> <span>DATE Jan-05-05</span> <span>TIME 11:39</span> <span>Anal DV</span> </div> <div style="margin-top: 10px;"> <p style="text-align: center;"><b>Sample Prep Methods Used</b></p> <div style="display: flex; flex-wrap: wrap;"> <div style="width: 50%;"><input type="checkbox"/> Acid</div> <div style="width: 50%;"><input type="checkbox"/> Chop</div> <div style="width: 50%;"><input type="checkbox"/> Shave</div> <div style="width: 50%;"><input type="checkbox"/> Heat</div> <div style="width: 50%;"><input type="checkbox"/> Burn</div> <div style="width: 50%;"><input type="checkbox"/> Grind</div> <div style="width: 50%;"><input type="checkbox"/> Tease</div> </div> </div>	<div style="border: 1px solid black; padding: 5px; margin-bottom: 5px;"> <b>NON-ASBESTOS</b> <span style="border: 1px solid black; padding: 2px;">1-5% Cellulose</span> </div> <div style="border: 1px solid black; padding: 5px;"> <b>NON-FIBROUS</b> <span style="border: 1px solid black; padding: 2px;">95-99% Glue, Calc, Other m.p.</span> </div>
---	---

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# POLARIZED LIGHT MICROSCOPY DATA SHEET

Asbestos TEM Laboratories, Inc.

Lab I.D. #: \_\_\_\_\_

Job Site: \_\_\_\_\_

Client I.D. #: \_\_\_\_\_

Location: \_\_\_\_\_

Description: \_\_\_\_\_

Color: \_\_\_\_\_

## MACROSCOPIC PROPERTIES:

Homogeneity: Low ☐ Mod. ☐ High ☐

Friable: ☐ Non-friable: ☐

Texture: \_\_\_\_\_

Macroscopic est. of asbestos: \_\_\_\_\_

Morphology	Color	Pleochroism	Ref. Indx Perp./Parall.	Birefr.	Extinct Angle	Sign of Elong.	Other Properties	%	I.D. (Non-Asb.)	%	I.D. (Asb)

Non-fibrous Material: \_\_\_\_\_

\_\_\_\_\_

Comments: \_\_\_\_\_

\_\_\_\_\_

☐ % Asbestos  
☐ % Non-Asbestos, Fibrous  
☐ % Non-Fibrous

Signature: \_\_\_\_\_

Date: \_\_\_\_\_

# POLARIZED LIGHT MICROSCOPY POINT COUNT DATA SHEET



Asbestos TEM Laboratories, Inc.  
Lab I.D. #: \_\_\_\_\_

Client I.D. #: \_\_\_\_\_

Description: \_\_\_\_\_

Job Site: \_\_\_\_\_

Location: \_\_\_\_\_

Color: \_\_\_\_\_

## MACROSCOPIC PROPERTIES:

Homogeneity: Low ☐ Mod. ☐ High ☐

Friable: ☐ Non-friable: ☐

Texture: \_\_\_\_\_

Macroscopic est. of asbestos: \_\_\_\_\_

Morphology	Color	Pleochroism	Ref. Indx Perp./Parall.	Birefr.	Extinct Angle	Sign of Elong.	Fiber Identity	Prep #	Chrys	Amph	Other
								Totals:	<input type="text"/>	<input type="text"/>	<input type="text"/>
								Total Asb. %:	<input type="text"/>		

Non-fibrous Material: \_\_\_\_\_

Comments: \_\_\_\_\_

Signature: \_\_\_\_\_

Date: \_\_\_\_\_

**RAPIDLY AND ACCURATELY DETERMINING  
REFRACTIVE INDICES OF ASBESTOS FIBERS  
BY USING  
DISPERSION STAINING METHOD**

A STANDARD OPERATION PROCEDURE FOR  
BULK ASBESTOS ANALYSIS BY  
POLARIZED LIGHT MICROSCOPY

**Shu-Chun Su, Ph.D.**

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USA

**Revision 2003**

For those laboratories that have received earlier versions of this paper, please replace them with this updated version. In one of the previous version, Table 25A (Anthophyllite  $n_z$  in 1.625) is identical to Table 22A (Actinolite  $n_z$  in 1.605) due to an editing error.

This version uses the optical data of NIST SRM 1867 to recalculate all tables for tremolite, actinolite, and anthophyllite.

This version also contains several more conversion tables for amphiboles, which should be useful in analyzing NVLAP Proficiency Testing samples.

If anyone has any questions or suggestions concerning this procedure or need the electronic files (Word or Pdf format), I can be reached at 302-995-3498 or [ssu@herc.com](mailto:ssu@herc.com).

Shu-Chun Su  
Technical Expert for NVLAP  
Bulk and Airborne Asbestos Programs  
2003

## Introduction

Refractive index (RI) is the most important optical properties of non-opaque minerals. It is also the leading diagnostic optical property used to identify asbestos components in bulk insulation or building materials by polarized light microscopy (PLM) using oil immersion method (Perkins and Harvey, 1993). Most environmental laboratories in the United States participate in the National Voluntary Laboratory Accreditation Program (NVLAP) administered by the National Institute of Standards and Technology (NIST), U.S. Department of Commerce. NVLAP requires the refractive indices  $\alpha$  and  $\gamma$  of asbestos fibers to be determined and recorded during routine bulk asbestos sample analysis. Generally, an attainable and reasonable accuracy is  $\pm 0.005$  for chrysotile, amosite, tremolite, actinolite, and anthophyllite, or  $\pm 0.010$  for crocidolite.

In many environmental laboratories, the high volume of samples demands analysts to minimize the amount of time spent on the determination of required optical properties, particularly the refractive indices. It is most desirable to determine both  $\alpha$  and  $\gamma$  from a single slide or a preparation (Su, 1993). Among the three methods for assessing the direction and magnitude of the mismatch between a solid and a surrounding liquid, Becke line (Bloss, 1961), dispersion staining (McCrone, 1987), and oblique illumination (Stoiber and Morse, 1994), only the later two, i.e., dispersion staining (DS) and oblique illumination (OI), can meet the specific needs for the routine PLM analysis of bulk asbestos samples in commercial environmental laboratories. The advantage of OI method is that it is as simple and accurate as DS and does not require special objective lens. In the meantime, it can be applied using high power objective lens (20X, 40X, etc.)

This paper provides a rapid and accurate procedure to enable bulk asbestos analysts to convert an observed DS color associated with  $\alpha$  or  $\gamma$  for a specific asbestos mineral in a specific immersion liquid through its corresponding matching wavelength ( $\lambda_0$ ) into corresponding numerical RI value.

## Procedure

### 1. Select a proper immersion liquid to mount the sample

Mount the suspected asbestos fibers in an appropriate liquid according to Table 1. For asbestos types other than chrysotile, there are two choices of immersion liquids. The first choice, which is the liquid outside the parentheses, gives higher accuracy than the second choice, which is the liquid inside the parentheses. For example, when measuring crocidolite, 1.700 liquid is a much better choice than 1.680. For routine analysis, 1.550 (for chrysotile), 1.620 (for tremolite, actinolite, and anthophyllite), and 1.700 (for amosite and crocidolite) are quite adequate to obtain good accuracy for both  $\alpha$  and  $\gamma$ . When higher accuracy is desirable (for example, when performing NVLAP Proficiency Testing), other liquids may be more appropriate and different liquids may be used for  $\alpha$  and  $\gamma$ . For example, use 1.615 for the  $\alpha$  and 1.635 for the  $\gamma$  of anthophyllite. Therefore, additional tables are included for higher accuracy work.

It is imperative to have fresh surface of asbestos fibers in direct contact with the surrounding liquid. Sometimes, the surface of an asbestos bundle may be coated by matrix or binder materials. In this case, true DS colors intrinsic to the asbestos/liquid combination might not be displayed.

Table 1. The Selection of Immersion Liquids for Asbestos Analysis

Suspected Asbestos		Immersion Liquids (Conversion Table Number)	
Type	RI	Proficiency Samples (Different liquids for $\alpha$ and $\gamma$ )	Routine Samples (Same liquid for both $\alpha$ and $\gamma$ )
Chrysotile	$\alpha$	1.550 (4A)	1.550 (4A/B)
	$\gamma$	1.560 (5B)	
Grunerite (Amosite)	$\alpha$	1.680 (6A)	1.700 (7A/B) [or 1.680 (6A/B)]
	$\gamma$	1.700 (7B)	
Riebeckite (Crocidolite)	$\alpha$	1.700 (9A)	1.700 (9A/B) [or 1.680 (8A/B)]
	$\gamma$	1.710 (11B)	
Tremolite	$\alpha$	1.605 (12A)	1.625 <sup>1</sup> (15A/B) [or 1.605 <sup>2</sup> (12A/B)]
	$\gamma$	1.635 <sup>1</sup> (16B)	
Actinolite	$\alpha$	1.615 <sup>1</sup> (18A)	1.625 <sup>1</sup> (20A/B) [or 1.605 <sup>2</sup> (17A/B)]
	$\gamma$	1.640 <sup>1</sup> (21B)	
Anthophyllite	$\alpha$	1.615 <sup>1</sup> (23A)	1.625 <sup>1</sup> (25A/B) [or 1.605 <sup>2</sup> (22A/B)]
	$\gamma$	1.635 <sup>1</sup> (26B)	

1. Cargille makes two series of immersion liquids in the range of 1.500 to 1.640: Series A (normal dispersion) and Series E (high dispersion). All these are Series E liquids. For tremolite, actinolite, and anthophyllite, the central-stop DS colors produced by these Series E high dispersion liquids are more intense and vivid than those produced by Series A liquids. Tables 12 to 26 are no longer applicable if Series A liquids are used instead.
2. For qualitative analysis, 1.605 liquid is adequate for these three amphiboles. When accurate RI measurement is required, 1.605 liquid should be avoided because in most cases both  $\alpha$  and  $\gamma$  are higher or significantly higher than 1.605 and exhibit yellow to pale yellow central-stop DS colors. The inherent error in converting DS colors to  $\lambda_0$  is always higher in that range than in the range of blue to orange.

A simple and effective way to bring out the true DS colors is to grind or rub the fiber bundle with a needle or probe to break the fiber bundle into finer bundles so that fresh surface is revealed and made in direct contact with the surrounding liquid.

## 2. Measure the temperature of the immersion liquid

Measure and record  $t$  (in EC), the temperature of the immersion liquid on the microscope slide. If the temperature of the liquid, slide, cover glass and sample can be reasonably assumed to be in equilibrium with the room temperature,  $t$  can be assumed to be equal to the room temperature. The temperature data is needed for making temperature correction. Certain microscopes tend to heat up the slide, resulting an increase 2E or more in the liquid temperature.

### 3. Check the alignment of the polarized light microscope

Make sure that the polarized light microscope is properly aligned:

- DS objective and its central stop is centered;
- substage condenser is centered (if possible, set the microscope to Köhler illumination);
- the vibration (or privileged) directions of polarizer and analyzer are parallel to the E-W and N-S cross hairs in the eyepiece, respectively.

### 4. Observe the central-stop DS color associated with $\alpha$ of the asbestos fibers

Assuming that the polarizer is parallel to the E-W cross hair, rotate the microscope stage until a fiber bundle is parallel to the E-W cross hair if the asbestos is suspected to be crocidolite or perpendicular to the E-W cross hair if the asbestos is suspected to be other five asbestos types (chrysotile, tremolite, actinolite, anthophyllite, and amosite). Although the  $\alpha$  of monoclinic amphiboles (tremolite and actinolite) is not exactly perpendicular to the fiber elongation, the RI at this orientation can be assumed to be reasonably close to  $\alpha$ . Adjust the aperture diaphragm and field diaphragm to optimize the DS color displayed by the asbestos fibers.

Usually, a range of DS color is displayed. Make sure that the DS color that gives the **lowest** RI is observed, i.e. the DS color corresponding to the **longest**  $\lambda_0$ . For example, if the DS color ranges from blue to light blue, choose light blue.

### 5. Convert the observed DS color into corresponding matching wavelength, $\lambda_0$ , between the asbestos fiber and the immersion liquid used by referring to Figure 1 or Table 2

### 6. Find out the numerical value of $\alpha$ corresponding to the observed $\lambda_0$ and $t$

Refer to one of the conversion tables to convert  $\lambda_0$  and  $t$  into the corresponding refractive index. Notice that each table is for a specific direction ( $\alpha$  or  $\gamma$ ) of a specific asbestos mounted in a specific RI liquid. If an RI liquid with a different  $n_D$  and/or a different dispersion coefficient [ $n_F - n_C$ ] is used, the current tables are no longer applicable. In this case, a new table may be calculated by using a FORTRAN computer program *SLIQUID* written by the author, which is available upon request (send a formatted 32" or 53" disk and a pre-addressed return envelope to the author). The algorithm used to compute all conversion tables in this paper can be found in Su (1993), which is included in this SOP as an Appendix.

### 7. Observe the DS color associated with $\gamma$ of the asbestos fibers

Rotate the microscope stage 90E and then repeat Steps 4 - 6 to determine  $\gamma$ . Again, a range of DS color is usually displayed. Make sure that the DS color that gives the **highest** RI is observed, i.e. the DS color corresponding to the **shortest**  $\lambda_0$ . For example, if the DS color ranges from purple to red-purple, choose red-purple.

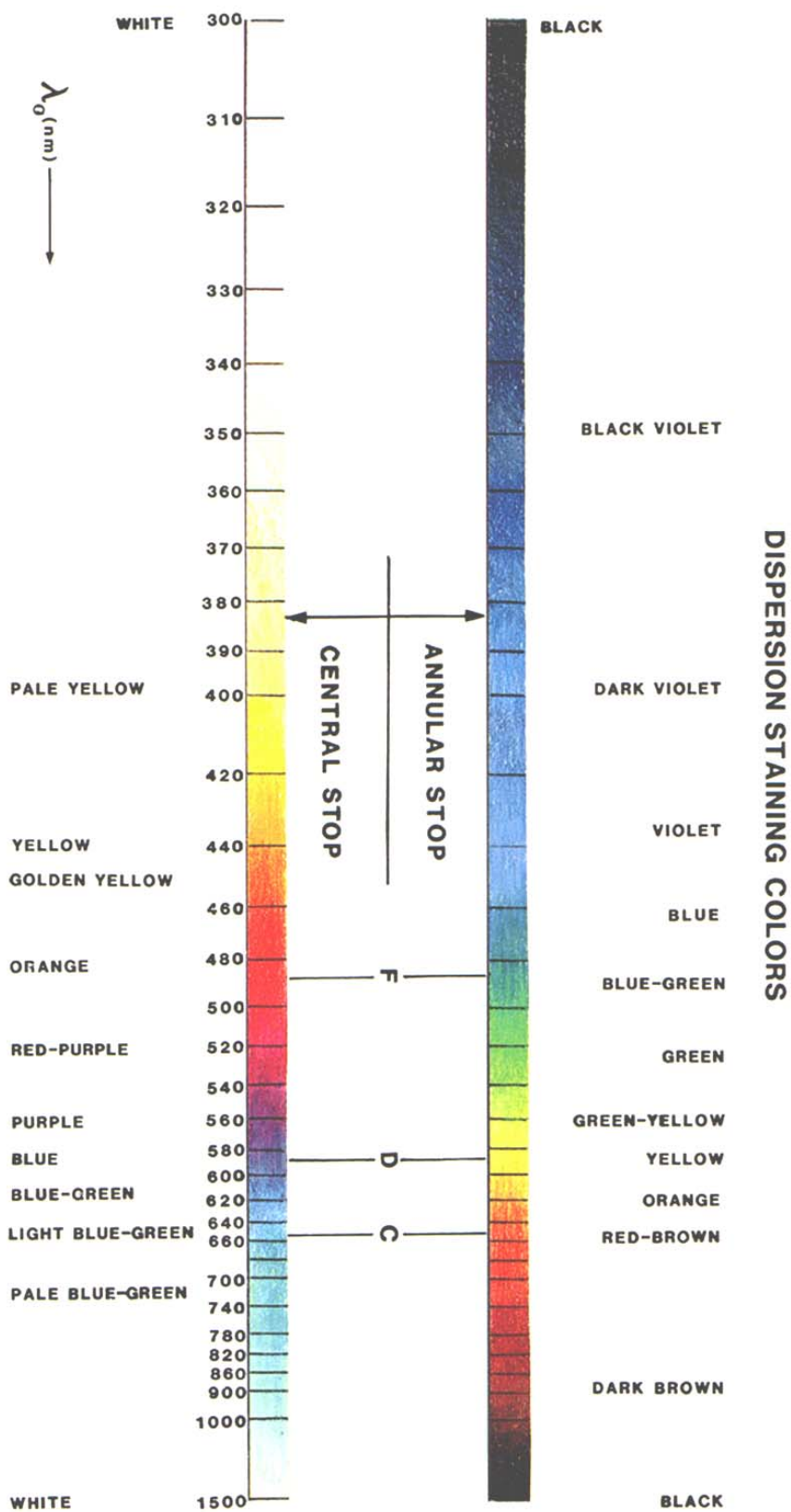
Fig. 1. Converting dispersion staining color to corresponding  $\lambda_0$  (McCrone, 1987)



Table 2. Converting dispersion staining color to corresponding  $\lambda_0$  (McCrone, 1987)

Matching Wavelength $\lambda_0$ , nm	Particle Edge Colors <sup>1</sup>		Becke Line Colors <sup>2</sup>	
	Annular Stop <sup>3</sup>	Central Stop <sup>4</sup>	Particle	Liquid
<340	Black violet	white	white	X
<400	dark violet	pale yellow	pale yellow	X
430	violet	yellow	pale yellow	X
455	blue	golden yellow	yellow	violet
485	blue-green	orange	orange	violet
520	green	red purple	orange-red	violet-blue
560	yellow-green	purple	red-orange	blue-violet
595	yellow	deep blue	red	blue
625	orange	blue-green	faint red	blue
660	red-brown	light blue-green	X	blue-green
700	dark red-brown	pale blue-green	X	pale blue-green
1500	black-brown	very pale blue-green	X	very pale blue-green

1. In focus
2. On focusing up
3. Observed on a brightfield
4. Observed on a darkfield

Table 3. Refractive Indices and Dispersion Coefficients [ $n_F - n_C$ ] of Six Asbestos Minerals

Mineral		$n_F$	$n_D$	$n_C$	$[n_F - n_C]$	Reference
Chrysotile	$\alpha$	1.5563	1.5490	1.5456	0.0107	NIST SRM 1866
	$\gamma$	1.5649	1.5560	1.5530	0.0119	
Grunerite (Amosite)	$\alpha$	1.6931	1.6790	1.6734	0.0197	NIST SRM 1866
	$\gamma$	1.7156	1.7010	1.6951	0.0205	
Riebeckite (Crocidolite)	$\alpha$	1.7132	1.7015	1.6971	0.0161	McCrone (1987) Figs. 104A and 104B
	$\gamma$	1.7206	1.7072	1.7032	0.0174	
Tremolite	$\alpha$	1.6128	1.6063	1.6036	0.0092	NIST SRM 1867
	$\beta$	1.6299	1.6230	1.6201	0.0098	
	$\gamma$	1.6423	1.6343	1.6310	0.0113	
Actinolite	$\alpha$	1.6201	1.6126	1.6095	0.0106	NIST SRM 1867
	$\beta$	1.6369	1.6288	1.6254	0.0115	
	$\gamma$	1.6485	1.6393	1.6355	0.0130	
Anthophyllite	$\alpha$	1.6227	1.6148	1.6116	0.0111	NIST SRM 1867
	$\beta$	1.6350	1.6273	1.6241	0.0109	
	$\gamma$	1.6449	1.6362	1.6326	0.0123	

1.  $[n_F - n_C]$  is the **only** parameter used in calculating all conversion tables. When changes in elemental composition, thermal history, etc. have caused variations in  $n_F$ ,  $n_D$ , and  $n_C$ , the dispersion coefficient  $[n_F - n_C]$  remains relatively unaffected or only slightly affected.
2. The dispersion coefficient of NIST SRM 1866 grunerite is much higher than that of the grunerite in McCrone (1987, Figs. 104A and 104B). Therefore, some values in Tables 6A and 6B, which are based on NIST grunerite, are markedly different from the values in McCrone (1989, p.51, Table I), which are based on the grunerite in Figs. 104A and 104B (McCrone, 1987).
3. For tremolite, actinolite and anthophyllite,  $n_C$  is close to  $\alpha$  and  $n_2$  to  $\gamma$ .

**Table 4A. Chrysotile  $\alpha$  (In Cargille Series E: 1.550)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.583	1.582	1.581	1.580	1.579	1.578	1.577
420	1.577	1.576	1.575	1.574	1.573	1.572	1.571
440	1.573	1.572	1.571	1.570	1.569	1.568	1.567
460	1.569	1.568	1.567	1.566	1.565	1.564	1.563
480	1.565	1.564	1.563	1.562	1.561	1.560	1.559
500	1.562	1.561	1.560	1.559	1.558	1.557	1.556
520	1.560	1.559	1.558	1.557	1.556	1.555	1.554
540	1.558	1.557	1.556	1.555	1.554	1.553	1.552
560	1.556	1.555	1.554	1.553	1.552	1.551	1.550
580	1.554	1.553	1.552	1.551	1.550	1.549	1.548
589	1.553	1.552	1.551	1.550	1.549	1.548	1.547
600	1.552	1.551	1.550	1.549	1.548	1.547	1.546
620	1.551	1.550	1.549	1.548	1.547	1.546	1.545
640	1.549	1.548	1.547	1.546	1.545	1.544	1.543
660	1.548	1.547	1.546	1.545	1.544	1.543	1.542
680	1.547	1.546	1.545	1.544	1.543	1.542	1.541
700	1.546	1.545	1.544	1.543	1.542	1.541	1.540
750	1.544	1.543	1.542	1.541	1.540	1.539	1.538
800	1.542	1.541	1.540	1.539	1.538	1.537	1.536

**Table 4B. Chrysotile  $\gamma$  (In Cargille Series E: 1.550)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.581	1.580	1.579	1.578	1.577	1.576	1.575
420	1.575	1.574	1.573	1.572	1.571	1.570	1.569
440	1.571	1.570	1.569	1.568	1.567	1.566	1.565
460	1.567	1.566	1.565	1.565	1.564	1.563	1.562
480	1.564	1.563	1.562	1.561	1.560	1.559	1.558
500	1.562	1.561	1.560	1.559	1.558	1.557	1.556
520	1.559	1.558	1.557	1.556	1.555	1.554	1.553
540	1.557	1.556	1.555	1.554	1.553	1.552	1.551
560	1.555	1.554	1.553	1.552	1.551	1.550	1.549
580	1.554	1.553	1.552	1.551	1.550	1.549	1.548
589	1.553	1.552	1.551	1.550	1.549	1.548	1.547
600	1.552	1.551	1.550	1.549	1.548	1.547	1.546
620	1.551	1.550	1.549	1.548	1.547	1.546	1.545
640	1.550	1.549	1.548	1.547	1.546	1.545	1.544
660	1.548	1.547	1.546	1.546	1.545	1.544	1.543
680	1.547	1.546	1.545	1.544	1.544	1.543	1.542
700	1.546	1.546	1.545	1.544	1.543	1.542	1.541
750	1.544	1.543	1.542	1.541	1.540	1.540	1.539
800	1.543	1.542	1.541	1.540	1.539	1.538	1.537

Table 5A. **Chrysotile  $\alpha$**  (In Cargille Series E: **1.560**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.592	1.591	1.590	1.589	1.588	1.587	1.586
420	1.586	1.585	1.584	1.584	1.583	1.582	1.581
440	1.582	1.581	1.580	1.579	1.578	1.577	1.576
460	1.578	1.577	1.576	1.575	1.574	1.573	1.572
480	1.575	1.574	1.573	1.572	1.571	1.570	1.569
500	1.572	1.571	1.570	1.569	1.568	1.567	1.566
520	1.569	1.569	1.568	1.567	1.566	1.565	1.564
540	1.567	1.566	1.565	1.564	1.563	1.563	1.562
560	1.565	1.564	1.563	1.562	1.562	1.561	1.560
580	1.564	1.563	1.562	1.561	1.560	1.559	1.558
589	1.563	1.562	1.561	1.560	1.559	1.558	1.557
600	1.562	1.561	1.560	1.559	1.558	1.557	1.556
620	1.561	1.560	1.559	1.558	1.557	1.556	1.555
640	1.559	1.558	1.557	1.556	1.556	1.555	1.554
660	1.558	1.557	1.556	1.555	1.554	1.553	1.552
680	1.557	1.556	1.555	1.554	1.553	1.552	1.551
700	1.556	1.555	1.554	1.553	1.552	1.551	1.550
750	1.554	1.553	1.552	1.551	1.550	1.549	1.548
800	1.552	1.551	1.550	1.549	1.548	1.547	1.546

Table 5B. **Chrysotile  $\gamma$**  (In Cargille Series E: **1.560**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.590	1.589	1.588	1.587	1.586	1.585	1.584
420	1.585	1.584	1.583	1.582	1.581	1.580	1.579
440	1.580	1.579	1.578	1.578	1.577	1.576	1.575
460	1.577	1.576	1.575	1.574	1.573	1.572	1.571
480	1.574	1.573	1.572	1.571	1.570	1.569	1.568
500	1.571	1.570	1.569	1.568	1.567	1.566	1.566
520	1.569	1.568	1.567	1.566	1.565	1.564	1.563
540	1.567	1.566	1.565	1.564	1.563	1.562	1.561
560	1.565	1.564	1.563	1.562	1.561	1.560	1.559
580	1.564	1.563	1.562	1.561	1.560	1.559	1.558
589	1.563	1.562	1.561	1.560	1.559	1.558	1.557
600	1.562	1.561	1.560	1.559	1.558	1.557	1.556
620	1.561	1.560	1.559	1.558	1.557	1.556	1.555
640	1.560	1.559	1.558	1.557	1.556	1.555	1.554
660	1.559	1.558	1.557	1.556	1.555	1.554	1.553
680	1.558	1.557	1.556	1.555	1.554	1.553	1.552
700	1.557	1.556	1.555	1.554	1.553	1.552	1.551
750	1.555	1.554	1.553	1.552	1.551	1.550	1.549
800	1.553	1.552	1.551	1.550	1.549	1.548	1.547

Table 6A. **Amosite  $\alpha$**  (In Cargille Series **B: 1.680**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.711	1.710	1.709	1.708	1.707	1.706	1.705
420	1.706	1.705	1.704	1.703	1.702	1.701	1.700
440	1.701	1.700	1.699	1.699	1.698	1.697	1.696
460	1.698	1.697	1.696	1.695	1.694	1.693	1.692
480	1.694	1.694	1.693	1.692	1.691	1.690	1.689
500	1.692	1.691	1.690	1.689	1.688	1.687	1.686
520	1.689	1.688	1.687	1.686	1.685	1.685	1.684
540	1.687	1.686	1.685	1.684	1.683	1.682	1.681
560	1.685	1.684	1.683	1.682	1.681	1.681	1.680
580	1.684	1.683	1.682	1.681	1.680	1.679	1.678
589	1.683	1.682	1.681	1.680	1.679	1.678	1.677
600	1.682	1.681	1.680	1.679	1.678	1.677	1.676
620	1.681	1.680	1.679	1.678	1.677	1.676	1.675
640	1.679	1.678	1.678	1.677	1.676	1.675	1.674
660	1.678	1.677	1.676	1.675	1.674	1.674	1.673
680	1.677	1.676	1.675	1.674	1.673	1.672	1.671
700	1.676	1.675	1.674	1.673	1.672	1.671	1.671
750	1.674	1.673	1.672	1.671	1.670	1.669	1.668
800	1.672	1.671	1.671	1.670	1.669	1.668	1.667

Table 6B. **Amosite  $\gamma$**  (In Cargille Series **B: 1.680**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.710	1.709	1.708	1.707	1.706	1.705	1.704
420	1.705	1.704	1.703	1.702	1.701	1.700	1.699
440	1.700	1.699	1.698	1.698	1.697	1.696	1.695
460	1.697	1.696	1.695	1.694	1.693	1.692	1.691
480	1.694	1.693	1.692	1.691	1.690	1.689	1.688
500	1.691	1.690	1.689	1.688	1.687	1.686	1.686
520	1.689	1.688	1.687	1.686	1.685	1.684	1.683
540	1.687	1.686	1.685	1.684	1.683	1.682	1.681
560	1.685	1.684	1.683	1.682	1.681	1.680	1.679
580	1.684	1.683	1.682	1.681	1.680	1.679	1.678
589	1.683	1.682	1.681	1.680	1.679	1.678	1.677
600	1.682	1.681	1.680	1.679	1.678	1.677	1.676
620	1.681	1.680	1.679	1.678	1.677	1.676	1.675
640	1.680	1.679	1.678	1.677	1.676	1.675	1.674
660	1.679	1.678	1.677	1.676	1.675	1.674	1.673
680	1.678	1.677	1.676	1.675	1.674	1.673	1.672
700	1.677	1.676	1.675	1.674	1.673	1.672	1.671
750	1.675	1.674	1.673	1.672	1.671	1.670	1.669
800	1.673	1.672	1.671	1.670	1.669	1.668	1.667

Table 7A. **Amosite  $\alpha$**  (In Cargille Series **B: 1.700**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.735	1.734	1.733	1.732	1.731	1.730	1.729
420	1.729	1.728	1.727	1.726	1.725	1.724	1.723
440	1.724	1.723	1.722	1.721	1.720	1.719	1.718
460	1.720	1.719	1.718	1.717	1.716	1.715	1.714
480	1.716	1.715	1.714	1.713	1.712	1.711	1.710
500	1.713	1.712	1.711	1.710	1.709	1.708	1.707
520	1.710	1.709	1.708	1.707	1.706	1.705	1.705
540	1.708	1.707	1.706	1.705	1.704	1.703	1.702
560	1.706	1.705	1.704	1.703	1.702	1.701	1.700
580	1.704	1.703	1.702	1.701	1.700	1.699	1.698
589	1.703	1.702	1.701	1.700	1.699	1.698	1.697
600	1.702	1.701	1.700	1.699	1.698	1.697	1.696
620	1.700	1.699	1.698	1.698	1.697	1.696	1.695
640	1.699	1.698	1.697	1.696	1.695	1.694	1.693
660	1.698	1.697	1.696	1.695	1.694	1.693	1.692
680	1.696	1.695	1.695	1.694	1.693	1.692	1.691
700	1.695	1.694	1.693	1.692	1.691	1.691	1.690
750	1.693	1.692	1.691	1.690	1.689	1.688	1.687
800	1.691	1.690	1.689	1.688	1.687	1.686	1.685

Table 7B. **Amosite  $\gamma$**  (In Cargille Series **B: 1.700**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.734	1.733	1.732	1.731	1.730	1.729	1.728
420	1.728	1.727	1.726	1.725	1.724	1.723	1.722
440	1.723	1.722	1.721	1.720	1.719	1.718	1.717
460	1.719	1.718	1.717	1.716	1.715	1.714	1.713
480	1.716	1.715	1.714	1.713	1.712	1.711	1.710
500	1.713	1.712	1.711	1.710	1.709	1.708	1.707
520	1.710	1.709	1.708	1.707	1.706	1.705	1.704
540	1.708	1.707	1.706	1.705	1.704	1.703	1.702
560	1.706	1.705	1.704	1.703	1.702	1.701	1.700
580	1.704	1.703	1.702	1.701	1.700	1.699	1.698
589	1.703	1.702	1.701	1.700	1.699	1.698	1.697
600	1.702	1.701	1.700	1.699	1.698	1.697	1.696
620	1.701	1.700	1.699	1.698	1.697	1.696	1.695
640	1.699	1.698	1.697	1.696	1.695	1.694	1.693
660	1.698	1.697	1.696	1.695	1.694	1.693	1.692
680	1.697	1.696	1.695	1.694	1.693	1.692	1.691
700	1.696	1.695	1.694	1.693	1.692	1.691	1.690
750	1.693	1.692	1.691	1.691	1.690	1.689	1.688
800	1.691	1.691	1.690	1.689	1.688	1.687	1.686

**Table 8A. Crocidolite  $\alpha$  (In Cargille Series B: 1.680)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.715	1.714	1.713	1.712	1.711	1.711	1.710
420	1.709	1.708	1.707	1.706	1.705	1.704	1.704
440	1.704	1.703	1.702	1.701	1.700	1.699	1.698
460	1.700	1.699	1.698	1.697	1.696	1.695	1.694
480	1.696	1.695	1.694	1.693	1.692	1.691	1.691
500	1.693	1.692	1.691	1.690	1.689	1.688	1.687
520	1.690	1.689	1.688	1.687	1.686	1.686	1.685
540	1.688	1.687	1.686	1.685	1.684	1.683	1.682
560	1.686	1.685	1.684	1.683	1.682	1.681	1.680
580	1.684	1.683	1.682	1.681	1.680	1.679	1.678
589	1.683	1.682	1.681	1.680	1.679	1.678	1.677
600	1.682	1.681	1.680	1.679	1.678	1.677	1.676
620	1.680	1.679	1.678	1.677	1.677	1.676	1.675
640	1.679	1.678	1.677	1.676	1.675	1.674	1.673
660	1.678	1.677	1.676	1.675	1.674	1.673	1.672
680	1.676	1.675	1.674	1.674	1.673	1.672	1.671
700	1.675	1.674	1.673	1.672	1.671	1.670	1.670
750	1.673	1.672	1.671	1.670	1.669	1.668	1.667
800	1.671	1.670	1.669	1.668	1.667	1.666	1.665

**Table 8B. Crocidolite  $\gamma$  (In Cargille Series B: 1.680)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.718	1.717	1.716	1.715	1.714	1.713	1.712
420	1.711	1.710	1.709	1.708	1.707	1.706	1.705
440	1.706	1.705	1.704	1.703	1.702	1.701	1.700
460	1.701	1.700	1.699	1.698	1.697	1.696	1.695
480	1.697	1.696	1.695	1.694	1.693	1.692	1.692
500	1.694	1.693	1.692	1.691	1.690	1.689	1.688
520	1.691	1.690	1.689	1.688	1.687	1.686	1.685
540	1.688	1.687	1.686	1.685	1.684	1.683	1.682
560	1.686	1.685	1.684	1.683	1.682	1.681	1.680
580	1.684	1.683	1.682	1.681	1.680	1.679	1.678
589	1.683	1.682	1.681	1.680	1.679	1.678	1.677
600	1.682	1.681	1.680	1.679	1.678	1.677	1.676
620	1.680	1.679	1.678	1.677	1.676	1.675	1.674
640	1.679	1.678	1.677	1.676	1.675	1.674	1.673
660	1.677	1.676	1.675	1.674	1.673	1.672	1.671
680	1.676	1.675	1.674	1.673	1.672	1.671	1.670
700	1.675	1.674	1.673	1.672	1.671	1.670	1.669
750	1.672	1.671	1.670	1.669	1.668	1.667	1.666
800	1.670	1.669	1.668	1.667	1.666	1.665	1.664

**Table 9A. Crocidolite  $\alpha$  (In Cargille Series B: 1.700)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.739	1.738	1.738	1.737	1.736	1.735	1.734
420	1.733	1.732	1.731	1.730	1.729	1.728	1.727
440	1.727	1.726	1.725	1.724	1.723	1.722	1.721
460	1.722	1.721	1.720	1.719	1.718	1.717	1.716
480	1.718	1.717	1.716	1.715	1.714	1.713	1.712
500	1.714	1.713	1.712	1.711	1.711	1.710	1.709
520	1.711	1.710	1.709	1.708	1.707	1.706	1.705
540	1.708	1.708	1.707	1.706	1.705	1.704	1.703
560	1.706	1.705	1.704	1.703	1.702	1.701	1.700
580	1.704	1.703	1.702	1.701	1.700	1.699	1.698
589	1.703	1.702	1.701	1.700	1.699	1.698	1.697
600	1.702	1.701	1.700	1.699	1.698	1.697	1.696
620	1.700	1.699	1.698	1.697	1.696	1.695	1.694
640	1.698	1.697	1.697	1.696	1.695	1.694	1.693
660	1.697	1.696	1.695	1.694	1.693	1.692	1.691
680	1.696	1.695	1.694	1.693	1.692	1.691	1.690
700	1.694	1.693	1.692	1.691	1.690	1.690	1.689
750	1.692	1.691	1.690	1.689	1.688	1.687	1.686
800	1.689	1.688	1.687	1.686	1.685	1.685	1.684

**Table 9B. Crocidolite  $\gamma$  (In Cargille Series B: 1.700)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.742	1.741	1.740	1.739	1.738	1.737	1.736
420	1.735	1.734	1.733	1.732	1.731	1.730	1.729
440	1.728	1.728	1.727	1.726	1.725	1.724	1.723
460	1.723	1.722	1.721	1.720	1.720	1.719	1.718
480	1.719	1.718	1.717	1.716	1.715	1.714	1.713
500	1.715	1.714	1.713	1.712	1.711	1.710	1.709
520	1.712	1.711	1.710	1.709	1.708	1.707	1.706
540	1.709	1.708	1.707	1.706	1.705	1.704	1.703
560	1.706	1.705	1.704	1.703	1.702	1.701	1.700
580	1.704	1.703	1.702	1.701	1.700	1.699	1.698
589	1.703	1.702	1.701	1.700	1.699	1.698	1.697
600	1.702	1.701	1.700	1.699	1.698	1.697	1.696
620	1.700	1.699	1.698	1.697	1.696	1.695	1.694
640	1.698	1.697	1.696	1.695	1.694	1.693	1.692
660	1.697	1.696	1.695	1.694	1.693	1.692	1.691
680	1.695	1.694	1.693	1.692	1.691	1.690	1.689
700	1.694	1.693	1.692	1.691	1.690	1.689	1.688
750	1.691	1.690	1.689	1.688	1.687	1.686	1.685
800	1.688	1.687	1.686	1.686	1.685	1.684	1.683



Table 10A. **Crocidolite  $\alpha$**  (In Cargille Series **M: 1.705**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.741	1.740	1.738	1.737	1.736	1.735	1.734
420	1.735	1.734	1.732	1.731	1.730	1.729	1.728
440	1.730	1.729	1.727	1.726	1.725	1.724	1.723
460	1.726	1.724	1.723	1.722	1.721	1.720	1.718
480	1.722	1.721	1.720	1.718	1.717	1.716	1.715
500	1.719	1.718	1.716	1.715	1.714	1.713	1.712
520	1.716	1.715	1.714	1.712	1.711	1.710	1.709
540	1.714	1.712	1.711	1.710	1.709	1.708	1.706
560	1.711	1.710	1.709	1.708	1.707	1.705	1.704
580	1.709	1.708	1.707	1.706	1.705	1.703	1.702
589	1.709	1.707	1.706	1.705	1.704	1.703	1.701
600	1.708	1.706	1.705	1.704	1.703	1.702	1.700
620	1.706	1.705	1.704	1.703	1.701	1.700	1.699
640	1.705	1.703	1.702	1.701	1.700	1.699	1.697
660	1.703	1.702	1.701	1.700	1.699	1.697	1.696
680	1.702	1.701	1.700	1.699	1.697	1.696	1.695
700	1.701	1.700	1.699	1.697	1.696	1.695	1.694
750	1.699	1.697	1.696	1.695	1.694	1.693	1.691
800	1.697	1.695	1.694	1.693	1.692	1.691	1.689

Table 10B. **Crocidolite  $\gamma$**  (In Cargille Series **M: 1.705**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.738	1.737	1.736	1.735	1.734	1.732	1.731
420	1.733	1.732	1.730	1.729	1.728	1.727	1.726
440	1.728	1.727	1.726	1.725	1.723	1.722	1.721
460	1.724	1.723	1.722	1.721	1.719	1.718	1.717
480	1.721	1.720	1.719	1.717	1.716	1.715	1.714
500	1.718	1.717	1.716	1.714	1.713	1.712	1.711
520	1.715	1.714	1.713	1.712	1.711	1.709	1.708
540	1.713	1.712	1.711	1.710	1.708	1.707	1.706
560	1.711	1.710	1.709	1.708	1.706	1.705	1.704
580	1.709	1.708	1.707	1.706	1.705	1.703	1.702
589	1.709	1.707	1.706	1.705	1.704	1.703	1.701
600	1.708	1.707	1.705	1.704	1.703	1.702	1.701
620	1.706	1.705	1.704	1.703	1.701	1.700	1.699
640	1.705	1.704	1.703	1.701	1.700	1.699	1.698
660	1.704	1.703	1.701	1.700	1.699	1.698	1.697
680	1.703	1.701	1.700	1.699	1.698	1.697	1.695
700	1.702	1.700	1.699	1.698	1.697	1.696	1.694
750	1.699	1.698	1.697	1.696	1.695	1.693	1.692
800	1.698	1.696	1.695	1.694	1.693	1.692	1.690

Table 11A. **Crocidolite  $\alpha$**  (In Cargille Series **M: 1.710**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.747	1.746	1.744	1.743	1.742	1.741	1.740
420	1.741	1.739	1.738	1.737	1.736	1.735	1.733
440	1.735	1.734	1.733	1.732	1.731	1.729	1.728
460	1.731	1.730	1.729	1.727	1.726	1.725	1.724
480	1.727	1.726	1.725	1.724	1.722	1.721	1.720
500	1.724	1.723	1.722	1.720	1.719	1.718	1.717
520	1.721	1.720	1.719	1.718	1.716	1.715	1.714
540	1.719	1.718	1.716	1.715	1.714	1.713	1.711
560	1.717	1.715	1.714	1.713	1.712	1.710	1.709
580	1.715	1.713	1.712	1.711	1.710	1.708	1.707
589	1.714	1.712	1.711	1.710	1.709	1.708	1.706
600	1.713	1.712	1.710	1.709	1.708	1.707	1.705
620	1.711	1.710	1.709	1.707	1.706	1.705	1.704
640	1.710	1.708	1.707	1.706	1.705	1.704	1.702
660	1.708	1.707	1.706	1.705	1.703	1.702	1.701
680	1.707	1.706	1.705	1.703	1.702	1.701	1.700
700	1.706	1.705	1.703	1.702	1.701	1.700	1.699
750	1.703	1.702	1.701	1.700	1.699	1.697	1.696
800	1.701	1.700	1.699	1.698	1.696	1.695	1.694

Table 11B. **Crocidolite  $\gamma$**  (In Cargille Series **M: 1.710**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.744	1.743	1.742	1.741	1.740	1.738	1.737
420	1.739	1.737	1.736	1.735	1.734	1.733	1.731
440	1.734	1.733	1.731	1.730	1.729	1.728	1.727
460	1.730	1.729	1.727	1.726	1.725	1.724	1.723
480	1.726	1.725	1.724	1.723	1.721	1.720	1.719
500	1.723	1.722	1.721	1.720	1.718	1.717	1.716
520	1.721	1.719	1.718	1.717	1.716	1.715	1.713
540	1.718	1.717	1.716	1.715	1.713	1.712	1.711
560	1.716	1.715	1.714	1.713	1.711	1.710	1.709
580	1.714	1.713	1.712	1.711	1.710	1.708	1.707
589	1.714	1.712	1.711	1.710	1.709	1.708	1.706
600	1.713	1.712	1.710	1.709	1.708	1.707	1.705
620	1.711	1.710	1.709	1.708	1.706	1.705	1.704
640	1.710	1.709	1.707	1.706	1.705	1.704	1.703
660	1.709	1.707	1.706	1.705	1.704	1.703	1.701
680	1.708	1.706	1.705	1.704	1.703	1.701	1.700
700	1.706	1.705	1.704	1.703	1.702	1.700	1.699
750	1.704	1.703	1.702	1.701	1.699	1.698	1.697
800	1.702	1.701	1.700	1.699	1.697	1.696	1.695

Table 12A. **Tremolite  $\alpha$**  (In Cargille Series E: **1.605**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.636	1.635	1.634	1.633	1.632	1.631	1.631
420	1.631	1.630	1.629	1.628	1.627	1.626	1.625
440	1.626	1.625	1.624	1.624	1.623	1.622	1.621
460	1.622	1.622	1.621	1.620	1.619	1.618	1.617
480	1.619	1.618	1.617	1.617	1.616	1.615	1.614
500	1.616	1.616	1.615	1.614	1.613	1.612	1.611
520	1.614	1.613	1.612	1.611	1.611	1.610	1.609
540	1.612	1.611	1.610	1.609	1.608	1.608	1.607
560	1.610	1.609	1.608	1.607	1.607	1.606	1.605
580	1.608	1.607	1.607	1.606	1.605	1.604	1.603
589	1.608	1.607	1.606	1.605	1.604	1.603	1.602
600	1.607	1.606	1.605	1.604	1.603	1.602	1.602
620	1.605	1.605	1.604	1.603	1.602	1.601	1.600
640	1.604	1.603	1.602	1.602	1.601	1.600	1.599
660	1.603	1.602	1.601	1.600	1.600	1.599	1.598
680	1.602	1.601	1.600	1.599	1.598	1.598	1.597
700	1.601	1.600	1.599	1.598	1.598	1.597	1.596
750	1.599	1.598	1.597	1.596	1.595	1.595	1.594
800	1.597	1.596	1.595	1.595	1.594	1.593	1.592

Table 12B. **Tremolite  $\gamma$**  (In Cargille Series E: **1.605**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.632	1.631	1.630	1.629	1.628	1.627	1.627
420	1.627	1.626	1.626	1.625	1.624	1.623	1.622
440	1.624	1.623	1.622	1.621	1.620	1.619	1.618
460	1.620	1.619	1.619	1.618	1.617	1.616	1.615
480	1.618	1.617	1.616	1.615	1.614	1.613	1.612
500	1.615	1.614	1.614	1.613	1.612	1.611	1.610
520	1.613	1.612	1.611	1.611	1.610	1.609	1.608
540	1.611	1.610	1.610	1.609	1.608	1.607	1.606
560	1.610	1.609	1.608	1.607	1.606	1.605	1.604
580	1.608	1.607	1.607	1.606	1.605	1.604	1.603
589	1.608	1.607	1.606	1.605	1.604	1.603	1.602
600	1.607	1.606	1.605	1.604	1.603	1.603	1.602
620	1.606	1.605	1.604	1.603	1.602	1.601	1.600
640	1.605	1.604	1.603	1.602	1.601	1.600	1.599
660	1.604	1.603	1.602	1.601	1.600	1.599	1.598
680	1.603	1.602	1.601	1.600	1.599	1.598	1.598
700	1.602	1.601	1.600	1.599	1.598	1.598	1.597
750	1.600	1.599	1.598	1.598	1.597	1.596	1.595
800	1.599	1.598	1.597	1.596	1.595	1.594	1.593

Table 13A. **Tremolite  $\alpha$**  (In Cargille Series E: **1.615**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.649	1.648	1.647	1.646	1.645	1.644	1.643
420	1.643	1.642	1.641	1.640	1.639	1.639	1.638
440	1.638	1.637	1.636	1.635	1.635	1.634	1.633
460	1.634	1.633	1.632	1.631	1.630	1.630	1.629
480	1.631	1.630	1.629	1.628	1.627	1.626	1.625
500	1.627	1.627	1.626	1.625	1.624	1.623	1.622
520	1.625	1.624	1.623	1.622	1.621	1.620	1.619
540	1.622	1.622	1.621	1.620	1.619	1.618	1.617
560	1.620	1.619	1.619	1.618	1.617	1.616	1.615
580	1.619	1.618	1.617	1.616	1.615	1.614	1.613
589	1.618	1.617	1.616	1.615	1.614	1.613	1.612
600	1.617	1.616	1.615	1.614	1.613	1.612	1.611
620	1.615	1.614	1.613	1.613	1.612	1.611	1.610
640	1.614	1.613	1.612	1.611	1.610	1.609	1.609
660	1.613	1.612	1.611	1.610	1.609	1.608	1.607
680	1.611	1.611	1.610	1.609	1.608	1.607	1.606
700	1.610	1.610	1.609	1.608	1.607	1.606	1.605
750	1.608	1.607	1.606	1.605	1.604	1.604	1.603
800	1.606	1.605	1.604	1.603	1.603	1.602	1.601

Table 13B. **Tremolite  $\gamma$**  (In Cargille Series E: **1.615**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.645	1.644	1.643	1.642	1.641	1.640	1.640
420	1.640	1.639	1.638	1.637	1.636	1.635	1.634
440	1.636	1.635	1.634	1.633	1.632	1.631	1.630
460	1.632	1.631	1.630	1.629	1.628	1.628	1.627
480	1.629	1.628	1.627	1.626	1.625	1.624	1.624
500	1.626	1.625	1.624	1.624	1.623	1.622	1.621
520	1.624	1.623	1.622	1.621	1.620	1.619	1.619
540	1.622	1.621	1.620	1.619	1.618	1.617	1.616
560	1.620	1.619	1.618	1.617	1.616	1.616	1.615
580	1.618	1.618	1.617	1.616	1.615	1.614	1.613
589	1.618	1.617	1.616	1.615	1.614	1.613	1.612
600	1.617	1.616	1.615	1.614	1.613	1.612	1.612
620	1.616	1.615	1.614	1.613	1.612	1.611	1.610
640	1.614	1.613	1.613	1.612	1.611	1.610	1.609
660	1.613	1.612	1.611	1.611	1.610	1.609	1.608
680	1.612	1.611	1.610	1.610	1.609	1.608	1.607
700	1.611	1.610	1.610	1.609	1.608	1.607	1.606
750	1.609	1.608	1.607	1.607	1.606	1.605	1.604
800	1.608	1.607	1.606	1.605	1.604	1.603	1.602

Table 14A. **Tremolite  $\alpha$**  (In Cargille Series E: **1.620**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.653	1.652	1.651	1.650	1.649	1.648	1.647
420	1.647	1.646	1.645	1.644	1.644	1.643	1.642
440	1.642	1.642	1.641	1.640	1.639	1.638	1.637
460	1.638	1.638	1.637	1.636	1.635	1.634	1.633
480	1.635	1.634	1.633	1.632	1.631	1.631	1.630
500	1.632	1.631	1.630	1.629	1.629	1.628	1.627
520	1.630	1.629	1.628	1.627	1.626	1.625	1.624
540	1.627	1.626	1.625	1.625	1.624	1.623	1.622
560	1.625	1.624	1.623	1.623	1.622	1.621	1.620
580	1.623	1.623	1.622	1.621	1.620	1.619	1.618
589	1.623	1.622	1.621	1.620	1.619	1.618	1.617
600	1.622	1.621	1.620	1.619	1.618	1.617	1.616
620	1.620	1.619	1.619	1.618	1.617	1.616	1.615
640	1.619	1.618	1.617	1.616	1.615	1.615	1.614
660	1.618	1.617	1.616	1.615	1.614	1.613	1.612
680	1.617	1.616	1.615	1.614	1.613	1.612	1.611
700	1.616	1.615	1.614	1.613	1.612	1.611	1.610
750	1.613	1.613	1.612	1.611	1.610	1.609	1.608
800	1.612	1.611	1.610	1.609	1.608	1.607	1.606

Table 14B. **Tremolite  $\gamma$**  (In Cargille Series E: **1.620**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.648	1.647	1.646	1.646	1.645	1.644	1.643
420	1.643	1.643	1.642	1.641	1.640	1.639	1.638
440	1.639	1.639	1.638	1.637	1.636	1.635	1.634
460	1.636	1.635	1.634	1.633	1.633	1.632	1.631
480	1.633	1.632	1.631	1.631	1.630	1.629	1.628
500	1.631	1.630	1.629	1.628	1.627	1.626	1.625
520	1.629	1.628	1.627	1.626	1.625	1.624	1.623
540	1.627	1.626	1.625	1.624	1.623	1.622	1.621
560	1.625	1.624	1.623	1.622	1.621	1.620	1.619
580	1.623	1.622	1.622	1.621	1.620	1.619	1.618
589	1.623	1.622	1.621	1.620	1.619	1.618	1.617
600	1.622	1.621	1.620	1.619	1.618	1.617	1.617
620	1.621	1.620	1.619	1.618	1.617	1.616	1.615
640	1.620	1.619	1.618	1.617	1.616	1.615	1.614
660	1.619	1.618	1.617	1.616	1.615	1.614	1.613
680	1.618	1.617	1.616	1.615	1.614	1.613	1.612
700	1.617	1.616	1.615	1.614	1.613	1.612	1.611
750	1.615	1.614	1.613	1.612	1.611	1.610	1.609
800	1.613	1.612	1.611	1.611	1.610	1.609	1.608

Table 15A. **Tremolite  $\alpha$**  (In Cargille Series E: **1.625**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.662	1.661	1.660	1.659	1.658	1.657	1.656
420	1.655	1.655	1.654	1.653	1.652	1.651	1.650
440	1.650	1.649	1.648	1.647	1.647	1.646	1.645
460	1.646	1.645	1.644	1.643	1.642	1.641	1.640
480	1.642	1.641	1.640	1.639	1.638	1.637	1.636
500	1.638	1.638	1.637	1.636	1.635	1.634	1.633
520	1.636	1.635	1.634	1.633	1.632	1.631	1.630
540	1.633	1.632	1.631	1.630	1.629	1.628	1.628
560	1.631	1.630	1.629	1.628	1.627	1.626	1.625
580	1.629	1.628	1.627	1.626	1.625	1.624	1.623
589	1.628	1.627	1.626	1.625	1.624	1.623	1.622
600	1.627	1.626	1.625	1.624	1.623	1.622	1.621
620	1.625	1.624	1.623	1.622	1.621	1.621	1.620
640	1.624	1.623	1.622	1.621	1.620	1.619	1.618
660	1.622	1.621	1.620	1.619	1.619	1.618	1.617
680	1.621	1.620	1.619	1.618	1.617	1.616	1.615
700	1.620	1.619	1.618	1.617	1.616	1.615	1.614
750	1.617	1.616	1.615	1.614	1.614	1.613	1.612
800	1.615	1.614	1.613	1.612	1.611	1.611	1.610

Table 15B. **Tremolite  $\gamma$**  (In Cargille Series E: **1.625**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.658	1.657	1.656	1.655	1.654	1.653	1.653
420	1.652	1.651	1.650	1.650	1.649	1.648	1.647
440	1.648	1.647	1.646	1.645	1.644	1.643	1.642
460	1.644	1.643	1.642	1.641	1.640	1.639	1.638
480	1.640	1.639	1.638	1.637	1.637	1.636	1.635
500	1.637	1.636	1.635	1.635	1.634	1.633	1.632
520	1.635	1.634	1.633	1.632	1.631	1.630	1.629
540	1.632	1.631	1.631	1.630	1.629	1.628	1.627
560	1.630	1.629	1.628	1.628	1.627	1.626	1.625
580	1.628	1.628	1.627	1.626	1.625	1.624	1.623
589	1.628	1.627	1.626	1.625	1.624	1.623	1.622
600	1.627	1.626	1.625	1.624	1.623	1.622	1.621
620	1.625	1.624	1.624	1.623	1.622	1.621	1.620
640	1.624	1.623	1.622	1.621	1.620	1.620	1.619
660	1.623	1.622	1.621	1.620	1.619	1.618	1.617
680	1.622	1.621	1.620	1.619	1.618	1.617	1.616
700	1.621	1.620	1.619	1.618	1.617	1.616	1.615
750	1.618	1.617	1.617	1.616	1.615	1.614	1.613
800	1.616	1.616	1.615	1.614	1.613	1.612	1.611

**Table 16A. Tremolite  $\alpha$  (In Cargille Series E: 1.635)**

$\lambda_0$	119°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.675	1.674	1.673	1.672	1.671	1.670	1.669
420	1.668	1.667	1.666	1.665	1.664	1.663	1.662
440	1.662	1.661	1.660	1.659	1.658	1.658	1.657
460	1.657	1.656	1.655	1.654	1.654	1.653	1.652
480	1.653	1.652	1.651	1.650	1.649	1.648	1.648
500	1.649	1.649	1.648	1.647	1.646	1.645	1.644
520	1.646	1.645	1.644	1.643	1.643	1.642	1.641
540	1.643	1.643	1.642	1.641	1.640	1.639	1.638
560	1.641	1.640	1.639	1.638	1.637	1.636	1.635
580	1.639	1.638	1.637	1.636	1.635	1.634	1.633
589	1.638	1.637	1.636	1.635	1.634	1.633	1.632
600	1.637	1.636	1.635	1.634	1.633	1.632	1.631
620	1.635	1.634	1.633	1.632	1.631	1.630	1.629
640	1.633	1.632	1.631	1.630	1.630	1.629	1.628
660	1.632	1.631	1.630	1.629	1.628	1.627	1.626
680	1.630	1.629	1.629	1.628	1.627	1.626	1.625
700	1.629	1.628	1.627	1.626	1.625	1.624	1.624
750	1.626	1.625	1.624	1.624	1.623	1.622	1.621
800	1.624	1.623	1.622	1.621	1.620	1.619	1.618

**Table 16B. Tremolite  $\gamma$  (In Cargille Series E: 1.635)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.671	1.670	1.669	1.668	1.667	1.666	1.665
420	1.665	1.664	1.663	1.662	1.661	1.660	1.659
440	1.660	1.659	1.658	1.657	1.656	1.655	1.654
460	1.655	1.654	1.653	1.652	1.652	1.651	1.650
480	1.651	1.651	1.650	1.649	1.648	1.647	1.646
500	1.648	1.647	1.646	1.645	1.645	1.644	1.643
520	1.645	1.644	1.644	1.643	1.642	1.641	1.640
540	1.643	1.642	1.641	1.640	1.639	1.638	1.637
560	1.641	1.640	1.639	1.638	1.637	1.636	1.635
580	1.639	1.638	1.637	1.636	1.635	1.634	1.633
589	1.638	1.637	1.636	1.635	1.634	1.633	1.632
600	1.637	1.636	1.635	1.634	1.633	1.632	1.631
620	1.635	1.634	1.633	1.632	1.632	1.631	1.630
640	1.634	1.633	1.632	1.631	1.630	1.629	1.628
660	1.632	1.631	1.631	1.630	1.629	1.628	1.627
680	1.631	1.630	1.629	1.628	1.627	1.627	1.626
700	1.630	1.629	1.628	1.627	1.626	1.625	1.624
750	1.628	1.627	1.626	1.625	1.624	1.623	1.622
800	1.625	1.625	1.624	1.623	1.622	1.621	1.620

Table 17A. **Actinolite  $\alpha$**  (In Cargille Series E: **1.605**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.633	1.632	1.631	1.631	1.630	1.629	1.628
420	1.628	1.628	1.627	1.626	1.625	1.624	1.623
440	1.624	1.624	1.623	1.622	1.621	1.620	1.619
460	1.621	1.620	1.619	1.618	1.618	1.617	1.616
480	1.618	1.617	1.616	1.616	1.615	1.614	1.613
500	1.616	1.615	1.614	1.613	1.612	1.611	1.610
520	1.613	1.613	1.612	1.611	1.610	1.609	1.608
540	1.612	1.611	1.610	1.609	1.608	1.607	1.606
560	1.610	1.609	1.608	1.607	1.606	1.605	1.605
580	1.608	1.607	1.607	1.606	1.605	1.604	1.603
589	1.608	1.607	1.606	1.605	1.604	1.603	1.602
600	1.607	1.606	1.605	1.604	1.603	1.603	1.602
620	1.606	1.605	1.604	1.603	1.602	1.601	1.600
640	1.605	1.604	1.603	1.602	1.601	1.600	1.599
660	1.603	1.603	1.602	1.601	1.600	1.599	1.598
680	1.603	1.602	1.601	1.600	1.599	1.598	1.597
700	1.602	1.601	1.600	1.599	1.598	1.597	1.596
750	1.600	1.599	1.598	1.597	1.596	1.595	1.594
800	1.598	1.597	1.596	1.596	1.595	1.594	1.593

Table 17B. **Actinolite  $\gamma$**  (In Cargille Series E: **1.605**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.629	1.628	1.627	1.626	1.625	1.624	1.623
420	1.625	1.624	1.623	1.622	1.621	1.620	1.619
440	1.621	1.621	1.620	1.619	1.618	1.617	1.616
460	1.619	1.618	1.617	1.616	1.615	1.614	1.613
480	1.616	1.615	1.615	1.614	1.613	1.612	1.611
500	1.614	1.613	1.613	1.612	1.611	1.610	1.609
520	1.612	1.612	1.611	1.610	1.609	1.608	1.607
540	1.611	1.610	1.609	1.608	1.607	1.606	1.606
560	1.609	1.609	1.608	1.607	1.606	1.605	1.604
580	1.608	1.607	1.606	1.606	1.605	1.604	1.603
589	1.608	1.607	1.606	1.605	1.604	1.603	1.602
600	1.607	1.606	1.605	1.604	1.604	1.603	1.602
620	1.606	1.605	1.604	1.603	1.602	1.602	1.601
640	1.605	1.604	1.603	1.602	1.602	1.601	1.600
660	1.604	1.603	1.602	1.602	1.601	1.600	1.599
680	1.603	1.603	1.602	1.601	1.600	1.599	1.598
700	1.603	1.602	1.601	1.600	1.599	1.598	1.597
750	1.601	1.600	1.599	1.598	1.598	1.597	1.596
800	1.600	1.599	1.598	1.597	1.596	1.595	1.595



Table 18A. **Actinolite  $\alpha$**  (In Cargille Series E: **1.615**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.646	1.645	1.644	1.644	1.643	1.642	1.641
420	1.641	1.640	1.639	1.638	1.637	1.636	1.635
440	1.636	1.636	1.635	1.634	1.633	1.632	1.631
460	1.633	1.632	1.631	1.630	1.629	1.628	1.627
480	1.629	1.629	1.628	1.627	1.626	1.625	1.624
500	1.627	1.626	1.625	1.624	1.623	1.622	1.621
520	1.624	1.623	1.622	1.622	1.621	1.620	1.619
540	1.622	1.621	1.620	1.619	1.618	1.618	1.617
560	1.620	1.619	1.618	1.617	1.617	1.616	1.615
580	1.618	1.618	1.617	1.616	1.615	1.614	1.613
589	1.618	1.617	1.616	1.615	1.614	1.613	1.612
600	1.617	1.616	1.615	1.614	1.613	1.612	1.611
620	1.615	1.615	1.614	1.613	1.612	1.611	1.610
640	1.614	1.613	1.612	1.612	1.611	1.610	1.609
660	1.613	1.612	1.611	1.610	1.609	1.609	1.608
680	1.612	1.611	1.610	1.609	1.608	1.608	1.607
700	1.611	1.610	1.609	1.608	1.607	1.607	1.606
750	1.609	1.608	1.607	1.606	1.605	1.604	1.603
800	1.607	1.606	1.605	1.604	1.604	1.603	1.602

Table 18B. **Actinolite  $\gamma$**  (In Cargille Series E: **1.615**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.642	1.641	1.640	1.639	1.638	1.637	1.636
420	1.637	1.636	1.635	1.635	1.634	1.633	1.632
440	1.634	1.633	1.632	1.631	1.630	1.629	1.628
460	1.630	1.629	1.629	1.628	1.627	1.626	1.625
480	1.628	1.627	1.626	1.625	1.624	1.623	1.622
500	1.625	1.624	1.623	1.623	1.622	1.621	1.620
520	1.623	1.622	1.621	1.621	1.620	1.619	1.618
540	1.621	1.620	1.620	1.619	1.618	1.617	1.616
560	1.620	1.619	1.618	1.617	1.616	1.615	1.614
580	1.618	1.617	1.617	1.616	1.615	1.614	1.613
589	1.618	1.617	1.616	1.615	1.614	1.613	1.612
600	1.617	1.616	1.615	1.614	1.613	1.613	1.612
620	1.616	1.615	1.614	1.613	1.612	1.611	1.610
640	1.615	1.614	1.613	1.612	1.611	1.610	1.609
660	1.614	1.613	1.612	1.611	1.610	1.609	1.608
680	1.613	1.612	1.611	1.610	1.609	1.608	1.607
700	1.612	1.611	1.610	1.609	1.608	1.608	1.607
750	1.610	1.609	1.608	1.608	1.607	1.606	1.605
800	1.609	1.608	1.607	1.606	1.605	1.604	1.603

Table 19A. **Actinolite  $\alpha$**  (In Cargille Series E: **1.620**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.653	1.652	1.651	1.650	1.649	1.648	1.647
420	1.647	1.646	1.645	1.644	1.644	1.643	1.642
440	1.642	1.642	1.641	1.640	1.639	1.638	1.637
460	1.638	1.638	1.637	1.636	1.635	1.634	1.633
480	1.635	1.634	1.633	1.632	1.631	1.631	1.630
500	1.632	1.631	1.630	1.629	1.629	1.628	1.627
520	1.630	1.629	1.628	1.627	1.626	1.625	1.624
540	1.627	1.626	1.625	1.625	1.624	1.623	1.622
560	1.625	1.624	1.623	1.623	1.622	1.621	1.620
580	1.623	1.623	1.622	1.621	1.620	1.619	1.618
589	1.623	1.622	1.621	1.620	1.619	1.618	1.617
600	1.622	1.621	1.620	1.619	1.618	1.617	1.616
620	1.620	1.619	1.619	1.618	1.617	1.616	1.615
640	1.619	1.618	1.617	1.616	1.615	1.615	1.614
660	1.618	1.617	1.616	1.615	1.614	1.613	1.612
680	1.617	1.616	1.615	1.614	1.613	1.612	1.611
700	1.616	1.615	1.614	1.613	1.612	1.611	1.610
750	1.613	1.613	1.612	1.611	1.610	1.609	1.608
800	1.612	1.611	1.610	1.609	1.608	1.607	1.606

Table 19B. **Actinolite  $\gamma$**  (In Cargille Series E: **1.620**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.648	1.647	1.646	1.646	1.645	1.644	1.643
420	1.643	1.643	1.642	1.641	1.640	1.639	1.638
440	1.639	1.639	1.638	1.637	1.636	1.635	1.634
460	1.636	1.635	1.634	1.633	1.633	1.632	1.631
480	1.633	1.632	1.631	1.631	1.630	1.629	1.628
500	1.631	1.630	1.629	1.628	1.627	1.626	1.625
520	1.629	1.628	1.627	1.626	1.625	1.624	1.623
540	1.627	1.626	1.625	1.624	1.623	1.622	1.621
560	1.625	1.624	1.623	1.622	1.621	1.620	1.619
580	1.623	1.622	1.622	1.621	1.620	1.619	1.618
589	1.623	1.622	1.621	1.620	1.619	1.618	1.617
600	1.622	1.621	1.620	1.619	1.618	1.617	1.617
620	1.621	1.620	1.619	1.618	1.617	1.616	1.615
640	1.620	1.619	1.618	1.617	1.616	1.615	1.614
660	1.619	1.618	1.617	1.616	1.615	1.614	1.613
680	1.618	1.617	1.616	1.615	1.614	1.613	1.612
700	1.617	1.616	1.615	1.614	1.613	1.612	1.611
750	1.615	1.614	1.613	1.612	1.611	1.610	1.609
800	1.613	1.612	1.611	1.611	1.610	1.609	1.608

Table 20A. **Actinolite  $\alpha$**  (In Cargille Series E: **1.625**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.659	1.658	1.657	1.657	1.656	1.655	1.654
420	1.653	1.652	1.652	1.651	1.650	1.649	1.648
440	1.648	1.648	1.647	1.646	1.645	1.644	1.643
460	1.644	1.643	1.642	1.642	1.641	1.640	1.639
480	1.641	1.640	1.639	1.638	1.637	1.636	1.635
500	1.638	1.637	1.636	1.635	1.634	1.633	1.632
520	1.635	1.634	1.633	1.632	1.631	1.630	1.630
540	1.633	1.632	1.631	1.630	1.629	1.628	1.627
560	1.630	1.630	1.629	1.628	1.627	1.626	1.625
580	1.629	1.628	1.627	1.626	1.625	1.624	1.623
589	1.628	1.627	1.626	1.625	1.624	1.623	1.622
600	1.627	1.626	1.625	1.624	1.623	1.622	1.621
620	1.625	1.624	1.623	1.623	1.622	1.621	1.620
640	1.624	1.623	1.622	1.621	1.620	1.619	1.618
660	1.623	1.622	1.621	1.620	1.619	1.618	1.617
680	1.621	1.621	1.620	1.619	1.618	1.617	1.616
700	1.620	1.619	1.619	1.618	1.617	1.616	1.615
750	1.618	1.617	1.616	1.615	1.614	1.613	1.613
800	1.616	1.615	1.614	1.613	1.612	1.612	1.611

Table 20B. **Actinolite  $\gamma$**  (In Cargille Series E: **1.625**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.655	1.654	1.653	1.652	1.651	1.650	1.649
420	1.650	1.649	1.648	1.647	1.646	1.645	1.644
440	1.645	1.645	1.644	1.643	1.642	1.641	1.640
460	1.642	1.641	1.640	1.639	1.638	1.637	1.637
480	1.639	1.638	1.637	1.636	1.635	1.634	1.633
500	1.636	1.635	1.634	1.634	1.633	1.632	1.631
520	1.634	1.633	1.632	1.631	1.630	1.629	1.628
540	1.632	1.631	1.630	1.629	1.628	1.627	1.626
560	1.630	1.629	1.628	1.627	1.626	1.626	1.625
580	1.628	1.627	1.627	1.626	1.625	1.624	1.623
589	1.628	1.627	1.626	1.625	1.624	1.623	1.622
600	1.627	1.626	1.625	1.624	1.623	1.622	1.622
620	1.626	1.625	1.624	1.623	1.622	1.621	1.620
640	1.624	1.624	1.623	1.622	1.621	1.620	1.619
660	1.623	1.622	1.622	1.621	1.620	1.619	1.618
680	1.622	1.621	1.621	1.620	1.619	1.618	1.617
700	1.621	1.620	1.620	1.619	1.618	1.617	1.616
750	1.619	1.618	1.618	1.617	1.616	1.615	1.614
800	1.618	1.617	1.616	1.615	1.614	1.613	1.612

Table 21A. **Actinolite  $\alpha$**  (In Cargille Series E: **1.640**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.679	1.678	1.677	1.676	1.675	1.674	1.673
420	1.672	1.671	1.670	1.669	1.668	1.667	1.667
440	1.666	1.665	1.665	1.664	1.663	1.662	1.661
460	1.662	1.661	1.660	1.659	1.658	1.657	1.656
480	1.658	1.657	1.656	1.655	1.654	1.653	1.652
500	1.654	1.653	1.652	1.651	1.650	1.649	1.649
520	1.651	1.650	1.649	1.648	1.647	1.646	1.645
540	1.648	1.647	1.646	1.646	1.645	1.644	1.643
560	1.646	1.645	1.644	1.643	1.642	1.641	1.640
580	1.644	1.643	1.642	1.641	1.640	1.639	1.638
589	1.643	1.642	1.641	1.640	1.639	1.638	1.637
600	1.642	1.641	1.640	1.639	1.638	1.637	1.636
620	1.640	1.639	1.638	1.637	1.636	1.635	1.634
640	1.638	1.637	1.637	1.636	1.635	1.634	1.633
660	1.637	1.636	1.635	1.634	1.633	1.632	1.631
680	1.636	1.635	1.634	1.633	1.632	1.631	1.630
700	1.634	1.633	1.633	1.632	1.631	1.630	1.629
750	1.632	1.631	1.630	1.629	1.628	1.627	1.626
800	1.629	1.628	1.628	1.627	1.626	1.625	1.624

Table 21B. **Actinolite  $\gamma$**  (In Cargille Series E: **1.640**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.674	1.673	1.672	1.672	1.671	1.670	1.669
420	1.668	1.667	1.667	1.666	1.665	1.664	1.663
440	1.663	1.663	1.662	1.661	1.660	1.659	1.658
460	1.659	1.658	1.657	1.657	1.656	1.655	1.654
480	1.656	1.655	1.654	1.653	1.652	1.651	1.650
500	1.653	1.652	1.651	1.650	1.649	1.648	1.647
520	1.650	1.649	1.648	1.647	1.646	1.645	1.644
540	1.648	1.647	1.646	1.645	1.644	1.643	1.642
560	1.645	1.645	1.644	1.643	1.642	1.641	1.640
580	1.644	1.643	1.642	1.641	1.640	1.639	1.638
589	1.643	1.642	1.641	1.640	1.639	1.638	1.637
600	1.642	1.641	1.640	1.639	1.638	1.637	1.636
620	1.640	1.639	1.638	1.638	1.637	1.636	1.635
640	1.639	1.638	1.637	1.636	1.635	1.634	1.633
660	1.638	1.637	1.636	1.635	1.634	1.633	1.632
680	1.636	1.636	1.635	1.634	1.633	1.632	1.631
700	1.635	1.634	1.634	1.633	1.632	1.631	1.630
750	1.633	1.632	1.631	1.630	1.629	1.628	1.628
800	1.631	1.630	1.629	1.628	1.627	1.626	1.626

**Table 22A. Anthophyllite  $\alpha$  (In Cargille Series E: 1.605)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.632	1.631	1.630	1.630	1.629	1.628	1.627
420	1.628	1.627	1.626	1.625	1.624	1.623	1.622
440	1.624	1.623	1.622	1.621	1.620	1.619	1.619
460	1.621	1.620	1.619	1.618	1.617	1.616	1.615
480	1.618	1.617	1.616	1.615	1.614	1.613	1.613
500	1.615	1.615	1.614	1.613	1.612	1.611	1.610
520	1.613	1.612	1.612	1.611	1.610	1.609	1.608
540	1.611	1.611	1.610	1.609	1.608	1.607	1.606
560	1.610	1.609	1.608	1.607	1.606	1.605	1.604
580	1.608	1.607	1.607	1.606	1.605	1.604	1.603
589	1.608	1.607	1.606	1.605	1.604	1.603	1.602
600	1.607	1.606	1.605	1.604	1.603	1.603	1.602
620	1.606	1.605	1.604	1.603	1.602	1.601	1.600
640	1.605	1.604	1.603	1.602	1.601	1.600	1.599
660	1.604	1.603	1.602	1.601	1.600	1.599	1.598
680	1.603	1.602	1.601	1.600	1.599	1.598	1.597
700	1.602	1.601	1.600	1.599	1.598	1.597	1.597
750	1.600	1.599	1.598	1.597	1.597	1.596	1.595
800	1.599	1.598	1.597	1.596	1.595	1.594	1.593

**Table 22B. Anthophyllite  $\gamma$  (In Cargille Series E: 1.605)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.631	1.630	1.629	1.628	1.627	1.626	1.625
420	1.626	1.625	1.625	1.624	1.623	1.622	1.621
440	1.623	1.622	1.621	1.620	1.619	1.618	1.617
460	1.620	1.619	1.618	1.617	1.616	1.615	1.614
480	1.617	1.616	1.615	1.614	1.614	1.613	1.612
500	1.615	1.614	1.613	1.612	1.611	1.610	1.610
520	1.613	1.612	1.611	1.610	1.609	1.608	1.608
540	1.611	1.610	1.609	1.609	1.608	1.607	1.606
560	1.610	1.609	1.608	1.607	1.606	1.605	1.604
580	1.608	1.607	1.606	1.606	1.605	1.604	1.603
589	1.608	1.607	1.606	1.605	1.604	1.603	1.602
600	1.607	1.606	1.605	1.604	1.603	1.603	1.602
620	1.606	1.605	1.604	1.603	1.602	1.601	1.601
640	1.605	1.604	1.603	1.602	1.601	1.600	1.600
660	1.604	1.603	1.602	1.601	1.600	1.600	1.599
680	1.603	1.602	1.601	1.600	1.600	1.599	1.598
700	1.602	1.601	1.601	1.600	1.599	1.598	1.597
750	1.601	1.600	1.599	1.598	1.597	1.596	1.595
800	1.599	1.598	1.597	1.596	1.596	1.595	1.594

Table 23A. **Anthophyllite  $\alpha$**  (In Cargille Series **E: 1.615**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.645	1.644	1.643	1.643	1.642	1.641	1.640
420	1.640	1.639	1.638	1.637	1.637	1.636	1.635
440	1.636	1.635	1.634	1.633	1.632	1.631	1.630
460	1.632	1.631	1.630	1.630	1.629	1.628	1.627
480	1.629	1.628	1.627	1.626	1.625	1.625	1.624
500	1.626	1.625	1.625	1.624	1.623	1.622	1.621
520	1.624	1.623	1.622	1.621	1.620	1.620	1.619
540	1.622	1.621	1.620	1.619	1.618	1.617	1.617
560	1.620	1.619	1.618	1.617	1.616	1.616	1.615
580	1.618	1.618	1.617	1.616	1.615	1.614	1.613
589	1.618	1.617	1.616	1.615	1.614	1.613	1.612
600	1.617	1.616	1.615	1.614	1.613	1.612	1.612
620	1.616	1.615	1.614	1.613	1.612	1.611	1.610
640	1.614	1.613	1.613	1.612	1.611	1.610	1.609
660	1.613	1.612	1.611	1.611	1.610	1.609	1.608
680	1.612	1.611	1.610	1.609	1.609	1.608	1.607
700	1.611	1.610	1.609	1.609	1.608	1.607	1.606
750	1.609	1.608	1.607	1.606	1.606	1.605	1.604
800	1.607	1.607	1.606	1.605	1.604	1.603	1.602

Table 23B. **Anthophyllite  $\gamma$**  (In Cargille Series **E: 1.615**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.644	1.643	1.642	1.641	1.640	1.639	1.638
420	1.639	1.638	1.637	1.636	1.635	1.634	1.633
440	1.635	1.634	1.633	1.632	1.631	1.630	1.629
460	1.631	1.630	1.630	1.629	1.628	1.627	1.626
480	1.628	1.627	1.627	1.626	1.625	1.624	1.623
500	1.626	1.625	1.624	1.623	1.622	1.621	1.620
520	1.624	1.623	1.622	1.621	1.620	1.619	1.618
540	1.622	1.621	1.620	1.619	1.618	1.617	1.616
560	1.620	1.619	1.618	1.617	1.616	1.615	1.615
580	1.618	1.617	1.617	1.616	1.615	1.614	1.613
589	1.618	1.617	1.616	1.615	1.614	1.613	1.612
600	1.617	1.616	1.615	1.614	1.613	1.612	1.612
620	1.616	1.615	1.614	1.613	1.612	1.611	1.610
640	1.615	1.614	1.613	1.612	1.611	1.610	1.609
660	1.613	1.613	1.612	1.611	1.610	1.609	1.608
680	1.613	1.612	1.611	1.610	1.609	1.608	1.607
700	1.612	1.611	1.610	1.609	1.608	1.607	1.606
750	1.610	1.609	1.608	1.607	1.606	1.605	1.604
800	1.608	1.607	1.606	1.605	1.604	1.604	1.603

**Table 24A. Anthophyllite  $\alpha$  (In Cargille Series E: 1.620)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.652	1.651	1.650	1.649	1.648	1.647	1.646
420	1.646	1.645	1.645	1.644	1.643	1.642	1.641
440	1.642	1.641	1.640	1.639	1.638	1.637	1.636
460	1.638	1.637	1.636	1.635	1.634	1.633	1.633
480	1.635	1.634	1.633	1.632	1.631	1.630	1.629
500	1.632	1.631	1.630	1.629	1.628	1.627	1.626
520	1.629	1.628	1.628	1.627	1.626	1.625	1.624
540	1.627	1.626	1.625	1.624	1.624	1.623	1.622
560	1.625	1.624	1.623	1.623	1.622	1.621	1.620
580	1.623	1.623	1.622	1.621	1.620	1.619	1.618
589	1.623	1.622	1.621	1.620	1.619	1.618	1.617
600	1.622	1.621	1.620	1.619	1.618	1.617	1.616
620	1.620	1.620	1.619	1.618	1.617	1.616	1.615
640	1.619	1.618	1.617	1.616	1.616	1.615	1.614
660	1.618	1.617	1.616	1.615	1.614	1.613	1.613
680	1.617	1.616	1.615	1.614	1.613	1.612	1.611
700	1.616	1.615	1.614	1.613	1.612	1.611	1.610
750	1.614	1.613	1.612	1.611	1.610	1.609	1.608
800	1.612	1.611	1.610	1.609	1.608	1.607	1.607

**Table 24B. Anthophyllite  $\gamma$  (In Cargille Series E: 1.620)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.650	1.649	1.648	1.647	1.646	1.645	1.644
420	1.645	1.644	1.643	1.642	1.641	1.640	1.639
440	1.640	1.639	1.639	1.638	1.637	1.636	1.635
460	1.637	1.636	1.635	1.634	1.633	1.632	1.631
480	1.634	1.633	1.632	1.631	1.630	1.629	1.628
500	1.631	1.630	1.629	1.628	1.628	1.627	1.626
520	1.629	1.628	1.627	1.626	1.625	1.624	1.623
540	1.627	1.626	1.625	1.624	1.623	1.622	1.621
560	1.625	1.624	1.623	1.622	1.621	1.621	1.620
580	1.623	1.622	1.622	1.621	1.620	1.619	1.618
589	1.623	1.622	1.621	1.620	1.619	1.618	1.617
600	1.622	1.621	1.620	1.619	1.618	1.617	1.617
620	1.621	1.620	1.619	1.618	1.617	1.616	1.615
640	1.619	1.619	1.618	1.617	1.616	1.615	1.614
660	1.618	1.617	1.617	1.616	1.615	1.614	1.613
680	1.617	1.616	1.616	1.615	1.614	1.613	1.612
700	1.616	1.616	1.615	1.614	1.613	1.612	1.611
750	1.614	1.614	1.613	1.612	1.611	1.610	1.609
800	1.613	1.612	1.611	1.610	1.609	1.608	1.607

Table 25A. **Anthophyllite  $\alpha$**  (In Cargille Series **E: 1.625**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.658	1.657	1.656	1.656	1.655	1.654	1.653
420	1.653	1.652	1.651	1.650	1.649	1.648	1.647
440	1.648	1.647	1.646	1.645	1.644	1.643	1.642
460	1.644	1.643	1.642	1.641	1.640	1.639	1.638
480	1.640	1.639	1.639	1.638	1.637	1.636	1.635
500	1.637	1.636	1.636	1.635	1.634	1.633	1.632
520	1.635	1.634	1.633	1.632	1.631	1.630	1.629
540	1.632	1.631	1.631	1.630	1.629	1.628	1.627
560	1.630	1.629	1.629	1.628	1.627	1.626	1.625
580	1.628	1.628	1.627	1.626	1.625	1.624	1.623
589	1.628	1.627	1.626	1.625	1.624	1.623	1.622
600	1.627	1.626	1.625	1.624	1.623	1.622	1.621
620	1.625	1.624	1.624	1.623	1.622	1.621	1.620
640	1.624	1.623	1.622	1.621	1.620	1.619	1.619
660	1.623	1.622	1.621	1.620	1.619	1.618	1.617
680	1.622	1.621	1.620	1.619	1.618	1.617	1.616
700	1.621	1.620	1.619	1.618	1.617	1.616	1.615
750	1.618	1.617	1.616	1.616	1.615	1.614	1.613
800	1.616	1.615	1.615	1.614	1.613	1.612	1.611

Table 25B. **Anthophyllite  $\gamma$**  (In Cargille Series **E: 1.625**)

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.656	1.655	1.654	1.653	1.652	1.652	1.651
420	1.651	1.650	1.649	1.648	1.647	1.646	1.645
440	1.646	1.645	1.645	1.644	1.643	1.642	1.641
460	1.643	1.642	1.641	1.640	1.639	1.638	1.637
480	1.639	1.638	1.638	1.637	1.636	1.635	1.634
500	1.637	1.636	1.635	1.634	1.633	1.632	1.631
520	1.634	1.633	1.632	1.631	1.631	1.630	1.629
540	1.632	1.631	1.630	1.629	1.628	1.628	1.627
560	1.630	1.629	1.628	1.627	1.627	1.626	1.625
580	1.628	1.628	1.627	1.626	1.625	1.624	1.623
589	1.628	1.627	1.626	1.625	1.624	1.623	1.622
600	1.627	1.626	1.625	1.624	1.623	1.622	1.621
620	1.626	1.625	1.624	1.623	1.622	1.621	1.620
640	1.624	1.623	1.622	1.622	1.621	1.620	1.619
660	1.623	1.622	1.621	1.620	1.619	1.619	1.618
680	1.622	1.621	1.620	1.619	1.618	1.618	1.617
700	1.621	1.620	1.619	1.618	1.617	1.617	1.616
750	1.619	1.618	1.617	1.616	1.615	1.614	1.614
800	1.617	1.616	1.615	1.614	1.614	1.613	1.612



**Table 26A. Anthophyllite  $\alpha$  (In Cargille Series E: 1.635)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.671	1.670	1.669	1.669	1.668	1.667	1.666
420	1.665	1.664	1.663	1.662	1.661	1.660	1.660
440	1.660	1.659	1.658	1.657	1.656	1.655	1.654
460	1.655	1.654	1.654	1.653	1.652	1.651	1.650
480	1.652	1.651	1.650	1.649	1.648	1.647	1.646
500	1.648	1.647	1.646	1.646	1.645	1.644	1.643
520	1.645	1.645	1.644	1.643	1.642	1.641	1.640
540	1.643	1.642	1.641	1.640	1.639	1.638	1.637
560	1.641	1.640	1.639	1.638	1.637	1.636	1.635
580	1.639	1.638	1.637	1.636	1.635	1.634	1.633
589	1.638	1.637	1.636	1.635	1.634	1.633	1.632
600	1.637	1.636	1.635	1.634	1.633	1.632	1.631
620	1.635	1.634	1.633	1.632	1.631	1.631	1.630
640	1.634	1.633	1.632	1.631	1.630	1.629	1.628
660	1.632	1.631	1.630	1.630	1.629	1.628	1.627
680	1.631	1.630	1.629	1.628	1.627	1.626	1.626
700	1.630	1.629	1.628	1.627	1.626	1.625	1.624
750	1.627	1.626	1.626	1.625	1.624	1.623	1.622
800	1.625	1.624	1.623	1.623	1.622	1.621	1.620

**Table 26B. Anthophyllite  $\gamma$  (In Cargille Series E: 1.635)**

$\lambda_0$	19°C	21°C	23°C	25°C	27°C	29°C	31°C
400	1.670	1.669	1.668	1.667	1.666	1.665	1.664
420	1.664	1.663	1.662	1.661	1.660	1.659	1.658
440	1.659	1.658	1.657	1.656	1.655	1.654	1.653
460	1.655	1.654	1.653	1.652	1.651	1.650	1.649
480	1.651	1.650	1.649	1.648	1.647	1.646	1.645
500	1.648	1.647	1.646	1.645	1.644	1.643	1.642
520	1.645	1.644	1.643	1.642	1.641	1.640	1.640
540	1.643	1.642	1.641	1.640	1.639	1.638	1.637
560	1.641	1.640	1.639	1.638	1.637	1.636	1.635
580	1.639	1.638	1.637	1.636	1.635	1.634	1.633
589	1.638	1.637	1.636	1.635	1.634	1.633	1.632
600	1.637	1.636	1.635	1.634	1.633	1.632	1.631
620	1.635	1.634	1.633	1.633	1.632	1.631	1.630
640	1.634	1.633	1.632	1.631	1.630	1.629	1.628
660	1.633	1.632	1.631	1.630	1.629	1.628	1.627
680	1.631	1.630	1.630	1.629	1.628	1.627	1.626
700	1.630	1.629	1.628	1.628	1.627	1.626	1.625
750	1.628	1.627	1.626	1.625	1.624	1.623	1.622
800	1.626	1.625	1.624	1.623	1.622	1.621	1.620

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## APPENDIX

G. W. Bailey and C. L. Rieder, Eds., Proc. 51st Annual Meeting of the Microscopy Society of America  
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# DETERMINATION OF REFRACTIVE INDEX OF SOLIDS BY DISPERSION STAINING METHOD: AN ANALYTICAL APPROACH

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When immersion liquids are used to determine the refractive index (RI) of non-opaque solids, the dispersion staining method is a simple, effective and precise way to determine the matching wavelength,  $\lambda_0$ , at which the RI of an immersion liquid equals that of the solid. A series of equations has been derived to calculate the RI at any given visible wavelength such as  $n_F$ ,  $n_D$  and  $n_C$ , i.e. the RI's at Fraunhofer spectral lines F (486 nm), D (589 nm), and C (656 nm), from  $\lambda_0$  data obtained by the dispersion staining method. Two methods have been established: the *Single Liquid Method* and the *Double Liquid Method*.

The *Single Liquid Method* is applicable for solids with known dispersion coefficients, which is defined as  $(n_F - n_C)$ . This method uses only one immersion liquid whose RI is close to the RI of the solid to be measured so that a match between the liquid and the solid occurs in the visible range. If the matching wavelength is determined to be  $\lambda_0$  (nm), the RI of the solid at wavelength  $i$  (nm) can be readily calculated using the following equation:

$$n_i^s = n_i^l + (\Delta^L - \Delta^s) \cdot k_i,$$

where  $n_i^s$  is the RI of the solid at wavelength  $i$  (nm);  $n_i^l$  the RI of the liquid at wavelength  $i$ ,  $\Delta^L$  the dispersion coefficient of the liquid,  $(n_F^l - n_C^l)$ ;  $\Delta^s$  the dispersion coefficient of the solid,  $(n_F^s - n_C^s)$ ; and  $k_i$  equals  $(X_0 - X_i)/(X_F - X_C)$ .  $X_0$ ,  $X_i$ ,  $X_F$ , and  $X_C$  are defined by replacing the  $\lambda$  in the expression  $(\lambda - 200)^{-1}$  with  $\lambda_0$ ,  $i$ , 486 and 656, respectively. Therefore,  $k_i = [(\lambda_0 - 200)^{-1} - (i - 200)^{-1}] / [(486 - 200)^{-1} - (656 - 200)^{-1}]$ , or  $[(\lambda_0 - 200)^{-1} - (i - 200)^{-1}] / 0.001304$ . In most cases,  $n_D^s$ , the RI of the solid at 589 nm, is to be determined. The above equation then becomes

$$n_D^s = n_D^l + (\Delta^L - \Delta^s) \cdot k_D,$$

where  $n_D^l$  is the RI of the liquid at 589 nm and  $k_D$  equals  $[(\lambda_0 - 200)^{-1} - (589 - 200)^{-1}] / [(\lambda_0 - 200)^{-1} - (656 - 200)^{-1}]$  or  $[(\lambda_0 - 200)^{-1} - 0.002571] / 0.001304$ . Because  $n_D^l$ ,  $\Delta^L$ , and  $\Delta^s$  are known, the only parameter that needs to be measured is  $\lambda_0$ . A table for quick conversion of  $\lambda_0$  to  $k_D$  is provided to minimize the calculations involved.

The *Single Liquid Method* is extremely useful for rapid identification of synthetic and natural fibers, such as polypropylene, polyethylene, nylon, cellulose, etc., as well as fibrous components in bulk insulation samples, such as the six fibrous asbestos minerals regulated by the Environmental Protection Agency: chrysotile, grunerite (or amosite), riebeckite (or crocidolite), tremolite, actinolite, anthophyllite. A single RI liquid mount (1.550 for chrysotile, 1.680 or 1.700 for grunerite and riebeckite and 1.605 for tremolite, actinolite and anthophyllite) is sufficient for

rapidly determining both  $n_{\perp}$  and  $n_{\parallel}$ , the RI's perpendicular and parallel to the fiber elongation, respectively, with reasonable accuracy. A series of conversion tables for the six asbestos minerals are presented in this paper to aid the conversion of an observed  $\lambda_0$  to its corresponding  $n_D^S$  value. Other applications of this method include the estimation of the composition of common rock-forming minerals such as plagioclase, olivine, orthopyroxene and augite.

The **Double Liquid Method** is applicable to any solid and requires *no* knowledge about its dispersion coefficient,  $(n_F^S - n_C^S)$ , or other optical properties. This method uses two immersion liquids whose RI's at 589 nm bracket the RI at 589 nm of the solid to be measured. If the matching wavelengths for Liquid #1 and Liquid #2 are measured to be  $\lambda_0^1$  (nm) and  $\lambda_0^2$  (nm), respectively, the RI of the solid at wavelength  $i$  (nm),  $n_i^S$ , can be readily calculated using the following equation:

$$n_i^S = n_i^1 + (n_i^2 - n_i^1) \cdot k_i,$$

where  $n_i^1$  is the RI of Liquid #1 at  $i$ ;  $n_i^2$  the RI of Liquid #2 at  $i$ ; and  $k_i$  equals  $(X_0^1 - X_i)/(X_0^1 - X_0^2)$ .  $X_i$ ,  $X_0^1$  and  $X_0^2$  are defined by replacing the  $\lambda$  in the expression  $(\lambda - 200)^{-1}$  with  $i$ ,  $\lambda_0^1$  and  $\lambda_0^2$ , respectively. Therefore,  $k_i = [(\lambda_0^1 - 200)^{-1} - (i - 200)^{-1}] / [(\lambda_0^1 - 200)^{-1} - (\lambda_0^2 - 200)^{-1}]$ . In most cases,  $n_D^S$ , the RI of the solid at 589 nm, is to be determined. The above equation then becomes

$$n_D^S = n_D^1 + (n_D^2 - n_D^1) \cdot k_D,$$

where  $n_D^1$  is the RI of Liquid #1 at 589 nm;  $n_D^2$  the RI of Liquid #2 at 589 nm; and  $k_D$  equals  $[(\lambda_0^1 - 200)^{-1} - 0.00257] / [(\lambda_0^1 - 200)^{-1} - (\lambda_0^2 - 200)^{-1}]$ . Because  $n_D^1$  and  $n_D^2$  are known, the parameters that need to be measured are  $\lambda_0^1$  and  $\lambda_0^2$ . A table for quick conversion of  $\lambda_0^1$  and  $\lambda_0^2$  to  $k_D$  is provided to minimize the calculations involved. Apparently, the **Double Liquid Method** is more accurate than the **Single Liquid Method**.

If  $t$ , the temperature of the liquid at the time of determination, is not 25°C, temperature correction must be applied to all RI's of the liquids used in the above calculations. The equation used for this purpose is

$$n_i^t = n_i^{25^\circ\text{C}} + (t - 25) \cdot dn/dt,$$

where  $n_i^t$  is the RI of a liquid at temperature  $t$  (°C) and wavelength  $i$  (nm);  $n_i^{25^\circ\text{C}}$  the RI of the liquid at 25°C and wavelength  $i$  (nm); and  $dn/dt$  the temperature coefficient of the liquid. It should be noted that  $dn/dt$  is a negative value. Therefore, if the  $t$  is higher than 25°C,  $n_i^t$  is lower than  $n_i^{25^\circ\text{C}}$ .

Besides dispersion staining, there are other techniques for determining the matching wavelength  $\lambda_0$  between the RI of a solid and that of its surrounding liquid medium, e.g., the traditional wavelength-variation method, double-variation method, oblique illumination method, etc. The above equations are equally applicable to the  $\lambda_0$  data obtained by these and *any* other techniques.



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis - CAL ARB 435 Method**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-4-2-PLM-02**

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#### **Purpose**

To outline the equipment and procedures used by Asbestos TEM Laboratories in the analysis of bulk asbestos by the technique of polarized light microscopy which is based upon the ARB Method 435. This method also relies heavily upon the EPA "Interim Method for the Determination of Asbestos in Bulk Insulation Samples", and the EPA document, "Asbestos-Containing Materials in Schools; Final Rule and Notice", 40 CFR Part 763.

#### **Scope / Field of Application**

Serpentine aggregate material or other rock/soil material which is suitable and requested for analysis by a client to use CAL ARB 435 Method.

#### **Serpentine Asbestos in California**

The geologic processes that formed the area now covered by the State of California occurred in such a way that a large number of ultra-mafic serpentinite outcrops are present throughout it. In particular, the Coast Ranges and the Sierra Foothills are having an abundance of serpentinite present. So much asbestos is present in the State of California that is the official state rock. In a number of locales, in particular the area near Coalinga, large outcrops of serpentine asbestos (chrysotile) were mined for asbestos which was used in a variety of building and insulation materials.

Unfortunately, voluminous medical evidence has been gathered indicating that exposure to asbestos has the potential to cause cancer in humans. This has led to a widespread effort to document the location, amount, and condition of asbestos-materials in buildings and in the natural environment, where exposure to humans could lead to adverse health effects. It is the purpose of Asbestos TEM Laboratories to assist in this effort to document the presence of asbestos in buildings, soils, and other materials through the application of proven scientific asbestos analytical techniques.

The minerals which have been designated as asbestos by the EPA (Chrysotile serpentine and the amphiboles -amosite, anthophyllite, tremolite, actinolite, and crocidolite) are those minerals which Asbestos TEM Laboratories calls asbestos when found in laboratory samples. Other minerals exist which also have asbestiform habits and similar physical characteristics, but these are not covered by current regulatory requirements and are not considered asbestos, i.e. attapulgite, sepiolite, palygorskite.

Over the past several years the California Air Resources Board has developed a method to control emissions of serpentine asbestos from rock quarries and road-beds which were known to contain serpentine rock and, quite likely, serpentine asbestos. This method, outlined below, is a point counting method which, when applied correctly, should lead to a high degree of confidence as to whether the material being tested contains asbestos.



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis - CAL ARB 435 Method**

Issue Date: **2004/12/16**

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#### **Analytical Hardware Requirements**

**A. Apparatus for Gross Examination**

1. Magnifying Lens, 10X
2. Light Source: fluorescent and incandescent lights
3. Hand Tools: tweezers, scalpel, razor blades, probes, etc.
4. Petri Dish: clean glass plate

**B. Apparatus for Sample Preparation**

1. Filtered Ventilation System
2. Microscope Slides
3. Cover Slips
4. Disposable Gloves
5. Hand Tools
6. Jaw Crusher
7. Pulverizer
8. Drying Oven

**C. Apparatus for Identification and Quantification**

1. Polarized Light Microscope
  - a. Polarizer
  - b. Analyzer
  - c. Port for Wave Retardation Plate
  - d. 360° Graduated Rotating Stage
  - e. Substage Condenser
  - f. Lamp
  - g. Lamp Iris
  - h. Condenser Diaphragm
  - i. Objective Lenses - 4X, 10X, 20X, 40X,
  - j. Dispersion Staining Objective Lens 10X
  - k. Ocular Reticle (10X) w/ Cross Hair
  - l. Retardation Plate - First Order Red, 550 nm

**D. Reagents for Sample Preparation**

1. Refractive Index Liquids: 1.490-1.570 and 1.590-1.720 in 0.002 or 0.004 Step Increments
2. Distilled Water
3. Mineral Oil
4. Dilute HCl acid

**E. Analytical Standard Reference Materials**



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **PLM Analysis - CAL ARB 435 Method**

Issue Date: **2004/12/16**

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1. U.C. Berkeley Mineral Collection Reference Standards
2. NIST Asbestos PAT Round Reference Sample Sets

## **Analytical Procedures**

### **A. Sampling**

The collection of bulk material that will be analyzed for asbestos content is beyond the scope of this method. The analyst must assume that the samples were taken according to the prescribed guidelines for sample collection described in the California Air Resources Board Method 435, Section 5. In the case of doubt as to the sampling procedures, the analyst will not proceed with the analysis until the client has been notified and the problem resolved.

### **B. Chain-of-Custody**

Before accepting the sample(s) for analysis the sample coordinator must be sure that proper chain-of-custody procedures have been followed. The documentation is thoroughly reviewed, the samples are checked to see if they are damaged or if any chain-of-custody seals have been broken or tampered with. Also, all paperwork is reviewed to be sure that it is in order and contains the clients name, and signatures of all sample custodians and the date and time at which custody exchanges occurred. If any problems are found, the client is immediately notified and made aware of the situation and that the laboratory can not accept the samples. Only after the samples pass these checks, are the samples accepted by the laboratory and logged in to the computerized Laboratory Information System (LIMS).

### **C. Data Review and Transcription**

Prior to sample analysis, all paperwork sent to the analyst after sample log-in (Covered in the QA/QC Manual) is reviewed by the analyst for accuracy. A sample is then chosen from the sample lot, and all relevant data concerning that sample is transcribed to the PLM Data Sheet (See Appendix A in section 5-4-2-PLM-01) to be used during the analysis. This information includes Laboratory Sample ID#, Client Sample ID#, Job Site, Location, and Description.

### **D. Initial Sample Preparation**

If the submitted samples (minimum 1 pint material) have not been ground to a nominal 200-mesh particle size, and are in gross bulk form, it is necessary for the analyst to reduce the material to the correct grain size. First, check to be sure the material is dry. If not, place in a drying oven at 350 degrees F and dry overnight. Then, set up the jaw crusher in the HEPA hood, and crush the sample material to approximately 1/8" size fragments. Be careful to



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thoroughly clean the crusher between samples with a brush and alcohol. Also, be mindful not to jam the jaw crusher as you can burn up the motor if you are not careful.

After crushing the material, the sample must be pulverized in the pulverizer to a nominal 200-mesh grain size (A nominal 200-mesh grain size is defined as a material that, when sieved with a Standard 200-mesh testing sieve, greater than 50% of the material passes through the screen). Set up the pulverizer in the HEPA hood and thoroughly clean the ceramic pulverizer plates and surrounding interior casing. Place the special plastic bags over the sample exit slot and fasten with a rubber band. Secure the removable facing, and dial in the pulverizer until a fairly stiff resistance is met. If the material is quite soft, as is the case with many serpentinites, the sample can be run in one pass. However, if the sample contains material such as included chert, grey-wacke, or other hard material, two passes are required. In this case, back off the pressure on the plates to allow a sand sized flow of particulate out of the unit, and then re-run at a tighter setting. Be careful not to over tighten the unit as the unit will then shatter the ceramic plates or burn up the motor. Check a portion of the sample to insure that the bulk of the material will pass through a 200-mesh sieve. Be sure to meticulously clean the unit between samples to prohibit the possibility of cross contamination. Also, between samples, flip the switch on the back of the unit to reverse the plate rotation direction to maximize pulverizer plate life.

After finishing the crushing and grinding, thoroughly clean the machines and the HEPA bench area as the work surfaces get very dusty and potentially covered with hazardous asbestos.

#### **E. Gross Examination**

Samples of serpentine aggregate taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification with a binocular stereo-zoom microscope. An initial estimate of the gross asbestos concentration is made and recorded on the count sheet. Positive identification of suspect fibers is then made by polarized light microscopy.

Notes are made concerning the following:

1. Homogeneity - If sample is heterogeneous, briefly describe the different materials.
2. Texture - e.g. fibrous, matted, rubber, clotted, etc.
3. Color
4. Gross estimated percentage of asbestos.





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The distinction between homogeneous and inhomogeneous material is subjective. A sample is considered to be inhomogeneous if discontinuities between material types are visually significant in hand specimen.


#### **F. Sample Preparation**

##### **1. General Cautionary Guidelines**

Sample preparation should be carried out within general guidelines with the understanding that each analyst will develop working habits which are suited to him/her. The general guidelines should ensure that the individual's habits promote good workmanship and not detract from the work quality. Any sample preparation style should conform to the following basic rules:

- a. All samples shall be opened within the confines of a filtered ventilation system.
- b. All spills shall be cleaned immediately and the work area cleaned periodically, regardless of the absence of visible debris.
- c. The slides, coverslips and refractive index liquids shall be placed under the fume hood and covered at all times when not in use.
- d. All solutions used for cleaning and analysis should be covered and clearly labeled.
- e. A sealable bag should be used for all debris generated during preparation. This bag should be considered as hazardous waste and should be handled and disposed of as such. The debris bag should remain in hood prior to disposal.
- f. All preparation tools should be kept exceptionally clean. Tools should be wiped thoroughly between sample preparations. Due to constant cleaning of the tools, be aware of cellulose contamination in the samples, especially when using the mortar and pestle.
- g. Refractive index liquids are toxic and should be used with extreme caution. Any spills should be cleaned immediately. All trash generated should be placed in the fume hood debris bag. Skin contamination from the liquids should be cleansed thoroughly. The laboratory equipment, such as the microscopes, tools, telephone and doorknobs, should not be contaminated with soiled hands. The work area should not smell of refractive index liquids. Disposable gloves should be available to the analyst. The index liquid dispenser should be kept clean and free of residue.

##### **2. Sample Preparation Procedures**

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With application of the above guidelines, a basic outline for sample preparation is as follows:

- a. After a thorough gross examination of the material (see III.C.) select several homogeneous tweezerfuls of the sample and place a few drops of the desired refractive index liquid on a microscope slide labeled with the laboratory sample ID# and, if necessary, the refractive index of the mounting oil.
- b. Stir, chop or mash the sample, blending the material evenly throughout the liquid. The material should be distributed so that the liquid is not clouded. Light should be capable of passing through all sections of the liquid equally.
- c. Place a coverslip over the sample. Remove the air pockets with applied pressure to the coverslip. The refractive index liquid and the sample should be evenly distributed to the edges of the coverslip.

The analysis of the material on the slide is a representation of the entire homogeneous portion of the material. The relationship between the prepared slide and the submitted sample is known only by the microscopist and should be kept clearly in mind during the analysis. Each microscopist should prepare the slides which he/she intends to analyze. The amount of samples prepared will not exceed the amount in which the analyst can clearly remember, i.e No more than five at a time.

#### G. Fiber Identification

Fiber identification will be performed utilizing all of the available optical properties. As described in the "Interim Method for the Determination of Asbestos in Bulk Insulation Samples", all materials identified as an asbestiform mineral will be distinguished by the following optical properties (See V. below for detailed description of these properties and how they are measured):

1. Morphology
2. Color
3. Pleochroism
4. Refractive index parallel and perpendicular to the fiber elongation direction
5. Birefringence & interference colors
6. Extinction characteristics
7. Sign of elongation
8. Other properties used to characterize a material

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.



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Personnel utilized for training in asbestos microscopy must have a minimum of a Master's or higher degree in the geological sciences. All data collected by the analyst will be entered onto the designated PLM Data Sheet and the laboratory database system.

#### **H. Quantification of Sample Contents**

The quantification of asbestos in bulk material samples will be performed by a point counting technique using a point counting technique. An ocular reticle (Chalkey 25-Point Array) is used to visually superimpose points on the microscope field of view. The microscope slide to be analyzed is positioned into an X-Y sample holder on the microscope stage. A field is chosen at random after checking for proper loading conditions (25%-50% open space). Each point on the Chalkey reticle is checked and counted as outlined below. The point counting rules are as follows.

1. Record the number of points positioned directly above each particle or fiber.
2. Record only one point if two points are positioned over the same particle.
3. Record the number of points positioned on the edge of a particle or fiber.
4. 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.
5. If a test point lies over an ambiguous structure, no particle or fiber is recorded. Examples of ambiguous structures are:
  - a. fibers whose dispersion colors/refractive indices Becke lines are difficult to see
  - b. structures too small to categorize.
6. A fiber mat or bundle is counted as one fiber.

For the purpose of the method, "asbestos fibers" are defined as mineral fibers having an aspect ratio greater than 3:1 and being positively identified as one of six minerals: chrysotile, crocidolite (riebeckite), amosite (grunerite), tremolite, actinolite, anthophyllite.

After completion of analysis of the field, another random field is chosen, as long as it is not the previous field and has the correct loading level. Analysis of the slide continues until 50 points are counted in at least two fields. Each slide is analyzed for 50 points up to a total of 8 slides. A total of 400 points superimposed on either asbestos fibers or non-asbestos matrix materials must be counted over at least eight different preparations of representative subsamples.

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 shall be performed at 100X magnification. Confirmation of the quantitative results shall be performed by reanalysis of the material by a second analyst as a quality control measure. Results must be within reasonable agreement or a reevaluation of the material must occur. All of the following optical properties shall be determined to positively identify asbestos:



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Morphology (3 to 1 minimum aspect ratio)  
Color and pleochroism  
Refractive indices (Becke Line or Dispersion Staining Methods) both parallel and perpendicular to the fiber axis  
Birefringence  
Extinction characteristics  
Sign of elongation

#### Exception I

If the sample is suspected of containing no asbestos a visual technique can be used to report that the sample does not contain asbestos. The rules are as follows:

1. Prepare three slide mounts as described above.
2. View 10 fields per preparation. Identify all fibers.
3. If all fibers are non-asbestos, report no asbestos fibers were found and that the visual technique was used.
4. If one fiber is determined to be asbestos, discontinue the visual method and perform the standard point counting technique as described above.

#### Calculations

The percent asbestos is calculated as follows:

% Asbestos =  $(a/n) \times 100\%$  where

a = number of asbestos fiber counts

n = number of non-empty points counted (400)

If a = 0, report "No asbestos detected"

If a > 0, report the calculated value to the nearest 0.25%

If "no asbestos detected" is reported by the point counting technique, the analyst may report the observation of asbestos fibers in the non-counted portions of the sample.

#### Exception II

If the sample is suspected to have an asbestos content in excess of ten percent, a visual technique can be used to report that the sample contains greater than ten percent asbestos. The standard operating procedure of the visual technique allowed in the National Institute of Standards and Technology's (NIST) National Voluntary Accreditation Program, Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTIR 88-3879 (October 1988), shall be followed.



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Upon completion of the analysis, the analyst is required to review his material closely for errors, enter the date of analysis on the PLM Data Sheet, and then to sign his/her name to the report. Any deviations from the standard method are described in a statement to be added to the cover letter.

Data to be entered into all ARB Method 435 PLM reports includes, at a minimum, the following:

1. Client name and contact address
2. Date samples submitted
3. Date report completed
4. Total samples submitted
5. Total samples analyzed
6. Client sample ID number for each sample
7. Lab ID number for each sample
8. Total Chrysotile Asbestos Fibers Counted
9. Total Amphibole Asbestos Fibers Counted
10. Total Non-asbestos Fibers and other Non-Fibrous Particles Counted
11. Percentage of asbestos detected in the sample to 0.25%
12. Name of the two major non-asbestos fibrous materials (if two or more present)
13. Description &/or location of sample material (if given by client)
14. Gross Sample Color

After the quantitative results are confirmed by reanalyzing the material by a second analyst, the data is input to the laboratory database system. A draft of the report is completed and this preliminary report is then faxed or verbally reported to the client according to the clients' request on their COC form. The preliminary report is reviewed by a laboratory QC reviewer and if no errors are found, the final report is sent to the client by mail.



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#### **Common Diagnostic Optical Tests and Related Principles Regarding PLM Analysis for Asbestos**

Refer to the same section in SOP # 5-04-2-PLM-01 Pages 10 – 21.

#### **Characteristic Optical Properties of the Six Asbestos Minerals**

Refer to the same section in SOP # 5-04-2-PLM-01 Pages 23 – 29.

#### **Characteristic Optical, Physical, & Chemical Properties of Common Asbestos Look-Alike Minerals & Materials**

##### **D. Silicates**

##### **ANTIGORITE**

Refractive Indices:  $\alpha = 1.558 - 1.567$

$\beta = \simeq 1.566$

$\gamma = 1.562 - 1.574$

Crystal System: Monoclinic

Birefringence: 0.004 - 0.007

Color and pleochroism: Green, green-blue, white. Colorless to pale green in thin section.

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = 37^\circ - 61^\circ$

Chemistry:  $\text{Mg}_3[\text{Si}_2\text{O}_5](\text{OH})_4$

Antigorite is a polymorph of the serpentine family of minerals, of which chrysotile is a member. Antigorite can be mistaken for asbestos if the analyst is not careful. Typically, antigorite is not seen in building materials. However, in soil and rock samples, particularly from the California coast ranges or foothills which often contain abundant serpentine minerals, it is quite common.



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Distinguishing antigorite from chrysotile can best be done on the basis of two criteria, 1) fiber morphology and, 2) refractive index. While antigorite often occurs in a fibrous habit, it is not as well developed as in chrysotile which is often hairlike in form. However, when chrysotile is present in small fiber bundles and does not manifest the fine fibrous morphology, it can be difficult to distinguish between the two. The other technique for differentiating the two minerals is by refractive index. While the two are quite close in refractive index, antigorite is consistently higher than 1.55, whereas chrysotile is equal to or less than 1.55.



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#### TALC

Refractive Indices:  $\alpha = 1.539 - 1.550$

$\beta = 1.589 - 1.594$

$\gamma = 1.589 - 1.600$

Crystal System: Monoclinic

Birefringence: 0.05

Color and pleochroism: Colorless, white, pale green, dark green, brown. Colorless in thin section.

Extinction: Parallel

Elongation: Length - slow (+)

Optic Sign: (-);  $2V = 0^\circ - 30^\circ$

Chemistry:  $\text{Mg}_6[\text{Si}_8\text{O}_{20}](\text{OH})_4$

Talc is an asbestos look-alike mineral when it occurs in a fibrous morphology, which is not uncommon. Talc is a hydrous magnesium silicate mineral with composition and optical properties which can lead to its misidentification as chrysotile or anthophyllite if care is not taken. However, talc has a variety of features by which it can be uniquely identified. These properties include: 1) Morphology, 2) Refractive Index and, 3) Birefringence.

The morphology of talc can be highly fibrous. However, it usually appears stiffer than chrysotile, and more flexible than amphiboles. The refractive index of talc, can mimic chrysotile or amphibole if the analyst does not look at both the parallel and perpendicular vibration directions. The refractive indices of talc can be identical to chrysotile in the perpendicular direction, and approximately equal to anthophyllite the parallel direction. However, looking at talc in both 1.55 and 1.604 refractive index oil, and by looking at the fibers in both the parallel and perpendicular directions, can lead to its quick and easy identification. The birefringence of talc is typically higher than chrysotile and closer to amphiboles.

Talc, when seen in building materials, is most commonly observed in flooring mastics and glues.





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#### COMMON HORNBLENDE

Refractive Indices:  $\alpha = 1.615 - 1.705$

$\beta = 1.618 - 1.714$

$\gamma = 1.632 - 1.730$

Crystal System: Monoclinic

Birefringence: 0.014 - 0.026

Color and pleochroism: Pale green, green, lt. yellow-brown to brown. Pleochroism variable in greens, yellow-green, bluish-green and brown.

Extinction: Commonly oblique,  $13^\circ - 34^\circ$ , though may show parallel extinction in certain sections.

Elongation: Length - slow

Optic Sign: (-);  $2V = 95^\circ - 27^\circ$

Chemistry:  $(\text{Na}, \text{K})_{0-1}\text{Ca}_2(\text{Mg}, \text{Fe}^{+2}, \text{Fe}^{+3}, \text{Al})_5$   
 $[\text{Si}_{6-7}\text{Al}_{2-1}\text{O}_{22}](\text{OH}, \text{F})_2$

Common hornblende and other related non-asbestos amphibole minerals can, on occasion, be difficult to differentiate from asbestiform amphiboles when they occur with aspect ratios of 3:1 or greater. However, usually their aspect ratio is very close to 3:1, unlike the asbestiform amphiboles. The most easily recognizable differences are 1) the stronger pleochroism of the iron rich hornblende varieties, 2) the higher refractive indices of the iron rich varieties. Differentiation of the iron-poor hornblende minerals, if they occur with aspect ratios of  $>3:1$ , is difficult and best performed through a combination of refractive index tests with consultation of refractive index vs. composition charts found in Deer, Howie & Zussman, as well as notation of the subtle pleochroism differences.



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#### WOLLASTONITE

Refractive Indices:  $\alpha = 1.616 - 1.640$

$\beta = 1.628 - 1.650$

$\gamma = 1.631 - 1.653$

Crystal System: Triclinic

Birefringence: 0.013 - 0.014

Color and pleochroism: Colorless in thin section.

Extinction: Commonly oblique,  $39^\circ$ , though may show parallel extinction in certain sections.

Elongation: Length - slow & Length - fast

Optic Sign: (-);  $2V = 95^\circ - 27^\circ$

Chemistry:  $\text{Ca}[\text{SiO}_3]$

Wollastonite is not commonly found in building materials, though it is occasionally present. Its morphology, when fibrous, is only weakly asbestiform (near 3:1 aspect ratio) however, on occasion it closely mimics tremolite/actinolite. It is distinguished from the amphibole forms of asbestos by its weaker birefringence, and its variable sign of elongation. To test the variable sign of elongation, view the sample with the retardation plate inserted and under crossed polars, and with a pair of tweezers, push on the coverslip and get the fiber to roll over. If it changes its sign of elongation, it is wollastonite and not tremolite/actinolite.



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#### QUARTZ

Refractive Indices:  $\omega = 1.544$

$\varepsilon = 1.553$

Crystal System: TRIGONAL

Birefringence: 0.009

Color and pleochroism: Colorless in thin section.

Extinction: Irregular to to conchoidal fracture.

Elongation: Not applicable

Optic Sign: (+)

Chemistry:  $\text{SiO}_2$

Quartz is commonly found in plasters, cementitious materials, and soils. It has no cleavage and commonly occurs as rounded grains. It is extremely hard and usually impossible to reduce in size. It has low birefringence and very constant refractive indices.



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#### ALKALI FELDSPARS

Refractive Indices:  $\alpha = 1.518 - 1.529$

$\beta = 1.518 - 1.533$

$\gamma = 1.521 - 1.539$

Crystal System: Monoclinic & Triclinic

Birefringence: 0.006 - 0.010 (Low)

Color and pleochroism: Colorless in thin section.

Extinction: Commonly oblique to twinning and exsolution lamellae, though may be parallel in certain sections.

Elongation: Not Applicable

Optic Sign: (+ or -);  $2V = 5^\circ - 20^\circ$

Chemistry:  $(K,Na)[AlSi_3O_8]$

Alkali feldspars rarely appear as fibers (though sanidine may). They are generally present in building materials as rounded grains in cementitious materials with quartz. They are commonly distinguished from quartz by the presence of twinning and its lower refractive indices. It is not necessary to differentiate between the alkali and the plagioclase feldspars and the term feldspar may be used.



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#### PLAGIOCLASE FELDSPARS

Refractive Indices:  $\alpha = 1.527 - 1.577$

$\beta = 1.532 - 1.585$

$\gamma = 1.534 - 1.590$

Crystal System: Triclinic

Birefringence: 0.007 - 0.013 (Low)

Color and pleochroism: Colorless in thin section.

Extinction: Commonly oblique to twinning and exsolution lamellae, though may be parallel in certain sections.

Elongation: Not Applicable

Optic Sign: (+ or -);  $2V = 45^\circ - 78^\circ$

Chemistry:  $\text{Na}[\text{AlSi}_3\text{O}_8] - \text{Ca}[\text{Al}_2\text{Si}_2\text{O}_8]$

Plagioclase feldspars do not appear as fibers. They are generally present in building materials as rounded grains in cementitious materials with quartz. They can commonly be distinguished from quartz by the presence of twinning and their lower refractive indices. It is not necessary to differentiate between the alkali and the plagioclase feldspars and the term feldspar may be used.



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#### CALCITE

Refractive Indices:  $\omega = 1.486 - 1.550$

$\varepsilon = 1.658 - 1.740$

Crystal System: TRIGONAL

Birefringence: 0.172 - 0.190

Color and pleochroism: Colorless in thin section.

Extinction: Symmetrical

Elongation: Not applicable

Optic Sign: (-)

Chemistry:  $\text{CaCO}_3$

Calcite is extremely common in building materials and may occur in floor tiles, ceiling tiles, spray-on ceilings, and in plasters and other cementitious materials. It does not occur as fibers, but often as rhombic sections. It is identified by its extremely high birefringence, and the common occurrence of twinning.

The presence of calcite can be easily tested by placing a portion of sample material into a drop of dilute acid, where it will vigorously fizz and dissolve.



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## **Documentation**

The following documents are managed:



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Required Record	Custodian
EPA: Interim Method for the Determination of Asbestos in Bulk Insulation Samples	Laboratory Manager
EPA: Asbestos-Containing Materials in Schools; Final Rule and Notice	Laboratory Manager
EPA: Method for the Determination of Asbestos in Bulk Building Materials	Laboratory Manager
CAL EPA - ARB: Method 435 Determination of Asbestos Content of Serpentine Aggregate	Laboratory Manager

### Reference Procedures

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### Revision History

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1	Dec. 16, 2004	Format and Wording

Approval

Yanxia Xie

Reviewer

12 / 16 / 2004

Date

Yanxia Xie Init: \_\_\_\_\_

Quality Assurance Manager

12 / 16 / 2004

Date





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**Polarized Light Microscopy Division**

### **PLM Analysis - CAL ARB 435 Method**

Issue Date: **2004/12/16**

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- APPENDIX -

Crushing & Pulverizing Operations Manual



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#### **CAL ARB 435**

#### **Crushing & Pulverizing Operations Manual**

##### **Introduction**

Soil and rock samples submitted for CAL ARB 435 analysis must be reduced in particle size such that the material to be analyzed is a 'nominal' 200 mesh particle size – i.e. when sieved with a 200-mesh screen, at least half of the material passes through. This typically requires a crushing & pulverizing procedure as described below.

##### **Large Volume Samples (>1 Pint material)**

If samples are large it may be necessary to split the sample into a smaller aliquot. If at all possible, do this before drying as the lab has a limited drying oven space. Sample splitting is best done by crushing the sample, then passing it through a sample splitter. The sample is placed into one tray then poured into the sample splitter which has two receiving trays below it where half the sample goes into each receiving tray. Split the sample until ~ 1 pint of material remains.

##### **Sample Crushing**

Asbestos TEM Labs has one small and one large jaw crusher. The large jaw crusher should be used for sample containing rock fragments >3/8" particle size, otherwise the samples can be run through the large pulverizer directly.

##### **Before Crushing:**

- Put on safety glasses & hearing protection
- Turn on negative air machine – Check to be sure air flow rate into sample crushing chamber is  $\geq 100$  ft./min.
- HEPA vacuum & wet wipe crushing jaws & sample receiving trays – Be sure all visible dust is removed from the crushing equipment to minimize possibility of sample contamination
- Connect HEPA vacuum to crusher exhaust port & turn on – This will greatly minimize the dust generated inside the chamber during crushing.
- Turn on crusher (Do not place sample into crusher until after unit is turned on) – Binding of crusher and burnout of the electric motor drive could occur.
- Pour sample into crusher. Do not look into crusher while it is crushing – Sample fragments may be ejected at high speed and could cause serious injury.
- Turn off crusher immediately upon completion of crushing & before retrieving sample. – Usually, only pass through the crusher is needed to adequately crush your sample. If further passes are need, run through again as documented above without re-cleaning.

##### **After Crushing:**

- Clean up your mess.



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#### Sample Drying

It is REQUIRED that all samples be dried in the drying oven for a minimum of 8-12 hours at 110-150°C prior to pulverizing. Failure to do so will result in the pulverizer becoming clogged and your sample reduced to a mud ball.

#### Sample Pulverizing

Asbestos TEM Laboratories has two sample pulverizers: a large one and a small one. In almost all cases, except where the sample size is very small, use the large pulverizer.

##### Before Pulverizing:

- Put on safety glasses & hearing protection
- Turn on negative air machine – Check to be sure air flow rate into sample crushing chamber is  $\geq 100$  ft./min.
- HEPA vacuum & wet wipe pulverizer plates & sample receiving trays – Be sure all visible dust is removed from the pulverizing equipment to minimize possibility of sample contamination.
- Connect HEPA vacuum to pulverizer exhaust port & turn on – This will greatly minimize the dust generated inside the chamber during pulverizing.
- Close up pulverizer and be sure left immovable plate is locked in place.
- Check pulverizer plates are not bound – You should be able to just rotate the pulverizer shaft by hand before turning on.
- Turn on pulverizer (Do not place sample into crusher until after unit is turned on) – Binding of pulverizer and burnout of the electric motor drive could occur.
- Pour sample into pulverizer – Adjust sample particle size output by tightening or releasing the plate tension using the right dial screw at end of pulverizer shaft.
- Turn off pulverizer immediately upon completion of pulverizing & before retrieving sample – Typically, three passes through the pulverizer are necessary to crush your sample to the required ‘nominal’ 200-mesh particle size. If the starting material is a clay or sand, fewer passes are needed. If the starting material is particularly hard, more may be needed. If further passes are needed, run through again as documented above without re-cleaning.

##### After Crushing:

- Clean up your mess.

#### Retrieving Samples From Crusher/Pulverizer Containment & Turning Off Negative Air & HEPA Vacuums

Be sure to place all samples into sealed containers before retrieving them from the negative air



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containment. Turn off negative air machine and HEPA vacuum only when you are completely done.



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**PLM Analysis – Gravimetric Point Count Method**

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### **Purpose**

To outline the equipment and procedures used by Asbestos TEM Laboratories in the analysis of bulk asbestos by the technique of polarized light microscopy which is based upon the gravimetric Point Count method.

### **Scope / Field of Application**

Materials which are suitable and requested for analysis by a client to use Gravimetric Point Count method.

### **Analytical Hardware requirements**

#### **A. Apparatus for Gross Examination**

1. Magnifying Lens, 10X
2. Light Source: fluorescent and incandescent lights
3. Hand Tools: tweezers, scalpel, razor blades, probes, etc.
4. Petri Dish: clean glass plate

#### **B. Apparatus for Ashing and Acid Digestion and Filtration**

1. Crucibles and lids
2. Balance
3. Furnace
4. HCL acid
5. Distilled water
6. 50ml plastic tube
7. Ultrasonic Cleaner
8. PC filters
9. Plastic Petri dish

#### **C. Apparatus for Sample Preparation**

1. Filtered Ventilation System
2. Microscope Slides
3. Cover Slips
4. Disposable Gloves
5. Hand Tools
6. Mortar and Pestle
7. Hot Plate
8. Cigarette Lighter

#### **D. Apparatus for Identification and Quantification**

1. Polarized Light Microscope



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- a. Polarizer
- b. Analyzer
- c. Port for Wave Retardation Plate
- d. 360° Graduated Rotating Stage
- e. Substage Condenser
- f. Lamp
- g. Lamp Iris
- h. Condenser Diaphragm
- i. Objective Lenses - 4X, 10X, 10X Central Stop Dispersion Staining, 20X, 40X,
- j. Ocular Reticule (10X) w/ Cross Hair
- k. Retardation Plate - First Order Red, 550 nm

#### **E. Reagents for Sample Preparation**

1. Refractive Index Liquids:  
1.490-1.570 and 1.590-1.720 in 0.002 or 0.004 Step Increments
2. Distilled Water
3. Mineral Oil
4. Dilute HCl acid

#### **F. Analytical Standard Reference Materials**

1. U.C. Berkeley Mineral Collection Reference Standards
2. NIST Asbestos PAT Round Reference Sample Sets

## **Analytical Procedures**

The analytical procedures of Gravimetric Point Count analysis is similar to a regular PLM 400 Point Count analysis except the sample needs to be ashed, digested and filtered before a regular 400 Point Count analysis can be performed. The sample ashing, digestion and filtration procedure is described below. Refer to SOP # 5-4-2-PLM-02 section 'Analytical Procedures' for the rest of the analytical procedures

### Gross Examination of the Sample

Refer to the same section in SOP # 5-04-2-PLM-01 on Page 3.

### Sample Ashing, Digestion and Filtration Procedure for PLM Gravimetric Point Count Analysis

#### **I. Ashing**

- a). Clean and dry crucibles and lids.
- b). Write down the crucible # on the Gravimetric Point Count data sheet (see Appendix)
- c). Turn on the balance and set 'zero'
- d). Weigh the crucible not including lid and write down the weight on the data sheet



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- e). Place sample into the crucible (use about 50mg material), then weight and write down the total weight of crucible plus sample on the data sheet
- f). Cover the crucible with a lid and place aside
- g). Repeat steps **b** through **f** for all samples
- h). Put all the crucibles with samples in the furnace, close the furnace door and set the knob at 0.8 position that corresponds to 480°C
- i). Ash samples overnight or at least 6-8 hours
- j). Turn off the furnace, open the door and let the samples cool down to the room temperature
- k). Weigh the crucible plus the ashed sample (without lid) and write down the weight on the data sheet

#### II. Acid (HCL) Digestion and Filtration

- a). Add 2-3 drops of HCL into crucible, stir and rinse the crucible with distilled water 3 times into a 50ml plastic tube. Rinse the lid
- b). Bring up the volume to 50 ml by adding more distilled water into the plastic tube. Cover the tube with cap. Number the tube and the cap
- c). Repeat steps **a** and **b** for all samples
- d). Ultrasonic solutions for 15 minutes to break up the solid materials as much as possible
- e). Take a small plastic Petri dish and mark the sample # on the top and the bottom.
- f). Weigh the Petri dish without the cover part and write down the weight on the data sheet
- g). Re-zero the scale, put PC filter into the Petri dish, weight the filter and write down on the data sheet
- h). Repeat steps **e** through **g** for all samples
- i). Set up the filtration system. Use preweighted PC filter (shiny side up)
- j). Shake tubes well and filter whole solution. Rinse tubes with water
- k). Place filters into the preweighted Petri dishes with appropriate sample numbers
- l). Dry filters under the lamp if needed
- m). Weigh the dry filters+Sample+Petri Dish system and write down the weight of filter plus sample part on the data sheet (To obtain the weight of filter plus sample, subtract the Petri dish weight in step **f** from the total weight)

#### III. Samples are now ready for regular PLM 400 Point Count analysis

Refer to the following sections in the SOP # 5-04-2-PLM-01 for the rest of the analytical procedure:

E. Fiber Identification (page 6)

F. Quantification of Sample Contents (2. Point Count Method, page 8)

#### IV. Calculation of the Percentage of the Asbestos

The final percentage of asbestos needs to be normalized to 100% of the original material by considering the percentage of the insoluble Non-Asbestos Inorganic component. For instance, if the percentage of the asbestos calculated using the regular 400 point count method is 5%, and the insoluble Non-Asbestos Inorganic component is 90%, then the actual asbestos percentage will be  $(5 \times 90 / 100)\% = 4.5\%$ .



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### **Report Generation & Data Review**

Refer to the same section in SOP # 5-04-2-PLM-01 on Page 9.

### **Common Diagnostic Optical Tests and Related Principles Regarding PLM Analysis for Asbestos**

Refer to the same section in SOP # 5-04-2-PLM-01 Pages 10 – 21.

### **Characteristic Optical Properties of the Six Asbestos Minerals**

Refer to the same section in SOP # 5-04-2-PLM-01 Pages 23 – 29.

### **Characteristic Optical, Physical, & Chemical Properties of Common Asbestos Look-Alike Minerals & Materials**

Refer to the same section in SOP # 5-04-2-PLM-01 Pages 31 – 40.

### **Characteristic Optical, Physical, & Chemical Properties of Other Common Minerals & Materials Found in Building Materials**

Refer to the same section in SOP # 5-04-2-PLM-01 Pages 41 – 44.

### **Reference Procedures**

SOP # 5-04-2-PLM-01

### **Revision History**

Revision	Date	Revision Notes
1	Dec. 16, 2004	Format and Wording

Approval

**Yanxia Xie**

Reviewer

**12 / 16 / 2004**

Date

**Yanxia Xie** Init: \_\_\_\_\_

Quality Assurance Manager

**12 / 16 / 2004**

Date





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### **Appendix Gravimetric Point Count Data Sheet**



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### Gravimetric Point Count Data Sheet

Analyst: \_\_\_\_\_ Analysis Date: \_\_\_\_\_ Sample ID: \_\_\_\_\_

### Steriobinocular Microscopy

Color: \_\_\_\_\_ Texture: \_\_\_\_\_ Homogeneity: \_\_\_\_\_ Homogenization: \_\_\_\_\_  
Probable Fibers: \_\_\_\_\_ Remark: \_\_\_\_\_

### Matrix Reduction

Ashing:

Time Start: \_\_\_\_\_ Time Finish: \_\_\_\_\_ Ashing Temperature: \_\_\_\_\_

Weights:

A. Crucible: \_\_\_\_\_

B. Crucible + Sample: \_\_\_\_\_

C. Crucible + Ashed Sample: \_\_\_\_\_

Acid Digestion and Filtration:

Weights:

D. Filter: \_\_\_\_\_

E. Filter + Sample: \_\_\_\_\_

Calculations	Formula (g)	Results (g)	Formula (%)	Results (%)
Untreated sample	$F=B-A$		$G=100\%$	
Organic Component	$H=F-(C-A)$		$I=H*G/F$	
Acid-insoluble inorganic component	$J=E-D$		$K=J*G/F$	
Acid-soluble inorganic component	$L=F-(J+H)$		$M=L*G/F$	

Remark: \_\_\_\_\_

### Polarized Light Microscopy

Refractive index liquid used:

Prep #	1	2	3	4	5	6	7	8
CHRY								
AMPH								
Other								

Quantification: \_\_\_\_\_

### Final Results:

Component	Asbestos 1:	Asbestos 2:	Organic	Acid-Soluble Inorganic	Insoluble Non-Asbestos Inorganic
Percentage					



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## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Estimation of Uncertainty**

Issue Date: 2004/12/16

Revision: 0

SOP#: 5-04-6-01

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#### **Purpose**

To estimate the uncertainty of measurement in a test method.

#### **Scope / Field of Application**

All measurements from test methods, except when the test methods preclude such rigorous calculations.

**Note** - it is important to understand the major factors of uncertainty and provide appropriate control for all such factors. Concurrent analysis of reference materials or control samples with the test portion can be performed in place of purely mathematical estimation of uncertainty. If possible, the reference material or control sample shall be of identical or similar matrix as the matrices routinely tested by the test method. The uncertainty of the method can be estimated for the class of matrix and the variation described as the uncertainty in testing the specific matrix class at the average amount of analyte detected.

#### **Definitions and Acronyms**

**Type A Uncertainty** – determined through calculation from a series of repeated observations using statistical methods.

**Type B Uncertainty** – determined through judgment, based on data in calibration certificates, previous measurement data, experience with the behavior of the instruments, manufacturer's specifications, and all other relevant information.

**Measurement Uncertainty** – parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.

#### **Responsibilities**

Technical Manager ensures that this procedure is utilized where appropriate.

#### **Materials Required**

Electronic spreadsheet capabilities

#### **Procedure**

##### ***Step 1. Specification***



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Write down a clear statement of what is being measured, including the relationship between the measurand and the parameters (e.g., measured quantities, constants, calibration standards, etc.) upon which it depends. Where possible, include corrections for known systematic effects. The specification information, if it exists, is normally given in the relevant standard operating procedure (SOP) or other method.

#### ***Step 2. Identify Uncertainty Sources***

List the possible sources of uncertainty. This will include sources that contribute to the uncertainty on the parameters in the relationship specified in Step 1, but may include other sources and must include sources arising from chemical assumptions. Examples for forming a structured list are given in the appendix.

#### ***Step 3. Quantify Uncertainty Components***

Measure or estimate the size of the uncertainty component associated with each potential source of uncertainty identified. It is often possible to estimate or determine a single contribution to uncertainty associated with a number of separate sources. It is also important to consider whether available data accounts sufficiently for all sources of uncertainty, and plan additional experiments and studies carefully to ensure that all sources of uncertainty are adequately accounted for.

#### ***Step 4. Calculate Total Uncertainty***

The information obtained in step 3 will consist of a number of quantified contributions to overall uncertainty, whether associated with individual sources or with the combined effects of several sources. The contributions have to be expressed as standard deviations, and combined according to the appropriate rules, to give a combined standard uncertainty. The appropriate coverage factor should be applied to give an expanded combined uncertainty.

### **Alternate Procedure**

Concurrently analyze reference materials or control samples with test samples to estimate uncertainty. This is generally achieved during validation of methods.

### **Documentation**

The following records are generated and managed:

Required Record	Custodian
Measurement Uncertainty	Laboratory Manager / Supervisor



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#### **Reference Procedures**

All test methods requiring uncertainty estimation

#### **References**

ISO "Guide to the Expression of Uncertainty in Measurement".

Eurachem / CITAC. Quantifying Uncertainty in Analytical Measurement (2<sup>nd</sup> Edition).

#### **Revision History**

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0	Dec. 16, 2004	Initial Publication

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***12 / 16 / 2004***

Date

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Quality Assurance Manager

***12 / 16 / 2004***

Date



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#### **Appendix**

Several example calculations are given below:



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### Example 1. Bacteria Count

All standard deviations expressed in terms of bacteria count units

Source of Uncertainty	Type A evaluation Standard deviation	Type B evaluation Standard deviation	Variance ( $S^2$ )	Notes
Uncertainty of wt used to calibrate balance		10	100	Obtain from weight calibration certificate
Uncertainty of pipette reference std		25	625	Obtain from pipette reference standard calibration certificate
Reproducibility (people) variation in count observed		200	40000	Type B estimate of uncertainty based on experience of tester
Balance Uncertainty	70		4900	Repeated measurements on balance
Pipette uncertainty	120		14400	
Environmental (Temperature)		200	40000	Estimate of variation in bacteria growth due to slight temperature change

$$\text{Total } S^2 = 100,026$$

$$\sqrt{S^2} = 316.27 \quad \text{Combined uncertainty}$$

$$\text{Times Coverage Factor} \quad \times \quad 2$$

$$\text{Expanded Uncertainty} = 632.54$$



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#### Example 2. Quantification of Sulfamethazine

All standard deviations expressed in terms of parts per billion

Source of Uncertainty	Type A evaluation Standard deviation	Type B evaluation Standard deviation	Variance ( $S^2$ )	Notes
Uncertainty of wt used to calibrate balance		1	1	Obtain from weight calibration certificate
Uncertainty of pipette reference std		3	9	Obtain from pipette reference standard calibration certificate
Reproducibility (people) variation in count observed		10	100	Type B estimate of uncertainty based on experience of tester
Balance uncertainty	5		25	Repeated measurements on balance
Pipette uncertainty	10		100	
Calibration curve		2	4	

$$\text{Total } S^2 = 239$$

$$\sqrt{S^2} = 15.46 \quad \text{Combined uncertainty}$$

$$\text{Times Coverage Factor} \quad \underline{\quad x \quad} \underline{\quad 2 \quad}$$

$$\text{Expanded Uncertainty} = 30.92$$





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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Analyst Proficiency Control**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-04-6-02**

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#### **Purpose**

To determine the accuracy and precision of the analysis performed

#### **Scope / Field of Application**

This procedure applies to all PLM analysts employed by Asbestos TEM Laboratories, Inc.

#### **Definitions and Acronyms**

**Accuracy** – the closeness of agreement between a test result and the accepted reference value (ISO 5725-1, ISO Guide 30)

**Precision** – the closeness of agreement between test results obtained under stipulated conditions (ISO 5725-1, ISO Guide 30).

#### **Responsibilities**

Technical Manager ensures that this procedure is utilized where appropriate.

#### **Materials Required**

Electronic spreadsheet capabilities

#### **Procedures**

##### Qualifications

All analysts have four-year college degrees in the physical sciences, with some academic training in optical mineralogy. The qualifications of current employees are given in the PLM staff sections of the PLM QA Manual (the large binder housing this document).

##### Training

Appendix in SOP # 5-02-1 describes the training process. It is designed to take new analysts of varying backgrounds and bring them up to the level of quality set forth in the PLM Laboratory. In addition, the training process promotes independent, logical assessment of difficult samples and an ability to go beyond a simple "by the book" attitude.

##### Performance checks

Periodic informal reviews of each analyst's technique and ability to adhere to the procedures are conducted by the QA Officer. These reviews include:



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#### a). Assessment of Analyst Accuracy -

The accuracy of a given analyst is a measure of the closeness with which the analyst is able to precisely measure the absolute concentration of asbestos in a sample. This QA test is performed by having the analyst analyze known, previously quantified NIST standards on a periodic basis and to chart the difference between the values obtained by the analyst to the characterized NIST values. The best way to track the accuracy of the analyst is to make two types of graphs as discussed below:

i) A normalized graph of the difference between the analyst's results and the "true" value of the NIST characterized samples versus the date of the analysis. Use the following formula:

$$AN = \text{ABS}(\text{Measured}\% - \text{NIST}\%) / \text{NIST}\%$$

where **AN** is the *normalized Accuracy*

**Measured%** is the *percent asbestos measured by the analyst for the sample on the date indicated*

**NIST%** is the *"true" asbestos content of the sample*

***Asbestos TEM Laboratories will have any analyst who obtains a value of AN greater than 1.0 for any sample review the sample with the QA Supervisor to see if the analyst is having problems with their quantitation skills, or to verify inhomogeneity problems in the sample.***

ii) A normalized graph of the average difference between the analyst's results and "true" value of the NIST characterized samples versus the date of the analysis. Use the following formula:

$$\bar{AN} = ((\sum_{i=1 \text{ to } n} \text{ABS}(\text{Measured}\%_i - \text{NIST}\%)) / \text{NIST}\%) / n$$

where  **$\bar{AN}$**  is the *normalized average Accuracy*

**Measured%<sub>i</sub>** is the *percent asbestos measured by the analyst for the sample for the *i*th time on indicated date*

**NIST%** is the *"true" asbestos content of the sample*

***Asbestos TEM Laboratories will have any analyst who maintains a value of  $\bar{AN}$  greater than 0.75 meet with the QA Supervisor to review their procedures to work to correct any problems that the analyst may be having with their quantitation skills.***

Over a period of time a convergence should be expected, resulting in a well "calibrated" analyst - See accompanying example chart. However, virtually all bulk materials have some inherent heterogeneity and, therefore, perfect agreement cannot be expected.



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### Analyst Proficiency Control

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Performance shall occur such that at least 2 NIST samples will be reviewed each month by each analyst.

b) Assessment of Analyst Precision -

The precision of a given analyst is a measure of the ability of a given analyst to reproduce his/her measurements of the asbestos concentration of a given sample. This QA test is performed by having the analyst analyze known, previously quantified NIST standards on a periodic basis and to keep a chart of the values measured by the analyst and compare these results to the NIST values. The best way to track the precision of the analyst is to make two types of graphs:

i) A normalized graph of the difference between the analyst's test results on a NIST characterized sample on date  $i$  and the previous analytical result obtained on the same sample by the same analyst on date  $i-1$ . This normalized difference is plotted versus the date of the analysis. Use the following formula:

$$P_N = \text{ABS}(\text{Measured}\%_i - \text{Measured}\%_{i-1}) / ((\text{Measured}\%_i + \text{Measured}\%_{i-1}) / 2)$$

where  $P_N$  is the *normalized Precision*

**Measured%** $_i$  is the *percent asbestos measured by the analyst for the sample for the  $i$ th time on indicated date*

***Asbestos TEM Laboratories will have any analyst who obtains a value of  $P_N$  greater than 0.75 for any sample review the sample with the QA Supervisor to see if the analyst is having problems with their quantitation skills, or to verify inhomogeneity problems in the sample.***

ii) A normalized graph of the average difference between the analyst's test results on a NIST characterized sample on date  $i$  and the previous analytical result obtained on the same sample by the same analyst on date  $i-1$ . This average normalized difference is plotted versus the date of the analysis. Use the following formula:

$$\overline{P}_N = \left( \sum_{i=2 \text{ to } n} \text{ABS}(\text{Measured}\%_i - \text{Measured}\%_{i-1}) / ((\text{Measured}\%_i + \text{Measured}\%_{i-1}) / 2) \right) / (n-1)$$

where  $\overline{P}_N$  is the *normalized average Precision*

**Measured%** $_i$  is the *percent asbestos measured by the analyst for the sample for the  $i$ th time on the date indicated*

***Asbestos TEM Laboratories will have any analyst who maintains a value of  $P_N$  greater than 0.5 meet with the QA Supervisor to review their procedures to work to correct any problems that the analyst may be having with their quantitation skills.***



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If the analyst has good precision, then the variability between analyses should be low. However, virtually all bulk materials have some inherent heterogeneity and, therefore, perfect agreement cannot be expected. Performance shall occur such that at least 2 NIST samples will be reviewed each month by each analyst.

#### Documentation

The following records are generated and managed:

Required Record	Custodian
Analysts' Proficiency Control Charts	Laboratory Manager
Analysts' Accuracy Control Charts	Laboratory Manager

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0	Dec. 16, 2004	Initial Publication

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12 / 16 / 2004

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Yanxia Xie Init: \_\_\_\_\_

Quality Assurance Manager

12 / 16 / 2004

Date



Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

**Polarized Light Microscopy Division**

**Laboratory Proficiency Control**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-04-6-03**

Page #: **1 of 4**

### **Purpose**

This section describes the quality control procedures applied by Asbestos TEM Laboratories to assure and improve the quality of the PLM laboratory analytical service.

### **Definitions and Acronyms**

**Quality Control** – the operational techniques and activities that are used to fulfill requirements for quality (ISO 8402).

### **Responsibilities**

Laboratory Manager ensures that this procedure is utilized where appropriate.

### **Materials Required**

Electronic spreadsheet capabilities

### **Procedures**

#### General

Quality Control procedures consist of repeating analyses internally, or checking results against those of known standards (i.e. NIST or AIHA Reference Materials or Proficiency Testing Materials) or other labs. These procedures serve three purposes. First, difficult samples may be examined by multiple analysts before a result is reported, improving the quality of the results. Second, any shortcomings in the techniques used by individual analysts or the lab as a whole can be identified and corrected. Finally, the level of accuracy achieved by the laboratory can be determined.

Samples re-analyzed for Quality Control represent at least 10% of all samples analyzed at Asbestos TEM Laboratories. QC analyses are conducted on a routine basis as set forth in the QA procedures schedule. Analysts are responsible for identifying difficult or unusual samples and requesting QC analyses by making notations on the PLM analysis data sheets. The QA Officer is responsible for selecting random samples for QC analysis and seeing that enough QC analyses are done. The selection of samples subjected to QA is semi-random, i.e., difficult or extraordinary samples will be subject to QC very often, while routine samples will be subject to less frequent random checks. In order to efficiently recognize and correct analytical errors, it is important to perform regular and timely QC checks on the analytical system. Asbestos TEM Labs reviews, and if necessary performs, QC analyses at least weekly to maintain an up to date.



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Monthly QC summaries are compiled to track lab and analyst accuracy. Quality Control also encompasses proficiency testing. The NIST PAT program and the analysis of known reference materials are effective checks on the accuracy of analyses.

Laboratory Blanks are described in the SOP # 5-04-6-04 on page 2, as they serve as a preventative measure as well as a means of detecting errors.

#### Inter-analyst comparisons

Samples are re-prepared and re-analyzed by one or more analysts other than the original analyst. At least half of the QC analyses should be of this type, as it gives a better measure of quality than same-analyst or same-prep QC.

#### Same-analyst comparisons

Samples re-prepared and re-analyzed by the same analyst at a later time.

#### Inter-analyst, same-prep

An analyst may have the slide he/she worked on analyzed by a different person. Note that this does not take into account variations due to sample heterogeneity.

#### Inter-Laboratory

Selected samples are exchanged with other laboratories and the results are compared. Any discrepancies in presence / absence or type of asbestos are immediately investigated and the cause identified and recorded in the laboratory QA records. Discrepancies in *the amounts* of asbestos present are recorded and actions are taken to reconcile any consistent differences in quantification.

- 1) From other labs. In the first type of inter-laboratory QC check, samples originally analyzed by another lab are run by one or more of our analysts. Normal turnaround is expected. The original results sent by the other lab are entered into the relevant monthly PLM QC report.
- 2) To other labs. In the second type of inter-laboratory QC check, selected samples are sent to other labs (a portion of each sample should be kept at our lab). When results are received, the QA Officer is responsible for entering them into the relevant monthly QC report.

#### Intra-laboratory Proficiency Testing



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Known standard materials from the store of NIST characterized asbestos samples are chosen by the Laboratory Manager and are analyzed as a test of the analysts' skills. Intra-laboratory proficiency testing is conducted at least quarterly for each analyst. Materials tested include known asbestos mineral standards as well as commonly encountered non-asbestos substances. Asbestos TEM Laboratories has an extensive collection of bulk samples and permanent mounts of minerals, mineraloids, inorganic amorphous substances, and organic substances commonly found in bulk materials. All six asbestos minerals are represented, as well as non-asbestiform serpentines, amphiboles, other asbestos look-alike minerals, fibrous glass, and cellulose. The provenance and description of each standard sample are documented. Sources include: government agencies (e.g., NIST, 1988a); bulk samples received from clients; academic institutions; other laboratories; and natural outcrops of various rock types.

### NIST Proficiency Analytical Testing (PAT)

The laboratory shall participate in the mandatory NVLAP proficiency testing program. In accordance with the being involved in the testing program, the lab shall:

- 1) Perform all proficiency testing in-house with in-house personnel. Analyses are not to be contracted out to another lab.
- 2) All Asbestos TEM Laboratories analysts participate in each round of the NIST proficiency testing programs. Each analyst independently analyses, and reports results for, each sample.  
The results are then used for inter-analyst comparisons, to determine the precision and accuracy for each analyst, and to assist the analyst in calibrating the quantitation values which he/she reports.
- 3) A single result is reported back to NIST, with documentation as to how the final results were obtained. The instructions that come with the samples are carefully followed, and the reporting forms are carefully completed and checked for accuracy.
- 4) Problems indicated by proficiency testing are discussed with appropriate laboratory personnel and documented.
- 5) Plans are developed and implemented for resolving problems and are documented
- 6) NIST proficiency materials are kept for use as standards.
- 7) No communication between any other asbestos analytical laboratory, or other consultant outside of Asbestos TEM Laboratories' (Berkeley) pool of in-house PLM analytical personnel, shall occur during the time Asbestos TEM Laboratories has an active set of NIST/NVLAP proficiency testing samples which have not been reported as final results to NIST/NVLAP. The proficiency samples are to be analyzed by Asbestos TEM Laboratories in-house staff alone, with no outside assistance.  
Furthermore, as Asbestos TEM Laboratories has two offices (one in Berkeley, CA and one in Sparks/Reno, NV) which are designated as separate accredited asbestos labs by NIST/NVLAP, no communication about the analytical results of a given NIST/NVLAP proficiency testing round shall occur until both labs have independently analyzed the samples and submitted their individual final results to NIST/NVLAP.



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### Documentation

The following records are generated and managed:

Required Record	Custodian
PLM Monthly QC Records	Quality Manager
NIST PAT Records	Senior PLM Analyst
Analysts' Proficiency Tests Records	Laboratory Manager

### Reference Procedures

SOP # 5-04-6-01 Uncertainty

SOP # 5-04-6-02 Analyst Proficiency Control

SOP # 5-04-6-04 Contamination Control

### References

ISO "Guide to the Expression of Uncertainty in Measurement".

Eurachem / CITAC. Quantifying Uncertainty in Analytical Measurement (2<sup>nd</sup> Edition).

### Revision History

Revision	Date	Revision Notes
0	Dec. 16, 2004	Initial Publication

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12 / 16 / 2004

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Quality Assurance Manager

12 / 16 / 2004

Date





Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Contamination Control**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-04-6-04**

Page #: **1 of 3**

#### **Purpose**

This section is to describe the plans and actions taken by the Asbestos TEM Laboratories, Inc. to avoid and detect the presence of asbestos contamination so as to avoid health risk or erroneous analytical results.

#### **Responsibilities**

Quality Manager ensures that this procedure is utilized where appropriate.

#### **Materials Required**

Electronic spreadsheet capabilities

#### **Procedures**

Unless great care is taken, asbestos from a sample can cause a health risk or can be transferred to another sample via the refractive index oils, the tools used in sample prep, the air and other sources, and can lead to erroneous results. Several measures are taken during sample preparation to ensure that samples will not be contaminated with asbestos or other foreign materials.

##### Maintenance of Clean Conditions in the PLM Laboratory

The PLM laboratory is to be kept clean at all times. This is important as a step in keeping the TEM lab, as well as the PLM lab, contamination-free. Specific measures taken are:

- a) The area under the hood is constantly cleaned during analysis and any spills are cleaned immediately.
- b) The floor is wet-mopped at the end of each work day. All surfaces are thoroughly wetwiped at the end of each workday. The last analyst in the lab or the Quality Manager is responsible for seeing that this is done.
- c) All persons involved in analysis and cleaning shall wash their hands immediately after such activities.
- d) Dry-sweeping, or any other activities that may result in the increase of airborne fiber concentrations are absolutely prohibited.
- e) A HEPA filter mask is available in the PLM laboratory for ready use in the event of selfcontamination of personnel.

##### Air Monitoring (Responsibility of the Quality Manager)



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As a check on the cleanliness and safety of the laboratory, air samples are collected in the PLM laboratory on a routine basis (quarterly or more often as needed). The air samples are analyzed by transmission electron microscopy (TEM). If the airborne asbestos concentration exceeds 0.01 f/cc, the PLM lab will be thoroughly cleaned and re-tested. If the problem continues, the HEPA hood will be checked for damage and the filter element replaced if necessary.

#### Handling of Samples During Analysis

- a) Opening of Samples. Bulk materials are removed from their original containers only under the negative air flow HEPA hood, without exception. Samples are opened one at a time to avoid cross contamination.
- b) Slides, tools, and other items are not allowed to leave the hood if sample dust is visible on them.

#### Laboratory Blanks

Lab blanks are designed to detect contamination from the refractive index oils, the sample prep tools, the air, or other sources. Lab blanks are always run using an **asbestos-free** material standard. It is recommended that a material such as cornstarch be used as it provides a good focusing medium, is fiber free, and enables easy detection of small amounts of asbestos contamination.

- a) Procedure. Laboratory blanks are prepared in exactly the same way as regular samples, but using instead the **asbestos-free** material. Refractive index oil is placed on a slide, chopped and homogenized using the regular preparation tools and materials. Unless otherwise noted, a blank analysis comprises tests of refractive index oils, distilled water, HCl acid, other preparation tools, cover-slips, and slide. Analyses of materials certified to contain no asbestos may also be used as laboratory blank analyses.
- b) Corrective action. If the blank is positive for asbestos, the Quality Manager is notified, and the following must be checked by running additional blanks and cleaned if necessary: slides and coverslips; tools; oil containers and eye droppers; rubber gloves; Petri dishes, mortars, pestles, and other materials. Any material that shows persistent contamination should be discarded in a hazardous waste container.
- c) Frequency. Laboratory blanks should be tested once every day, assuming samples are tested during the week. The analysts are responsible for seeing this is done.
- d) Recording of results. The results of blanks are recorded in the Laboratory Database System.



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### Documentation

The following records are generated and managed:

	Custodian
Air Monitoring Records	Laboratory Manager
Laboratory Blank Tests	PLM Analysts

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## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Equipment (Including Computers)**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-05-1-01**

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#### **Purpose**

To establish a plan for safe handling, transport, storage, use and maintenance (including calibration) of measuring equipment, and appropriate use of correction factors to ensure proper functioning and in order to prevent contamination or deterioration.

#### **Scope / Field of Application**

Chemistry and / or microbiology equipment generally includes the following types:

- a) *General Service Equipment* – not used for making measurements or with minimal influence on measurements (e.g., hot plates, stirrers, non-volumetric glassware and glassware used for rough volume measurements such as measuring cylinders) and laboratory heating or ventilation systems.
- b) *Volumetric Equipment* – flasks, pipettes, burettes.
- c) *Measuring Instruments* – thermometers, timers, spectrometers, chromatographs, electrochemical meters, balances, etc.
- d) *Computers and Data Processors* – physical equipment and software.

#### **Responsibilities**

The performance of an instrument is checked out and appraised by a qualified person before use. This involves a visual inspection and verification of its operation, including the zero and full-scale calibration.

#### **Materials Required**

The equipment and the required calibration and maintenance items as specified by the manufacturer.

#### **Procedure**

##### ***Overall Requirements***

Generally, the handling, transport, storage, use, and maintenance of equipment is outlined in the manufacturer's manual. Specific requirements are outlined in a standard operating procedure for the instrument or equipment type.



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### **Equipment (Including Computers)**

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All handling, transport, storage, packaging, preservation, and delivery of equipment is verified by laboratory personnel using the appropriate standard operating procedures or manufacturer's specifications.

The manufacturer's manual is critical in describing the safe handling requirements of the equipment, to avoid any damage, alteration, contamination, change of integrity or reliability and condition of the equipment (or samples). The manufacturer's manual also provides guidance for suitable environmental conditions for the calibrations, inspections, measurements and tests performed.

To ensure the proper environment is maintained to prevent contamination or deterioration, the laboratory is monitored on a monthly basis, as appropriate to the analysis (e.g., monitor airborne and surface microbial contamination using air samplers, settle plates, contact plates or swabs).

Pre- and post-testing checks verify the performance of an instrument during its operation and could reveal the occurrence of measurement drift.

Laboratory personnel utilize the appropriate correction factors to ensure proper functioning of equipment.

#### ***General Equipment***

General service equipment is maintained by performing cleaning and safety checks as necessary. Calibrations or performance checks will be necessary where the setting can significantly affect the test or analytical result (e.g., the temperature of a muffle or constant temperature bath).

#### ***Volumetric Equipment***

The correct use of volumetric equipment is critical to analytical measurements and is suitably maintained and calibrated as specified in laboratory procedures. The correct functioning of some specialist volumetric (and related) glassware is dependent on 'wetting' and surface tension characteristics, which may be affected by cleaning methods. Such an apparatus may therefore require more regular calibration, depending on use. For the highest accuracy, measurements can often be made by mass rather than by volume.

Attention is paid to the possibility of contamination arising from the equipment or cross-contamination from previous use. The type used, cleaning, storage and segregation of volumetric equipment is critical, particularly for trace analyses when leaching or adsorption can be significant.

#### ***Measuring Equipment***

Correct use combined with periodic servicing, cleaning and calibration will not necessarily ensure an instrument is performing adequately. Where appropriate, periodic performance checks are carried out (e.g., to check response, stability and linearity of sources, sensors and detectors,



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the separating efficiency of chromatographic systems, the resolution, alignment and wavelength accuracy of spectrometers).

The frequency of such performance checks is determined by experience and based on need, type and previous performance of the equipment. Intervals between checks are shorter than the time the equipment has been found to take to drift outside acceptable limits.

It is often possible to build performance checks – system suitability checks – into test methods (e.g., based on the levels of expected detector or sensor response to calibration standards, the resolution of calibration standards in separating systems, the spectral characteristics of calibration standards etc). These checks are completed before the equipment is used.

The standardization of instruments is performed using reference standards when these are available, or against certified standard instruments when they are not. This is done before the instrument is used.

Calibrations are conducted under the same instrumental and chemical conditions as those that will exist during the measurement process. The frequency of calibration depends on the accuracy requirements of the investigation and the stability of the instruments. Daily calibration checks are recommended when the instrument is in daily use; calibration checks are performed immediately prior to a series of measurements at other times. For unstable instruments, the calibration is checked prior to each series of measurements, in between measurements, and after the last measurement.

The calibration process is vital to all measurement programs and is governed by a calibration plan. The calibration plan provides for:

- calibration procedures and record forms
- stated calibration frequencies
- appropriate sources for obtaining certified and high quality standards, or the best means of producing accurate in-house standards
- a list of all calibration standards (including nomenclature and assigned identification numbers)
- specifications of environmental conditions
- intended range of validity

Calibration procedures include information on the following:

- the specific equipment or groups of equipment to which the procedure is applicable
- a brief description of the scope, principle, or theory of the calibration method (an example and a reference may also be included)
- calibration specifications, such as the number of calibration points, environmental requirements, and precision and accuracy requirements
- a list of the calibration standards and accessory equipment needed to perform an effective calibration, manufacturer's name, and instrument model number



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## Equipment (Including Computers)

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- a complete, clear, concise, step-by-step written calibration procedure
- specifications for calibration facilities, equipment, temperature, and humidity, and physical protection for calibration standards
- specific instructions for obtaining and recording the test data (includes data collection forms)

### *Computers and Data Processors*

1. Operating manuals and supplementary procedures are available to operators.
2. Deviations from established procedures are documented to an extent appropriate to repeat the procedures at a later date.
3. Special procedures relating to security and file management (including archiving, file repair, file back-ups) are outlined in SOP# 4-12-1.
4. The computers and their software are considered validated when correct operation (or expected answer) occurs after the input of well-characterized parameters. This is known as a black-box system. The degree of validation necessary depends on the exact use of the computer. Consider testability, traceability, maintainability, and repeatability.
5. When software is updated, a record is kept of the revision history.

### **Documentation**

Documents on standardization, calibration, maintenance, equipment safety, and spare parts accompany each instrument.

Equipment inventory includes the following information:

- name
- manufacturer
- serial number
- model number
- company asset number
- date received
- date placed in service
- current location
- condition when received (new, used, reconditioned)
- manufacturer's manuals and location
- calibration period
- calibration records and location
- maintenance records and location

### **Reference Procedures**

Equipment manuals

SOP 5-05-1-02 Microscope Maintenance



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# Standard Operating Procedure

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## Equipment (Including Computers)

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Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.

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## Equipment (Including Computers)

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### Appendix

#### Calibration and Maintenance Matrix

Equipment	Requirements	Frequency
Balances	1. Linearity, zero point, and accuracy (using standard weights) 2. Clean 3. Service	Daily / each use  Daily / each use Annually
Volumetric glassware	1. Accuracy, precision (pipettes/burettes) 2. Clean and/or sterilize	Depends on use  Each use
Hydrometers (working)	One point calibration versus reference hydrometer	Annually
Barometers	One point calibration using standard of known specific gravity	5 years
Timers	Accuracy	2 years
Thermometers (working)	Check specific points against reference thermometer	Annually
Chromatographs (general)	1. Overall system checks, precision of repeat sample injections, carry-over 2. Column performance (capacity, resolution, retention) 3. Detector performance (output, response, noise, drift, selectivity, linearity) 4. System heating / thermostating (accuracy, precision, stability, ramping characteristics) 5. Autosampler (accuracy and precision of time routines)	Daily / each use
Liquid and ion chromatography	1. Composition of mobile phase 2. Mobile phase delivery system (precision, accuracy, pulse-free)	Daily / each use
Electrode/meter systems, including conductivity, pH and ion-selective	1. Electrode drift or reduced response 2. Fixed point and slope checks using standard solutions 3. Clean electrode	Daily /each use
Temperature controlled equipment (refrigerators, incubators, etc.)	1. Periodic calibration of temperature sensing system using the appropriate standard thermometer 2. Thermal stability, reproducibility 3. Heating / cooling rates and cycles 4. Ability to achieve and sustain pressure or vacuum 5. Monitor temperature 6. Clean and disinfect internal surfaces	Annually  Annually  Annually  Daily Weekly
Spectrometers (atomic absorption,	1. Selected wavelength accuracy,	Each use



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Equipment	Requirements	Frequency
fluorimetric, inductively coupled plasma – optical emission, infra-red, luminescence, mass, nuclear magnetic resonance, ultra-violet/visible and x-ray fluorescence	precision, stability 2. Source stability and blank reading 3. Detector performance (resolution, selectivity, stability, linearity, accuracy, precision) 4. Signal to noise ration 5. Detector calibration (mass, ppm, wavelength, frequency, absorbance, transmittance, bandwidth, intensity) 6. Internal temperature controllers and indicators where applicable 7. Clean	Each use Each use Each use Each use Each use Each use As recommended by manufacturer
Microscopes	1. Resolving power 2. Graticule calibration (for length measurement) 3. Clean 4. Full maintenance 5. Check alignment	Annually Annually Daily / each use Annually Daily / each use
Sterilizing oven	1. Establish stability / uniformity 2. Monitor temperature	Initially and after repair Each use
Autoclave	1. Establish characteristics for typical load / cycle 2. Monitor temperature / time (with spore vials or strips) 3. Visual checks of gasket, clean / drain chamber 4. Full service 5. Safety check of pressure vessel	Initially and after repair Each use (and weekly) Each use Annually Annually
Safety cabinet	1. Establish performance 2. Particle count monitoring 3. Air flow monitoring 4. Full service and mechanical check	Initially and after repair Weekly Monthly Annually
Laminar air flow cabinet	1. Establish performance 2. Sterility plates 3. Service and mechanical check	Initially and after repair Weekly Annually
Still deionizer and reverse osmosis unit	1. Conductivity 2. Microbial contamination 3. Clean or replace cartridge / membrane	Daily Monthly As recommended by manufacturers
Gravimetric diluters	1. Weight and volume (weight) dispensed 2. Check dilution ratio 3. Clean 4. Service	Daily Monthly Daily / each use Annually
Media dispensers	1. Volume dispensed 2. Decontaminate, clean and sterilize as appropriate	Daily / each use / each adjustment
Pipetters / pipettes	1. Accuracy and precision of volume dispensed 2. Clean	Regularly (defined by taking account of the frequency and nature of use) Each use
Spiral platers	1. Establish performance against conventional method	Initially and annually



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Equipment	Requirements	Frequency
	2. Check stylus condition and the start and end points 3. Check volume dispensed 4. Service 5. Decontaminate, clean, and sterilize	Daily / each use Monthly Annually Each use
Colony counters	1. Check against manual number counted	Monthly
Anaerobic jar / incubator	1. Anaerobic indicator 2. Clean / disinfect	Each use
Centrifuge	1. Service 2. Clean and disinfect	Annually Each use



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

Polarized Light Microscopy Division

### **HEPA Hood/Microscope Maintenance**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-05-1-02**

Page #: **1 of 5**

#### **Purpose**

To provide assurance that samples are prepared and analyzed under optimum conditions, the equipment used for PLM analysis is regularly checked and maintained. This section is to describe procedures of regularly check and maintenance of HEPA Hoods, Microscopes, and utensils used by PLM analysts to ensure proper functioning of these equipments.

#### **Responsibilities**

PLM analysts perform the maintenance of polarized light microscopes and utensils as well as a regular check of the functioning of the HEPA hood.

#### **Procedure**

Equipment which has been broken or mishandled or has given suspect results is taken out of service until the problem has been identified and repaired. After repairs, equipment must be shown to be performing satisfactorily before it is placed back in service.

##### HEPA Hood

The negative air flow HEPA hood is checked at least every six months using a vaneometer, unless air monitoring tests are positive for asbestos, in which case additional inspections may result. During the checks, the average face velocity of the hood as measured over four different quadrants of the hood will be determined.

##### Microscope

The microscope receives periodic maintenance and is subjected to items below as needed to correct irregularities in its performance that are noticed during analyses.

- a) Continuous cleaning. Lenses are kept clean by wiping them (use only lens cleaning paper) periodically, or whenever image quality is affected or dirt is noticed.
- b) Dusting of other parts. The gypsum plate, analyzer, and substage lenses are dusted periodically.
- c) Alignment.
  - 1) Substage at proper height - lowest mag objective, 1st get a particle on a slide in focus, then almost completely close down the base illumination iris, raise or lower substage until iris image is clear and in focus.
  - 2) Stage centered- lowest mag objective, center of rotation at cross-hairs
  - 3) Substage centered- lowest mag objective, center illumination on cross-hairs
  - 4) Higher Mag Objectives centered- center of rotation at cross-hairs



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## Standard Operating Procedure

Polarized Light Microscopy Division

### HEPA Hood/Microscope Maintenance

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- 5) Dispersion Staining Lens central stop alignment - using centering telescope
  - 6) Polarizer oriented- below substage; line up marks
  - 7) Analyzer oriented- with no sample, completely dark when analyzer is in
  - 8) Cross Hairs are vertical/horizontal and that they are parallel to the privileged directions of the polarizer and analyzer. (The ocular containing the cross-hairs must be locked into it's standard slot at all times to insure proper alignment).
  - 9) Gypsum plate at 45 degrees to analyzer- pull out plate and check orientation of crystal by placing it on stage and rotating to extinction
- d) Other moving parts (focusing gears, etc.) of the microscopes are also maintained in good working order according to the manufacturers' instructions. The instructions can be found in the PLM laboratory or in the PLM Laboratory Manual.

#### Utensils

Knife blades and other tools are replaced when necessary to allow efficient and effective sample prep.

#### Documentation

The following records are generated and managed:

Required Record	Custodian
Daily PLM Microscope Calibration Record	Senior PLM Analyst

#### Reference Procedures

Equipment manuals

SOP # 5-05-1-01 Equipment



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

Polarized Light Microscopy Division

### **HEPA Hood/Microscope Maintenance**

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Quality Assurance Manager

***12 / 16 / 2004***

Date



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## ***Standard Operating Procedure***

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### **HEPA Hood/Microscope Maintenance**

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**Appendix: Daily Microscope Calibration Data Sheet**

Asbestos TEM Laboratories, Inc.  
Daily PLM Microscope Calibration Sheet

[illegible]





Asbestos TEM Labs, Inc.

## **Standard Operating Procedure**

Polarized Light Microscopy Division

### **Reference Standards and Reference Materials**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-06-1-01**

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#### **Purpose**

To outline the program for calibration of reference standards. To outline 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.

#### **Scope / Field of Application**

*Physical Standards* – such as reference weights and reference thermometers

*Chemical Standards* – such as standard solutions

#### **Responsibilities**

**Facility Clerk** – responsible for receiving, transport, and storage of reference standards and materials.

**Laboratory Personnel** – responsible for handling and use of reference standards and materials.

#### **Materials Required**

Certificates of Analysis  
Inventory of Reference Materials  
Standards Logbook

#### **Procedure**

##### **General**

Upon receipt, all reference standards and materials are inventoried as outlined in SOP# 4-6-1. Reference standards and accompanying certificates are stored and used in a manner consistent with preserving the calibration status. Particular consideration is given to any storage advice given in the documentation supplied with the standard.

Records are kept for the date of receipt, opening, and expiration.

##### **Physical**

1. Instructions for the use of the reference standard are cited in the appropriate SOP.
2. Calibration of working standards is performed and documented according to the appropriate SOP and reference is made to the original standard.
3. The working standard is labeled immediately after calibration with the identity, date performed, expiration date, and the person who calibrated it.



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## ***Standard Operating Procedure***

Polarized Light Microscopy Division

### **Reference Standards and Reference Materials**

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#### **Chemical**

1. Instructions for preparation and standardization of the standards are cited in the appropriate test method or SOP.
2. The preparation of standard solutions is recorded in the Standard Preparation Sheet.
3. The assigned expiration date of a prepared standard will be three months. The analyst should check the expiration date before using a standard solution.
4. Dilutions of the stock solutions are made with distilled water and high purity acids exclusively.
5. To avoid contamination of standards, use new one-use pipette tips for taking aliquots from a reagent bottle and carefully clean spatulas or spoons before and after insert them into a bottle that contains solid material. Replace cap of container tightly and clean up any spills immediately.
6. The purity and traceability of all standards utilized in the analytical process is critical to the quality of laboratory generated data. Certified standards are purchased from well established and reputable vendors, and are traceable to the National Institute of Standards and Technology (NIST).
7. Standards are purchased from more than one vendor to allow for independent source cross checks of standards during the analytical process.
8. Stock and working standards are checked frequently for signs of degradation.

#### **Documentation**

Inventory of reference materials includes the following information:

- name
- manufacturer
- lot number
- reference material, lab control number
- date received
- date opened
- expiration date
- reference material location
- manufacturer's certificate of analysis
- location

#### **Reference Procedures**

Equipment manuals

Appropriate test methods and / or specific equipment calibration and maintenance standard operating procedures

SOP 4-06-1 - Purchasing

SOP 5-06-1-02 Refractive Index Liquid Calibration



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## Reference Standards and Reference Materials

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### References

Eurachem Guidance Document No. 1. 1993. Accreditation for Chemical Laboratories.

Eurachem. 1996. Accreditation for Laboratories Performing Microbiological Testing. 1<sup>st</sup> Edition.

Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.

### Revision History

Revision	Date	Revision Notes
0	Dec. 16, 2004	Initial Publication

Approval

Yanxia Xie

Author

12 / 16 / 2004

Date

Yanxia Xie Init: \_\_\_\_\_

Quality Assurance Manager

12 / 16 / 2004

Date



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### Refractive Index Liquid Calibration

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#### Purpose

If refractive index oils are contaminated with other chemicals, or are left open for days or even hours, their refractive indices may change and this will lead to erroneous analytical results. This section is to describe the procedure for refractive index liquid calibration using Cargille precision calibrated optical glasses.

#### Responsibilities

PLM analysts perform monthly RI oil calibration

#### Materials and Equipment Required

Cargille calibrated optical glass standards

Polarized Light Microscope with dispersion staining objective

Thermometer with at least 1°C division

#### Procedure (Su, 2002)

1. Measure and record the room temperature,  $t$ , with an accuracy of  $\pm 2^\circ\text{C}$
2. Select a Cargille calibrated glass standard whose Refractive Index is closest to that of the liquid to be calibrated. For instance, use 1.55 glass standard for 1.550 liquid, 1.60 glass standard for 1.605 liquid, and 1.68 glass standard for 1.680 liquid
3. Mount the glass in the liquid and observe the glass particles' ***predominant*** dispersion staining color on a polarized light microscope. Central stop mode is preferred.  
*Caution: Sometimes glass fragments with flat edges that are vertical to or at very high angle with the glass slide surface may display false central stop dispersion staining color at these edges, e.g., orange central stop dispersion staining color for 1.55 glass fragments in 1.550 liquid. If such false central stop dispersion staining color appears, observation should be focused on the interior of glass fragments or fragments without those types of steep flat edges.*
4. Convert the observed central stop dispersion color into the corresponding matching wavelength,  $\lambda_0$ , by referring to Table 1 (Appendix 1) or a dispersion staining color chart (Appendix C in SOP # 5-04-2-PLM-01)
5. Convert  $\lambda_0$  and  $t$  into the corresponding  $n_D^{25^\circ\text{C}}$ , i.e., the calibrated  $n_D^{25^\circ\text{C}}$  or  $(n_D^{25^\circ\text{C}})_{\text{calibrated}}$ , by referring to an appropriate conversion table (Appendix 3 to 6):



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Cargille RI liquid to be Calibrated		Cargille Calibrated Glass Standard	Conversion Table No.
Nominal or Labeled $n_D^{25^\circ\text{C}}$	Series	Nominal or Labeled RI*	
1.550	E	1.55	3
1.605	E	1.60	4
1.680	B	1.68	5
1.700	B	1.70	6

\*The precise and accurate indices at different wavelengths are listed in the accompanying optical constant table

6. Compare the calibrated  $n_D^{25^\circ\text{C}}$  or  $(n_D^{25^\circ\text{C}})_{\text{calibrated}}$  with the nominal  $n_D^{25^\circ\text{C}}$  or  $(n_D^{25^\circ\text{C}})_{\text{labeled}}$  on the bottle of the RI liquid. If the *absolute difference* between these two values is less or equal than 0.004, this liquid can be used for bulk sample analysis. If the *absolute difference* is greater than 0.004, it should no longer be used.
7. Record the calibration result on the monthly RI calibration data sheet (Appendix 7)

#### Example

When the Cargille 1.55 glass of Lot C is used to calibrate a Cargille 1.550 (Series E) RI liquid at the room temperature of 21°C, the predominant central stop dispersion color observed is bluish purple, whose corresponding  $\lambda_0$  is found to be 580 nm according to Table 1. By referring to Table 4 (1.550 liquid with 1.55 glass of Lot C),  $\lambda_0 = 580$  and  $t = 21^\circ\text{C}$  yield 1.549, which is the interpolation between 1.548 (20°C) and 1.549 (22°C).

The calibration result shows that the RI of this bottle of 1.550 liquid at 589 nm and 25 °C, or  $(n_D^{25^\circ\text{C}})_{\text{calibrated}}$ , is **actually** 1.549. Because the difference is 0.001, using the  $\pm 0.004$  criterion, this RI liquid is considered to be acceptable for being used in bulk sample analysis.



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Procedure for liquid-glass combinations not listed in Tables 3 to 6 (Appendix 3 to 6)

This procedure is illustrated by using a worksheet as follows:

Data/Parameter		How to obtain it	Symbol	Value
Cargille liquid to be calibrated	The dispersion coefficient	Read from the label of the liquid bottle	$\Delta^L$	
	The temperature coefficient	Read from the label of the liquid bottle (negative value)	$dn/dt$	
	The liquid or room temperature	Read from thermometer	$t$	
Cargille glass used in calibration	The labeled $n_D^{25^\circ C}$	Read from the Column for 5893 Å of the optical data sheet	$n_D^S$	
	The dispersion coefficient	Subtract Column 6563 Å from Column 4861 Å of the optical data sheet	$\Delta^S$	
	Central dispersion staining color	Observed by using Central Stop Dispersion Staining (CSDS) mode	CSDS color	
	Corresponding matching wavelength	Read from Table 1 based on the observed CSDS color	$\lambda_0$	
	Corresponding conversion factor	Read from Table 2 based on the $\lambda_0$	$k_D$	
RI of the liquid at 589.3 nm and $t^\circ C$		$n_D^L = n_D^S - (\Delta^L - \Delta^S) \cdot k_D$	$n_D^L$	
RI of the liquid at 589.3nm and $25^\circ C$		$n_D^{25^\circ C} = n_D^L - (25 - t) \cdot dn/dt$	$(n_D^{25^\circ C})_{calibrated}$	

Compare the calibrated value  $(n_D^{25^\circ C})_{calibrated}$  with the  $n_D^{25^\circ C}$  on the label of the liquid bottle. If the absolute value of the difference is  $>0.004$ , the liquid should not be used.

### Documentation

The following records are generated and managed:

Required Record	Custodian
Monthly Refractive Index Oil Calibration Record	Senior PLM Analyst



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### Reference Procedures

SOP # 5-06-1-01 Reference Standards and Materials

### References

Refractive Index Liquid Calibration Using Calibrated Refractive Index Solids, A Standard Operation Procedure for Bulk Asbestos Analysis By Polarized Light Microscopy, Shu-Chun Su, Nov. 2002, Hercules Inc.

Dispersion Staining: Principles, Analytical Relationships and Practical Applications to the Determination of Refractive Index, *Microscope*, 1998, Shu-Chun Su

### Revision History

Revision	Date	Revision Notes
0	Feb. 16, 2005	Initial Publication

Approval

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Quality Assurance Manager

02 / 16 / 2005

Date



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**Appendix 1: Table 1. Converting dispersion staining color to corresponding  $\lambda_0$  (McCrone, 1987)**

**Appendix 2: Table 2. Conversion from  $\lambda_0$  to  $k_D$  (Su, 1998)**

**Appendix 3: Table 3. Conversion Table for Calibrating RI Liquid 1.550 (Series E) Using Cargille Calibrated Refractive Index Solid 1.55**

**Appendix 4: Table 4. Conversion Table for Calibrating RI Liquid 1.605 (Series E) Using Cargille Calibrated Refractive Index Solid 1.60**

**Appendix 5: Table 5. Conversion Table for Calibrating RI Liquid 1.680 (Series B) Using Cargille Calibrated Refractive Index Solid 1.68**

**Appendix 6: Table 6. Conversion Table for Calibrating RI Liquid 1.700 (Series B) Using Cargille Calibrated Refractive Index Solid 1.70**

**Appendix 7: Monthly Refractive Index Calibration Data Sheet**





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### Appendix 1

Table 1. Converting dispersion staining color to corresponding  $\lambda_0$  (McCrone, 1987)

Matching Wavelength $\lambda_0$ , nm	Particle Edge Colors <sup>1</sup>		Becke Line Colors <sup>2</sup>	
	Annular Stop <sup>3</sup>	Central Stop <sup>4</sup>	Particle	Liquid
<340	Black violet	White	White	X
<400	Dark violet	Pale yellow	Pale yellow	X
430	Violet	Yellow	Pale yellow	X
455	Blue	Golden yellow	Yellow	Violet
485	Blue-green	Orange	Orange	Violet
520	Green	Red purple	Orange-red	Violet-blue
560	Yellow-green	Purple	Red-orange	Blue-violet
595	Yellow	Deep blue	Red	Blue
625	Orange	Blue-green	Faint red	Blue
660	Red-brown	Light blue-green	X	Blue-green
700	Dark red-brown	Pale blue-green	X	Pale blue-green
1500	Black-brown	Very pale blue-green	X	Very pale blue-green

1. In focus
2. On focusing up
3. Observed on a bright field
4. Observed on a dark field



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### Appendix 2

Table 2. Conversion from  $\lambda_0$  to  $k_D$  (Su, 1998)

$\lambda_0$ (nm)	$k_D$
500	0.59
510	0.50
520	0.43
530	0.35
540	0.29
550	0.22
560	0.16
570	0.10
580	0.05
590	-0.00
600	-0.05
610	-0.10
620	-0.14
630	-0.19
640	-0.23
650	-0.27
660	-0.30
670	-0.34
680	-0.37
690	-0.40
700	-0.44
710	-0.47
720	-0.50
730	-0.53
740	-0.55
750	-0.67
800	-0.69



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### Appendix 3

Table 3. Conversion Table for Calibrating RI liquid **1.550** (Series E) Using Cargille  
Calibrated Refractive Index Solid **1.55**

$\lambda_0$ (nm)	20°C	22°C	24°C	26°C	28°C	30°C
520	1.542	1.543	1.544	1.545	1.546	1.547
540	1.544	1.545	1.546	1.547	1.548	1.549
560	1.546	1.547	1.548	1.549	1.550	1.551
580	1.548	1.549	1.550	1.551	1.551	1.552
600	1.549	1.550	1.551	1.552	1.553	1.554
620	1.551	1.552	1.553	1.554	1.554	1.555
640	1.552	1.553	1.554	1.555	1.556	1.557
660	1.553	1.554	1.555	1.556	1.557	1.558
680	1.554	1.555	1.556	1.557	1.558	1.559
700	1.555	1.556	1.557	1.558	1.559	1.560

Solid:  $n_F = 1.55862$ ;  $n_D = 1.55077$ ;  $n_C = 1.54753$ ;  $\Delta^S = (n_F - n_C) = 0.01109$

Liquid:  $\Delta^L = (n_F - n_C) = 0.0267$ ;  $dn/dt = -0.00049$



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#### Appendix 4

Table 4. Conversion Table for Calibrating RI liquid **1.605** (Series E) Using Cargille  
Calibrated Refractive Index Solid **1.60**

$\lambda_0$ (nm)	20°C	22°C	24°C	26°C	28°C	30°C
560	1.597	1.598	1.599	1.600	1.601	1.602
580	1.598	1.599	1.600	1.601	1.602	1.603
600	1.599	1.600	1.601	1.602	1.603	1.604
620	1.600	1.601	1.602	1.603	1.604	1.604
640	1.601	1.602	1.603	1.603	1.604	1.605
660	1.601	1.602	1.603	1.604	1.605	1.606
680	1.602	1.603	1.604	1.605	1.606	1.606
700	1.603	1.603	1.604	1.605	1.606	1.607
720	1.603	1.604	1.605	1.606	1.607	1.608
740	1.604	1.604	1.605	1.606	1.607	1.608
760	1.604	1.605	1.606	1.607	1.608	1.608
780	1.604	1.605	1.606	1.607	1.608	1.609
800	1.605	1.606	1.607	1.607	1.608	1.609

Solid:  $n_F = 1.61227$ ;  $n_D = 1.60106$ ;  $n_C = 1.59657$ ;  $\Delta^S = (n_F - n_C) = 0.01570$

Liquid:  $\Delta^L = (n_F - n_C) = 0.0243$ ;  $dn/dt = -0.00044$



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### Appendix 5

Table 5. Conversion Table for Calibrating RI liquid **1.680** (Series **B**) Using Cargille Calibrated Refractive Index Solid **1.68**

$\lambda_0$ (nm)	20°C	22°C	24°C	26°C	28°C	30°C
560	1.672	1.673	1.674	1.675	1.676	1.677
580	1.674	1.675	1.676	1.677	1.678	1.679
600	1.677	1.678	1.679	1.680	1.680	1.681
620	1.679	1.680	1.681	1.682	1.683	1.683
640	1.681	1.682	1.682	1.683	1.684	1.685
660	1.682	1.683	1.684	1.685	1.686	1.687
680	1.684	1.685	1.686	1.687	1.688	1.689

Solid:  $n_F = 1.68654$ ;  $n_D = 1.67785$ ;  $n_C = 1.67424$ ;  $\Delta^S = (n_F - n_C) = 0.01230$

Liquid:  $\Delta^L = (n_F - n_C) = 0.0348$ ;  $dn/dt = -0.00048$



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## Appendix 6

Table 6. Conversion Table for Calibrating RI liquid **1.700** (Series **B**) Using Cargille  
Calibrated Refractive Index Solid **1.70**

$\lambda_0$ (nm)	20°C	22°C	24°C	26°C	28°C	30°C
520	1.691	1.692	1.693	1.694	1.695	1.696
540	1.694	1.695	1.696	1.697	1.698	1.699
560	1.696	1.697	1.698	1.699	1.700	1.701
580	1.699	1.699	1.700	1.701	1.702	1.703
600	1.701	1.701	1.702	1.703	1.704	1.705
620	1.702	1.703	1.704	1.705	1.706	1.707
640	1.704	1.705	1.706	1.707	1.708	1.709

Solid:  $n_F = 1.71406$ ;  $n_D = 1.70189$ ;  $n_C = 1.69697$ ;  $\Delta^S = (n_F - n_C) = 0.01709$

Liquid:  $\Delta^L = (n_F - n_C) = 0.0370$ ;  $dn/dt = -0.00048$



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## APPENDIX 7

### Monthly Refractive Index Oil Calibration Sheet

Asbestos TEM Laboratories, Inc.  
Monthly Refractive Index Oil Calibration Sheet

[illegible]





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## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Handling of Test Items**

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#### **Purpose**

To outline the procedures for the receipt, handling, protection, storage, retention and/or disposal of test and/or calibration items.

To outline the procedures and appropriate facilities for avoiding deterioration, loss, or damage to the test item during storage, handling, preparation, and testing.

#### **Scope / Field of Application**

This procedure applies to all test items.

Efforts are made to minimize errors that can be introduced as a result of handling the sample.

#### **Definitions and Acronyms**

**Holding time** - time elapsed from the date of sampling until the start of testing.

#### **Responsibility**

Laboratory personnel

#### **Materials Required**

Refrigerator / freezer

Shelves / racks

#### **Procedure**

##### **Advising Clients of the proper procedures for submitting samples**

Clients may be unaware of several items that will ultimately affect the accuracy of the results. Inexperienced clients may be unaware of these considerations. The most important step is to inform all new clients of the following items:

- 1) The need for complete and accurate documentation of samples with complete descriptions including client identification numbers, sampling locations, and sample descriptions. A lack of such information can lead to mistaken analyses or confusion regarding reports.
- 2) Bulk asbestos samples should be properly bagged to prevent leakage of sample material causing a health hazard and potential cross-contamination.
- 3) Air samples should never be shipped with bulk samples due to the great potential for crosscontamination of asbestos into the air samples.



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#### **Receiving Samples**

Samples received are immediately checked for danger of asbestos contamination on the exterior of the packaging. The package is checked for damage or disruption of any chain-of-custody seals. Acceptable packages are opened. Unacceptable/damaged packages are either not accepted for delivery, or they are immediately carried to the PLM laboratory and placed into the PLM HEPA hood. At some time prior to log-in, the samples are inspected (under the HEPA hood if necessary). If samples are missing, the client is contacted. If any of the following problems occur, the samples are rejected and the client is notified immediately.

- 1) Asbestos or other hazardous materials on sample container exteriors.

If the outsides of any sample containers are coated with dust or potentially hazardous material (e.g. Tar), the samples are immediately replaced into the package and taken to the PLM lab and checked in the HEPA hood.

- 2) Inadequate Containers / Container Damage.

Reject samples if packaging is inadequate, broken or torn such that lab safety may be jeopardized or cross-contamination could occur.

- 3) Tampering.

Chain of custody seals, if present, must be intact. The chain of custody form should be briefly reviewed to ensure the sample was not tampered with. A notation is made on the log sheet if the sample was previously analyzed or opened by the client for any reason.

- 4) Faulty Documentation.

The documentation must include the client's sample identification numbers or sample location descriptions. Clients are encouraged to use a unique number for each sample. If there are any omissions, if the samples do not match the documentation or if there is any illegible information, the client will be contacted to ensure that the proper information is obtained.

#### **Logging of samples**

If the sample has arrived undamaged, has not been tampered with, and has adequate documentation, it is introduced into the laboratory sample track. The samples are placed in a large zip-lock baggy and remain in the PLM laboratory. The relevant information on the custody sheet is entered into the laboratory computer system through the log-in procedure.

- 1) Procedure. The log-in process consists of running a computer program which prompts the operator for the required information as below:

- a) Date of Sample Receipt.

- b) Condition of Test Item

- c) Sample Acceptance - Only samples that are accepted for analysis are logged in to the computer samples. Rejected samples are held, and clients are contacted concerning the reasons for rejection. Depending on the outcome of the client contact, samples are either accepted and logged in to the system, or they are disposed of or held pursuant to the clients wishes.

- d) Unique Sample I.D.- Each sample is assigned a unique laboratory identification number to ensure that, in the event that two different samples in the laboratory have the same ID number given to them by the clients, there will not be confusion over sample identification. This laboratory ID number is in the form "Client number" - "lot number" - "sample number" (e.g.



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100-1-1). Samples that arrive together, have the same requested turnaround, and are to be billed to the same job are grouped together into a lot.

e) Client Sample ID Number.

f) The sample description given on the client's documentation is transferred verbatim to the log-in sheet. In cases where there is not enough room for all of the client's information, abbreviations may be used for some words.

g) Initials of Person Logging in the Samples.

Once all of the information concerning a sample lot has been entered, a hard copy log-in sheet is printed.

2) Review of Log-in Sheet. Each Log-in sheet is checked for accuracy against the sampling documentation.

### **Sample Tracking**

When a sample lot has been logged in, an entry is made in the sample log-in record in the laboratory computer system. This track includes all the sample lots in the order in which they were logged in, the requested turnaround times, and spaces where the completion dates for verbal and written reports can be entered. This system enables the laboratory sample coordinator to determine at a glance what progress has been made in the analysis and reporting on a given sample and which samples are high priority each day. Also, all reports which have been faxed out are noted as to the client phone #, client-lot number of the faxed report, date faxed, and person faxing the information.

### **Handling and Archival of samples after analysis**

Microscope slide preparations are disposed of in an appropriate manner. Slides are discarded soon after analysis in an appropriately sealed and labeled container. Non-permanent slide preparations temporarily set aside for QA analysis should be covered prior to re-analysis, and eventually discarded.

After analysis the remainder of all analyzed bulk samples are resealed, the exteriors of the containers are wet-wiped if necessary, and they are then taken to either a secure on-site or off-site storage area. Samples are stored by Asbestos TEM Laboratories for at least one year after receipt, unless clients desire otherwise. Thereafter, the samples are disposed at an appropriate manner in accordance with all federal, state and local regulations. Documentation of sample disposal, and/or return of samples to the clients will be maintained. All hazardous waste shall be placed in the hazardous material receptacle in the storage facility. Said waste is removed periodically by a certified hazardous waste hauler.

### **References**

Garfield, F.M. 1991. Quality Assurance Principles for Analytical Laboratories. AOAC. Arlington, VA.



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#### **Revision History**

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0	Dec. 16, 2004	Initial Publication

Approval

***Yanxia Xie***

Author

***12 / 16 / 2004***

Date

***Yanxia Xie*** Init: \_\_\_\_\_

Quality Assurance Manager

***12 / 16 / 2004***

Date



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**Polarized Light Microscopy Division**

### **Handling of Test Items**

Issue Date: **2004/12/16**

Revision: **0**

SOP#: **5-07-1**

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**Appendix A: Example of Sample Login Sheet**

**Appendix B: Example of Sample Tracking Screen**



Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

### **Handling of Test Items**

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- APPENDIX A -

EXAMPLE OF SAMPLE LOGIN SHEET



Asbestos TEM Labs, Inc.

# ***Standard Operating Procedure***

**Polarized Light Microscopy Division**

## **Handling of Test Items**

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Asbestos TEM Labs, Inc.

## ***Standard Operating Procedure***

Polarized Light Microscopy Division

### **Handling of Test Items**

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Revision: **0**

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- APPENDIX B -

EXAMPLE OF SAMPLE TRACKING SCREEN



## PLM BULK SAMPLE LOGIN REPORT

INVOICE #: \_\_\_\_\_

ANALYSIS REQUESTED: **PLM-STANDARD** URGENCY: **3-5 DAYS**DATE / **Oct-08-04**  
TIME IN: **3:00 pm**CLIENT NO: **1068** LOT NO: **00062** Total Samples: **10**DATE / **Oct-13-04**  
TIME DUE: \_\_\_\_\_JOB SITE: **R-66**JOB NO: **R-66**Logged by: **yx**CONTACT: **Mr. James Rich**SAMPLE CONDITIONS: **ACCEPTABLE**DELIVERED BY: **Client**SPECIAL INSTRUCTIONS **Stop at first positive (>1%)**☒ **FAX** **(916) 374-1025**☒ **E-MAIL** **richmarcenv@msn.com**Invoice To:  
**richmarcenv@msn.com**

REVIEWED \_\_\_\_\_ E-MAILED \_\_\_\_\_

CL#-LOT-SAMP

CLIENT#

DESCRIPTION

1068-00062-001	RFSHNG-116-1	tar shingles w/rock topping Roof Shingle
1068-00062-002	RFSHNG-116-2	tar shingles w/rock topping Roof Shingle
1068-00062-003	RFSHNG-116-3	tar shingles w/rock topping Roof Shingle
1068-00062-004	RFP-117-1	Paper Roof
1068-00062-005	RFP-117-2	Paper Roof
1068-00062-006	RFP-117-3	Paper Roof
1068-00062-007	RFP-117-4	Paper Roof
1068-00062-008	RFPEN-118-1	Black Penetration Mastic
1068-00062-009	RFCOMP-119-1	Black Flat built-up roof layers Roofing composite
1068-00062-010	RFCOMP-119-2	Black Flat built-up roof layers Roofing composite
1068-00062-011	RFCOMP-119-3	Black Flat built-up roof layers Roofing composite

\*\*\* LABMANAGER \*\*\* - [Form - TRACKER]

File Edit View Data Utilities Options Program Window Help

LOGINFO Login No

REPORT TRACKING ENTRY FORM

Login #

CI-Lot #

Date In

Time In

Logger

047892

606-02551

Jan-06-05

9:40 am

ry

68864

Job Site:

Job No.

Contact:

Boston Prop ECZ Fl. 2

B7006.AS

Mr. Brian Brooks

GET RECORD

MULTI-TRACK

Analysis Type

Samples

Turnaround

Date Due

PCM-NIOSH 7400

6

<4 HR-ASAP

Jan-06-05

MAIN MENU

Notes:

Analyzed	Faxed	Verbal	Reviewed	E-Mailed	FTP Site	Mailed	Archived
Jan-06-05	Jan-06-05	Jan-06-05	Jan-06-05			Jan-06-05	
11:02 am	11:35 am	11:35 am	1:55 pm				
SK	SK	SK	PB			RY	
Date/Time	Date/Time	Date/Time	Date/Time	Date/Time	Date/Time	Date	Date

E-Mail Addresses

#1

#2

#3

INV

E-Mail Subject

Ready

NUM

INS



### **Scope**

Metals in solution may be readily determined by Direct-Aspiration Atomic Absorption Spectroscopy (FAAS) or by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS).

When FAAS do not provide adequate sensitivity, graphite furnace is used.

This method describes the use of the FAAS (Make Perkin-Elmer Model 2380) for the determination of lead, chromium, cadmium and zinc in different matrix after acid digestion.

### **Description of Test Items**

Acid digestions of paint chips, soils, air filters, wipes, and wastes.

### **Holding Times**

The digestates of the samples will be held until the report is complete.

The digestates known to contain < 60% the sewer discharge limit of toxic metals are neutralized and discarded down the sink.

The digestates known to contain > 60% the sewer discharge limit of toxic metals are discarded in the waste barrel.

Waste barrel is removed periodically by a certified hazardous waste hauler.

### **Quantities to be Tested**

N/A

### **Materials and Equipment Required**

Atomic Absorption Spectrometer. Perkin-Elmer Model 2380

Hollow cathode lamps: lead, cadmium, chromium and zinc.

Air Compressor

Micropipettes of 1 ml and 20  $\mu$ L

Dilution cups

### **Reagents**

Metal standard solution for preparing the calibration standards. Standard Grade

Metal standard solutions for preparing the ICV/CCV solution. Standard Grade.

Concentrated Nitric Acid ( $\text{HNO}_3$ ). Reagent Grade

Distilled water

Air compressed, filtered

Acetylene

### **Physical Environmental Conditions Required (Incubation Times and Temperatures, pH Requirements)**

N/A



## Description of Procedures

### Preparation of Standards:

- I. Prepare the calibration standards. Add aliquots of calibration standard to 250 ml volumetric flasks. The usual standards are 0.2 ppm (50  $\mu$ l), 1 ppm (250  $\mu$ l), and 5 ppm (1250  $\mu$ l). Dilute to volume with 10%  $\text{HNO}_3$ . Prepare fresh standards at least quarterly.
- II. Prepare ICV and CCV solutions (see Quality Control Plan Chapter for definitions). Add an aliquot of standard solution to a 100 ml volumetric flask. Dilute to volume with 10%  $\text{HNO}_3$ . Prepare fresh standards at least quarterly.
- III. Prepare an ICB/CCB solution (10%  $\text{HNO}_3$  solution with distilled water).

### Measurement:

- I. Set the Pb wavelength to 283.3 nm, the slit to 0.7/Normal. Maximize the energy.
- II. Turn on the power switch on the wall. The hood should start running.
- III. Turn on the power switch on the AA (right side).
- IV. Set the **SIGNAL Knob** to **LAMP** and turn the **LAMP Knob** until reading 10 on the screen.
- V. Set the **SIGNAL Knob** to **SET UP** and turn the **GAIN Knob** until reading 90 on the screen.
- VI. Set the **SIGNAL Knob** to **ABS**, introduce 3 with the keyboard and press **t**.
- VII. Set background correction to **AA**.
- VIII. Set **MODE** to **cont**.

Allow the lamp to warm up for a minimum of 10 minutes.

- A. Turn on the exhaust fan.
- B. Turn on the compressor.
- C. Turn on the Acetylene bottle.
- D. Turn the Oxidant Knob to **Air**.
- E. Flip the Fuel Valve to **ON**.
- F. Light the flame. Adjust the flame for the metal to be analyzed.
- G. Absorb the ICB/CCB solution. Press **A/Z**.
- H. Absorb the first standard. Wait until the result is on the screen, and write down the reading.
- I. Absorb the second standard. Wait until the result is on the screen, and write down the reading.
- J. Absorb the third standard. Wait until the result is on the screen, and write down the reading.
- K. Absorb the ICB/CCB solution. Check the results are inside the acceptance limits.
- L. Absorb the ICV solution. Check the results are inside the acceptance limits. Write down the absorbance value.
- M. Absorb the Quality Control Samples prepared with the digestion. Check the results are inside the acceptance limit. Write down the absorbance values.
- N. If the QC samples are inside the acceptance limit. Start absorbing and reading the samples.
- O. If any the QC samples are not inside the acceptance limits. Recalibrate again.
- P. During the run, absorb the CCV solution and the ICB/CCB after the first 5 samples and then every 10 samples and at the end of the run. Write down the absorbance value after each reading. If the CCV reading is out of acceptance limits, absorb the third calibration standard. Reread the CCV solution and the ICB/CCB and all the samples read after the last verification.



If the ICB/CCB is out of acceptance limits press *A/Z*. Reread the ICV and CCV solutions and the ICB/CCB and all the samples read after the last verification.

If the sample reads *Concentration Higher than Standards*, dilute the sample using the dilution cups and the pipettes.

For solving any problem with the instrument check the Instrument Operation Manual.

#### Shut Down:

- A. Turn off the Acetylene bottle. Allow flame to go out before proceeding.
- B. Flip the Fuel Valve to OFF.
- C. Turn off the exhaust fan, the air compressor, and the lamp.
- D. Wait for the air flow gauge to drop to zero.
- E. Turn the Oxidant Knob to Off.

#### **Sample Identification**

N/A

#### **Method of Recording Observations and Results**

The results are recorded in the Metal Data Sheets.

#### **Safety Measures**

Care should be taken when handling the digestates of the samples.

Wear lab coat while working in the lab.

Wearing glove is recommended while working with the AA Spectrometer.

#### **Documentation**

PE2380 Maintenance Log.

Metal Data Sheets.

#### **Sensitivity of Method**

- The *sensitivity* of the instrument is the concentration of the element (mg/L) required to produce a signal of 1% absorption (**0.0044 abs. unit**).  
The sensitivity of the instrument for lead is **0.19 mg/L** (with impact bead nebulizer, at 283.3 nm and 0.7 slit).
- The *sensitivity check* is the standard concentration that will give a reading approximately of **0.2 abs units** at the wavelength and slit width listed using optimum conditions.  
The sensitivity check of the instrument for lead is **9 mg/L** (with impact bead nebulizer, at 283.3 nm and 0.7 slit).
- The Reported Detection Limit is 0.20 mg/L.  
The reported limit must be at least twice the MDL and equal to or less than 20% of lowest relevant action level or regulatory limit of interest, except for lead wipe samples (50%). The MDL must be checked yearly for the different digestion methods.



For sensitivity of cadmium, chromium and zinc, check the instrument operating instructions.

### **Quality Control Plan**

The instrument calibration is verified initially by reading an Initial Calibration Verification (ICV) solution and an Initial Calibration Blank (ICB).

During the run the calibration is verified by reading a Continuing Calibration Verification (CCV) solution and a Continuing Calibration Blank (CCB). These two solutions are read after the first 5 samples and then every 10 samples.

For ICV, the concentration must be near the minimum level of regulatory concern; i.e., 1 ppm for wipes and soil samples (which are diluted if they are close to the regulatory level of 8 ppm), and 3 ppm for paint samples and air filters. It has to be prepared from a different source than the calibration standards.

For CCV the same solution can be used for all analyses; it is a 10% HNO<sub>3</sub> solution of a concentration that falls in the middle of the calibration curve (normally 3 ppm).

For ICB and CCB the same solution can be used (ICB/CCB solution) and it is a 10% HNO<sub>3</sub> solution.

### **Calibration Verification**

- A. To verify the calibration made by the instrument, manually calculate a calibration curve with the standard absorbance values taken during the calibration.

The type of the curve is  $C = K_1 * A + K_2$ . Where C is concentration and A is absorbance.

The correlation factor (r) of the calculated curve has to be higher than 0.995. If is lower than 0.995, the calibration is not acceptable and the reading has to be repeated.

- B. Calculate the concentration values of the ICV and CCV solutions and QC samples with the absorbance values and the calculated curve.

Compare the calculated concentration with the criteria given in SOP 5-4-6-02.

If QC samples are out of acceptance limits, the instrument calibration is not acceptable and the reading has to be repeated.

### **Reference Procedures**

EPA Method 7000A, Atomic Absorption Methods. Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. 3rd Ed., Revision 2, December 1996. SW-846.

EPA Method 7420, Lead (Atomic absorption, direct aspiration). Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. 3rd Ed., Revision 2, December 1996. SW-846.

EPA Method 7130, Cadmium (Atomic absorption, direct aspiration). Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. 3rd Ed., Revision 2, December 1996. SW-846.

EPA Method 7190, Chromium (Atomic absorption, direct aspiration). Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. 3rd Ed., Revision 2, December 1996. SW-846.

EPA Method 7950 (Atomic absorption, direct aspiration). Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. 3rd Ed., Revision 2, December 1996. SW-846.



Asbestos TEM Laboratories, Inc.

## *Standard Operating Procedure* **METALS BY ATOMIC ABSORPTION**

Issue Date: 25 Nov 2008

Revision: 6

SOP 5-4-3-AA-080

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NIOSH Method 7082, Lead by Flame AAS. NIOSH Manual of Analytical Methods (NMAM) 4<sup>th</sup> edition, May 1996.

### **References**

Perkin-Elmer. Analytical Methods for AAS.

Perkin-Elmer, Model 2380 Operating Instructions.

### **Revision History, Authorship and Approval**

Revision	Date	Revision Notes
0	24 Jun 2004	Initial Publication
1	26 Aug 2004	Modify Description of Procedures and Include Calibration Verification Section
2	14 Sept 2007	Changed Approval person to Mark Bailey
3	05 Nov 2007	Changed personnel
4	31 Jul 2008	Changed personnel, added shut down section, revised ICV prep. To new standards
5	30 Sep 2008	Added reference to SOP 5-4-6-2; deleted RPD table
6	25 Nov 2008	Added calibration solution instructions; corrected ICV/CCV

**Approved by:**

\_\_\_\_\_  
*Jane Zhang, Technical Manager*

\_\_\_\_\_  
*Date*

\_\_\_\_\_  
*Lawrence King, Quality Manager*

\_\_\_\_\_  
*Date*

# STANDARD OPERATING PROCEDURE

## FUNGAL MICROSCOPIC EXAMINATION

### BIOAEROSOL NON-CULTURABLE

#### (Quantitative Analysis)

1. **Goal of Analysis:** To identify fungal spores and determine airborne spore levels from a non-culturable air impaction sample (spore-trap); measured in spores per volume of air (spores/m<sup>3</sup>).
2. **Procedure:**
  - 2.1. Examine the mounted sample using 10X objective to locate the impaction trace.
    - 2.1.1. Locate and start at the right hand end of the trace (as observed through the microscope).
  - 2.2. Use 20X objective to identify and enumerate large spore types and scan for variation across the sample.
    - 2.2.1. The field diameter at 20X is approximately the width of the impaction trace.
    - 2.2.2. By slowly moving along the trace from right to left, it is possible to traverse the entire trace and provide accurate counts of fungal spores for the large-spored types occurring on the trace.
    - 2.2.3. For large-spored types 100% of the trace is examined and enumerated.
    - 2.2.4. A wide variety of fungal spores can be detected at 20X, but it may be necessary to rotate to 40X to examine specific spores to accurately identify or confirm identity. Then return to 20X and continue the 100% trace examination.
      - 2.2.4.1. Common fungi recorded at 20X include *Alternaria*, *Bipolaris/Drechslera*, *Chaetomium*, *Curvularia*, *Epicoccum*, Rusts, Smuts/Myxomycetes, *Stachybotrys*, and *Ulocladium*.
      - 2.2.4.2. Many other fungi are encountered less frequently and should be identified to genus if possible (see Fungal Identification training slides and reference materials).
      - 2.2.4.3. If identity of a given spore is uncertain (no distinguishing features, intermediate morphology, damaged spores, etc.), it should be recorded as 'Unidentified conidia'.
    - 2.2.5. Assess the overall level of biological and non-biological particulates collected on the sample using a 1-5 scale (see Particulate Density training slides).
      - 2.2.5.1. Optimal particulate density is ranked as a 3.
      - 2.2.5.2. Slightly light or heavy traces are ranked as 2 or 4 respectively.



- 2.2.5.3. Extremely light traces are ranked 1 and extremely heavy traces are ranked 5.
- 2.2.6. Note presence of other significant biological or non-biological particles while scanning at 20X.
  - 2.2.6.1. Record presence of hyphal fragments, pollen, insect/arthropod parts and fiberglass particles.
- 2.3. Use 40X for spore counts of smaller spore types and detailed scans of the impaction trace.
  - 2.3.1. Start at the left-hand end of the trace (as observed in the microscope), where you completed the 20X scan.
  - 2.3.2. For routine samples (typically 75 L samples with overall particulate levels of 2, 3 or 4), examining 20% of the particulate trace at 40X is appropriate.
  - 2.3.3. To examine 20%, make vertical passes through the trace at intervals along the entire length of the trace.
    - 2.3.3.1. From the starting point, measure off five fields of view. Move over one field at a time, using objects at the right hand edge of the field to move precisely 5 field-diameters.
    - 2.3.3.2. Traverse the fifth field from top to bottom.
  - 2.3.4. Carefully scan down and record all small fungal spores detected.
    - 2.3.4.1. Primary spore types recorded at 40X include Ascospores, Basidiospores, *Cladosporium* and *Penicillium/Aspergillus*.
    - 2.3.4.2. Other less common spore types may also be detected at this magnification (see training and reference materials).
    - 2.3.4.3. Do not re-record large fungal spore types that have previously been recorded during the 20X scan.
    - 2.3.4.4. Occasionally, large spore types may be observed which were not detected at the 20X scan; add these to others recorded during original pass.
  - 2.3.5. Identify and enumerate all spores in the first traverse, then move 5 fields right and repeat.
  - 2.3.6. The number of vertical traverse passes will vary from microscope to microscope depending on the field diameter and will vary slightly from sample to sample depending on particulate loading and drift of particles off the primary trace.
    - 2.3.6.1. See microscope calibration document to determine measurements and field diameters of the microscope you are using.
    - 2.3.6.2. By recording every fifth pass, it ensures that 20% of the trace is examined.
    - 2.3.6.3. Generally at 40X, about 5-6 passes will be made.
    - 2.3.6.4. If there is an odd number of fields, read the equivalent proportion (i.e., 20%) of the end portion.
- 2.4. The aim of the analysis is to examine an adequate portion of the impaction trace as needed to ensure the final assessment is representative of the whole sample.
  - 2.4.1. In situations where the trace is very light (particulate level 1-2), it is useful and practical to examine a greater portion of the trace to provide more accurate quantification of spore levels.

- 2.4.1.1. For samples with very short sample times or in very clean environments (e.g., clearance samples after air scrubbing), 50% or 100% of the trace may be examined at 40X.
- 2.4.2. In situations where the trace is very heavy (wall cavities, containments with active remediation, long sampling times, etc.) it may be necessary to observe the particulate trace using the 100X oil-immersion objective.
- 2.4.2.1. Where sample times are long (150 L or more) or the trace is heavy (particulate level 4-5), read 10% of the trace with the 100X objective to allow for better detection of small spore types and obtain a more accurate estimate of spore levels on the sample.

### 3. Analytical Report:

- 3.1. The following are required for the analytical report:
  - 3.1.1. Calculate and record the total number of spores of each fungal type detected or estimated for the sample.
  - 3.1.2. Record presence of hyphal fragments, pollen, insect parts, and fiberglass particles (quantitative analysis may also be required for other biological particulates; see Non-fungal particle analysis protocol).
  - 3.1.3. Determine the density of total biological particulates and total non-biological particulates.
  - 3.1.4. Provide comments regarding other noteworthy observations (dominant fungi, other potentially significant/indicator fungi, unusual non-biological debris, etc.).
- 3.2. Calculations of spores detected on the sample are made using the database.
  - 3.2.1. Input the total volume of air sampled (e.g., 75 L).
  - 3.2.2. Input the fungal spores identified, the raw count, and the percent of the particulate trace examined for each spore type.
  - 3.2.3. The Sample Count is obtained by multiplying the raw count by the appropriate factor based on the percent read to reflect the estimate of the total number present on the entire sample.
  - 3.2.4. The Sample Count is multiplied by the sensitivity (1000 L divided by total volume sampled) to get the Calculated Count.
    - 3.2.4.1. Sensitivity (Limit of Detection): The minimum possible number of spores that could be detected per volume of air (spores/m<sup>3</sup>) based on the volume of air sampled.
  - 3.2.5. Calculated Counts are rounded to two significant figures.
    - 3.2.5.1. Calculated Count Totals are not rounded.
  - 3.2.6. Record the Sample Count and Calculated Count for each spore type and the TOTALS for the Sample Count and Calculated Count columns.

See Sample Calculations below:

e.g., *Cladosporium*: 10 spores detected in the 20% examined  
Sample Count = 10 spores x 5 = 50 spores/sample

e.g., Sensitivity: 1 spore detected in a 75 L sample  
 $1000 \text{ L} \div 75 \text{ L} = 13.333333 \text{ spores/m}^3$

e.g., # spores detected in sample x Sensitivity  
 $50 \text{ spores/sample} \times 13.333333 \text{ spores/m}^3 = 670 \text{ spores/m}^3$

3.3. Record Summary statements as appropriate. See additional training documents to aid in assessment of significant findings, and include statements for the following:

3.3.1. Dominant fungal spores detected on sample.

3.3.2. Potentially significant/indicator fungi detected (or "Other potentially significant/indicator fungi ..." if dominant fungi were also significant).

3.3.3. Overall low levels of fungal spores detected ( $< 100 \text{ spores/m}^3$ ).

3.3.4. Record sensitivity.

3.3.5. Record other significant observations (high levels of other particulates, pollen, etc.).

3.3.6. It is typically required to list a statement of sample limitations for very heavy traces.

3.3.6.1. If level of non-biological particulates is extremely high and may have interfered with sample analysis, record a statement similar to the following: "extremely high levels of non-biological particulates may have reduced or affected the detection of small spores; calculated counts are approximate and the total number of spores present may have been underestimated due to this limitation."

3.3.6.2. If level of spores is extremely high and estimates of total numbers are based on a small percentage of the trace examined, record a statement similar to the following: "High levels of spores present on sample may have reduced or affected accuracy of calculated counts."

#### 4. Example:

4.1. See attached sample Report and Summary Table for format and terminology.

# STANDARD OPERATING PROCEDURE

## FUNGAL MICROSCOPIC EXAMINATION

### TAPE SAMPLE

#### (Qualitative Analysis)

**Goal of Analysis:** To assess whether active microbial growth is occurring at the site sampled and to identify the fungi present on a given surface. A qualitative assessment of the approximate level of fungal growth is provided.

#### I. Analytical Procedure

##### Macroscopic Examination:

- Examine the tape sample with the naked eye or using the stereomicroscope to scan for evidence of particulates or other debris adhering to sample.
  - Record macroscopic observations, including visible mold growth, deposits of colored material, and the presence of large particulates.
  - Estimate density of contaminant material detected on sample (light, medium or heavy).

##### Microscopic Examination:

- Examine the tape using 10X objective to scan for variation across the sample.
- Use 40X for detailed scans and fungal identification.
- Choose a representative area to start towards one end of the sample.
- Move from one end of sample to the other. Scan all areas of the sample (10X or 20X) and use 40X to examine as many different types of fields (spore types and density, particulates, etc.) as needed to ensure the final assessment is representative of the whole sample.
- Avoid the edges of the sample (to avoid possible areas of cross contamination during sample collection process).
- Identify all fungi detected on sample.
  - Generally genus ID is provided where possible. Some fungi may be identified only to broad category (e.g., yeasts, basidiomycetes, smuts/myxomycetes, rusts, etc.) or genera may be grouped together (e.g., *Penicillium/Aspergillus*, *Bipolaris/Drechslera*, etc.).
  - Rarely it may be possible and useful to provide a species identification, although species confirmation typically requires a culture.
- Differentiate fungi with visible growth elements (hyphae, conidiophores, etc.) from miscellaneous spores.

## II. Analytical Report

- For the analytical report: record each fungal type detected, evidence of growth and an estimate the qualitative rank of growth present for that fungus on sample.
- Level 1-5: Confirmed growth of identified fungi. Choose appropriate qualitative level (as outlined below) and list growth structures identified (e.g., spores, hyphae, conidiophores, ascocarps, pycnidia, sporangia, sclerotia, chlamydospores, etc.) for each.
  - **Nearly confluent growth (Level 5)**; 81-100% of sample covered by organisms listed. Sample dominated by growth, generally heavy growth, covering all or nearly all of the sampled area.
  - **Extensive growth (Level 4)**; 61-80% of sample covered by organisms listed. Sample with substantial growth of this organism, but not covering the entire sample area.
  - **Moderate growth (Level 3)**; 41-60% of sample covered by organisms listed. Growth covering approximately half of the sample.
  - **Limited growth (Level 2)**; 21-40% of sample covered by organisms listed. Often present as one area of substantial growth or few small patches of growth throughout the sample.
  - **Sparse/minimal growth (Level 1)**; 1-20% of sample covered by organisms listed. Generally only a very small area of growth detected or very few spores and growth structures detected in one or two areas on the sample.
- Level 0: No confirmed growth of identified fungus present on sample; < 1% of sample covered by these organisms. List fungi identified from miscellaneous spores and note presence of hyphal fragments. These may represent fungal spores from settled air flora, deposition of material from growth in adjacent areas or evidence of former growth in the area sampled; provide observations of likely/possible source(s) of spores.
- Also record presence of pollen, insect parts, and fiberglass particles; make comments regarding other noteworthy observations (growth of dominant fungi, other microorganisms, unusual non-biological debris, etc.).

## III. Example

- See attached sample Report and Summary Table for format and terminology.

# **APPENDIX B**

## **MGA SOPs**

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## ***SOP: BULK ASBESTOS SAMPLING***

The following procedures are to be used to collect a bulk samples from building. These procedures may be modified in the field, based on field and site conditions after appropriate annotations have been made in the bound field log book. These procedures are intended for sampling of bulk building materials for asbestos analyses.

The following is a list of equipment for bulk building material sampling:

- Zip lock bags or other air tight containers;
- Spray bottles with water with soap added (“amended” water);
- Plastic drop cloths;
- Coring tool or knife;
- Duct tape or Encapsulant;
- Pre-moistened disposable cloths;
- HEPA vacuum (if needed);
- Camera;
- Indelible ink pen;
- Asbestos disposal bag;
- Sample location and identification labels;
- Locations (map and/or list);
- Appropriate health and safety equipment; and
- Field logbook; and

Bulk samples for asbestos testing must be representative samples of the material to be tested; the sample should contain all layers of the questioned material. Samples from multiple locations should be bagged separately to avoid cross-contamination.

Bulk samples for asbestos must be submitted in a sealed container. Zipped plastic bags are recommended containers.

Although no special sample preservation is necessary, the sample should be handled without exposure to extreme conditions or rough handling so that the received sample is intact and all layers of material in the sample may be examined by analysts handling the material.

Samples must be clearly identified and submitted with corresponding chain of custody.

### **The field sampling procedure is as follows:**

Prior to collecting the sample, ensure the required personal protective equipment (respirator, gloves, etc.) and an approved encapsulant is available for use.

1. Wherever practical, the sample should be collected during quiet hours or when the area surrounding the sampling location is unoccupied.
2. If the material being sampled is friable in nature (i.e. fireproofing, mechanical insulation, etc.), first spray the material in the immediate area surrounding the point of collection with a light misting of water.
3. Where possible, sample collection should be performed adjacent to a point of existing damage. Avoid any unnecessary contact or disturbance.
4. Depending on the condition of the material being sampled, significant amounts of airborne fibers can be discharged during sample collection. The use of a respirator is mandatory in such instances.
5. To avoid possible sample cross-contamination, ensure the knife (or any other instruments) used to collect the sample is properly cleaned using a damp rag following the collection of each individual sample.
6. Should additional fragments or pieces of the material being sampled break off during sample collection, the associated debris must be cleaned up using a HEPA equipped vacuum or damp rag. Unless otherwise indicated through subsequent analysis, dispose of all debris collected as asbestos-containing waste.
7. Place each sample collected in an independently labeled plastic bag (c/w zip-lock closure. Ensure container being used is clean and dry. The exterior of the container must also be wiped clean using a damp cloth to ensure the removal of any visible debris following sample collection.
8. Samples shall be identified with the following information:
  - Date Sampled;
  - Sample ID;
  - Sample Description (i.e. cold water piping, boiler exhaust or sprayed fire proofing, etc.);
  - Sample Location (i.e. building, room number, etc.); and
  - Name and phone number of the individual who collected the sample.
9. Materials of differing composition or appearance should be sampled separately. Mechanical insulation must be sampled separately on a system-by-system basis as well as differentiating between the material present on the straight runs of the piping from material present on any fittings (i.e. tees, valves, elbows, etc.).
10. Ensure full-depth samples are collected for as many products as possible. Products such as finishing plasters or mechanical insulation often involve multiple layers of application or coatings.
11. Following sample collection, temporarily repair jacketing or seal exposed edges of underlying insulation using duct tape or approved asbestos encapsulant (i.e. Serpiflex Shield or approved equivalent).



12. Record sample location on a drawing and through a system of on-site labeling where appropriate. Ensure the data outlined in section 7 above is recorded in the field logbook and maintained on file prior to submitting the sample to the lab.



## **Environmental Lead Sampling Procedures**



## **Procedure for Wipe, Soil, and Paint Samples for Lead**

### **A. Wipe Sampling of Settled Dust**

#### **1.0 Equipment**

- 1.1 Latex or plastic gloves
- 1.2 Sample collection container, 50 mL screw-capped tube
- 1.3 Ghost wipe or other ASTM E 1792-compliant wipe material
- 1.4 Sampling template with 1 square foot opening
- 1.5 Masking tape
- 1.6 Indelible marker
- 1.7 Chain of custody forms (COCs)

#### **2.0 Procedure For Unconfined Areas**

- 2.1 Place the template on the surface carefully to avoid disturbing the settled dust. Tape the edges of the template to hold it in place. Alternatively, a 1 square foot area may be defined using masking tape only.
- 2.2 Put on a pair of latex or plastic gloves.
- 2.3 Open and unfold the Ghost wipe. Drape the Ghost wipe over the fingers of your gloved hand and wipe the area using a side-to-side wiping motion and starting at the corner furthest from you.
- 2.4 Turn the wipe 180 degrees and make a second side-to-side pass in the reverse direction. The 180-degree turn is made so the wiping is always in the same direction to maximize dust pickup.
- 2.5 Fold the wipe once and repeat the wiping using a top-to-bottom wiping motion. At the end of this sweep, use a slight rolling motion to pick up any ridge of dust that may have formed ahead of the wipe.
- 2.6 Fold the wipe again and use a clean side to perform a wipe around the perimeter of the template and clean the corners of any remaining dust.
- 2.7 Fold the wipe again and place in a screw-capped 50 mL tube. Cap the tube tightly.
- 2.8 Label the tube with the sample number and record the sampling information on the chain of custody.

#### **3.0 Procedure for Confined Areas**

- 3.1 Wipe the area as outlined above, using side-to-side, then top-to-bottom, then the perimeter of the area.
- 3.2 Place the sample in a screw-capped 50 mL tube. Label the tube with the sample number.
- 3.3 Measure the area sampled and record this information as well as all other Sample information on the chain of custody. Make sure to include the units of measure.



#### 4.0 Shipping

- 4.1 Return ship by overnight delivery. No refrigeration or preservation is required.

### B. Sampling of Soils

#### 1.0 Equipment

- 1.1 Sample collection container, 50 mL screw-capped tube, clean glass 4 oz jar, or resealable plastic bag.
- 1.2 Spoon, for scoop sampling
- 1.3 Metal or plastic measuring tape or ruler
- 1.4 Core sampling device with sample removal plunger having 0.5 inch stop and plunger without stop
- 1.5 Water, drinking quality, used for cleaning coring equipment
- 1.6 Disposable towelette, used for cleaning coring equipment
- 1.7 Latex or plastic gloves
- 1.8 Indelible marker

#### 2.0 Scoop Sampling Procedure for Friable Soils Using 50 mL tubes.

- 2.1 Put on a pair of clean gloves.
- 2.2 Using a 50 mL tube, scoop the soil to a depth of  $\frac{1}{2}$  inch for a length of 6-12 inches.
- 2.3 Wipe away any soil clinging to the tube and cap it.
- 2.4 Label the tube with a sample number and record all sampling information on the chain of custody.

#### 3.0 Scoop Sampling Procedure for Friable Soils Using A Spoon.

- 3.1 Put on a pair of clean gloves.
- 3.2 Using a measuring tape and a spoon, dig a small test hole near the sampling area to a depth of  $\frac{1}{2}$  inch. Clean the spoon using a wet wipe to remove all traces of soil.
- 3.3 Scoop the soil down to the depth indicated by the test hole and place the sample in the sampling container. Continue until a hole approximately 2 inches diameter by  $\frac{1}{2}$  inch has been created.
- 3.4 Take 2 more samples within a 1-foot diameter circle around the first sample location using the same procedure. Add these to the same sample container.
- 3.5 Seal the container in such a manner as to minimize the air contained in the container.
- 3.6 Label the sample container with a sample number and record the sampling information on the chain of custody.
- 3.7 Clean the spoon using wipes and water to remove all traces of soil.



#### 4.0 Sampling procedure for Nonfriable Soils Using a Core Sampler

- 4.1 Put on a pair of clean gloves.
- 4.2 Grip the coring tool firmly and push it into the soil to a depth of at least two inches using a twisting motion. For very hard soils, a hammer or similar device may be used.
- 4.3 If penetration is less than ½ inch, document the actual depth achieved as part of the sampling information.
- 4.4 Carefully remove the coring tool from the ground while retaining the soil core in the tool.
- 4.5 Insert a clean plunger equipped with stop into the top end of the coring probe. Push out all but ½ inch of soil. Use a gloved finger to wipe off the excess soil protruding from the end of the probe. Do not drop the excess soil on the sampling area.
- 4.6 Using the plunger without stop, push out the remaining ½ inch of soil into the sampling container.
- 4.7 Collect two more cores within a 1-foot diameter circle around the first sample location using the same procedure. Add these to the same sample container.
- 4.8 Label the sample container with a sample number and record the sampling information on the chain of custody.
- 4.9 Clean the coring tools using water and wet wipes to remove all soil traces.

#### 5.0 Shipping

- 5.1 Return ship by overnight delivery. No refrigeration or preservation is required.

### **B. Sampling of Paint Chips**

#### 1.0 Equipment

- 1.1 Sample collection container, resealable plastic bag
- 1.2 Razor scraper
- 1.3 Latex or plastic gloves
- 1.4 Disposable towelette, used for cleaning scraper
- 1.5 Collection device (clean creased piece of paper or cleanable tray)
- 1.6 Indelible marker
- 1.7 Measuring tape or ruler

#### 2.0 Sampling Procedure

- 2.1 During this procedure, make every attempt to remove paint chips without removing the underlying substrate. Including substrate will dilute the reported lead content of the paint. A sample from 2 - 4 square inches is sufficient .
- 2.2 Put on a pair of clean gloves.
- 2.3 Use of a heat gun is recommended to remove paint without underlying substrate. Hold the heat gun at least six inches from the surface. Discontinue heating when softening or blistering is observed. Do not scorch the surface.
- 2.4 Use the razor scraper to remove the softened paint from the substrate and place the



sample in a resealable plastic bag.

- 2.5 If a result in milligrams per square centimeter ( $\text{mg}/\text{cm}^2$ ) is desired, you must measure the area sampled and include this area on the chain of custody.
- 2.6 Label the bag with a sample number and record the sampling information on the chain of custody.

### 3.0 Shipping

- 3.1 Return ship by overnight delivery. No refrigeration or preservation is required.

# Fungal Sampling Methods

Sean P. Abbott, Ph.D.  
Natural Link MOLD LAB, Inc.

## General Considerations

Currently, the method of choice for assessing potential exposures to airborne molds and mycotoxins in indoor environments involves the collection and identification of fungal propagules. Determining types and prevalence of various species of fungi present on surfaces and in the air allows for assessment of active growth within buildings. In many cases, the underlying question is simply “Is active mold growth occurring indoors”? A genus level identification is sufficient in most cases, but for particular cases where correlations are being made to health effects it may be necessary to have all fungi identified to species. In groups that commonly produce secondary metabolites, such as *Penicillium* or *Aspergillus*, the type, quantity, and toxicity of these compounds varies considerably among the species. Indoor sampling protocols should involve a variety of sample types to get a well-rounded assessment. Areas of visible mold may be sampled directly by sending bulk material or tape samples for identification and settled dust may be collected for analysis. Sampling should include air monitoring in selected problem and non-problem areas with an outdoor comparison sample. Culturable and non-culturable methods have differing pros or cons. Although either method will usually detect major problems, a combination of the two provides for the most reliable interpretation.

## Non-Culturable Bioaerosol (Spore-trap)

Non-culturable (also known as “non-viable”) air samples are collected using a variety of spore-trap samplers. Air is drawn across an adhesive, impacting and trapping all particulate matter in the air, including fungal spores. Some sample collection devices utilize adhesive-coated glass microscope slides, including the Burkard, Allergenco MK-3, and BioSIS 2000. Other disposable sampling types include the Air-O-Cell® cassettes using the Zefon Mini-Pump, and the Cyclex-d™ or Micro5 cassettes. Spore-trap samplers have demonstrated an excellent ability to allow sensitive detection of *Stachybotrys* spores present in low levels. They allow for rapid analysis when required and adequately determine the levels and proportions of various spore types determined to genus or broad category. Although they lack some specificity in identification, they recover types of spores that do not grow or compete well in culture. They have the additional advantage of collecting all airborne particles for microscopic observation. Elements such as pollen, insect parts, mites, epithelial cells, fiber glass and carbonaceous debris may be detected to further broaden the scope of the IAQ investigation. An example of a sampling protocol using the Air-O-Cell® cassette is provided below: The Zefon pump is calibrated to a flow rate of 15 liters/minute (Lpm) using a factory-supplied rotameter (annually calibrated by the manufacturer). The tape seal on the cassette inlet and outlet is removed, and the outlet is connected directly to the Mini-Pump port. Make sure the rectangular orifice with a slit inlet is facing outwards on the sampler. Per manufacturers recommendations, each sample is collected for 5 minutes under normal building and outdoor conditions. If the area is very clean (e.g., environmentally controlled office building) with little airborne

dust, the sampling time may be extended to 10 minutes. Likewise, when the environment is highly contaminated or dusty, the sampling time should be reduced accordingly. After sampling is completed, replace the seals over the cassette inlet and outlet. The sample is labeled in conjunction with the sample number and information on the chain of custody (COC) form. Remember to include the sampling time and flow rate (or total sample volume) on the COC so calculations of the total spores/m<sup>3</sup> can be made. Samples will not deteriorate in transit and may be shipped at room temperature to the laboratory at your convenience.

## Culturable Bioaerosol

Culturable (also known as “viable”) air samples are collected on agar culture media. One of the most common types of samplers are the vacuum pump sieve-impaction samplers such as the Andersen N6, a high-volume vacuum pump calibrated to a flow rate of 28.3 liters per minute (Lpm) using a factory-supplied rotameter (annually calibrated by the manufacturer). Other sieve impactors have higher flow rates, including the Surface Air System (SAS Super 100), Millipore MAirT, and the EM Science MAS 100. These samplers employ standard 100 mm Petri dish plates or contact plates for impaction. Alternatively, The Biotest RCS is a centrifugal sampler that uses agar strips for culturable sample collection. Once at the lab, all of these sample types are incubated to induce the impacted fungal spores to grow, allowing identification on both a genus and species level. Sampling recommendations for fungal bioaerosols are consistent with the American Industrial Hygiene Association *Field Guide for the Determination of Biological Contaminants in Environmental Samples* (AIHA, 1996) as well as the manufacturers’ instructions for each sampling device utilized. The limitations of interpreting results obtained from these sampling methodologies are described therein. The primary advantage of culturable bioaerosol sampling is that precise identifications are possible, crucial for species ID of *Penicillium* and *Aspergillus*, and important for the recovery and recognition of a wide variety of potentially toxigenic molds such as *Paecilomyces*, *Fusarium*, *Trichoderma*, *Phoma*, *Acremonium*, and *Wallemia*. An example of a sampling protocol using the Andersen-N6 sampler is provided below. Before each sample is collected, the sampler housing (orifice and base) should be disinfected and cleaned with 70% alcohol and allowed to air dry immediately before the culture plate is placed inside. The sampler should be positioned such that the inlet is facing directly upward at a height corresponding roughly to sitting or standing breathing zones. Remove the cover of the agar plate and insert on the base of the N6. The inlet cone is placed on top of the perforated plate and the entire assembly is sealed using the connecting clamps. A variety of media can be used for sampling depending on the strategy and sampling conditions. Generally for routine analysis of a wide spectrum of fungal species, malt extract agar (MEA) and/or potato dextrose agar (PDA) amended with antibiotics are recommended. Dichloran glycerol 18% agar (DG18) is used to recover some xerophilic (dry tolerant) fungi such as certain *Aspergillus*, *Penicillium* and *Wallemia*. Cellulose agar (CEL) is useful to detect cellulolytic fungi such as *Stachybotrys*, *Chaetomium* and *Alternaria*. Per manufacturers recommendations, each sample should be collected for 2-3 minutes (unless very clean or highly contaminated conditions prevail). After sampling is completed, the cover is replaced and the culture plate is sealed with laboratory film. The collection plate is labeled in conjunction with the sample number and information on the COC form. Remember to record the sampling time and flow rate (or total sample volume) on the COC for calculations of the total CFU/m<sup>3</sup>. Samples are perishable and should be shipped via overnight courier to the laboratory. If sample storage is required prior to shipping, they should be refrigerated. Plates may be transported in insulated coolers (with or without cold packs depending on conditions



expected during shipping) to protect samples from environmental fluctuations during periods when samples might be expected to experience temperature extremes.

## **Surface Tape (Microscopy)**

If you see visible or suspected fungal growth on dry, hard surfaces, the quickest and most cost effective sampling method to confirm mold growth is to sample with a piece of clear tape. Use a piece of Scotch Transparent Tape about 1.5 inches long. Fold one end over to provide a 'handle' and press the sticky surface using light pressure against the area in question; do not vigorously rub back and forth or use excessive pressure. Peel off the tape immediately, and make sure some of the surface material is adhering to the tape. Do not fold the tape on itself, but rather, place the sample inside a clean freezer-strength Zip Lock® bag, or other similar bag, or mount it on a glass microscope slide (ensure slides are adequately protected to prevent breakage in transport). The sample is labeled in conjunction with the sample number and information on the COC form. Samples will not deteriorate in transit and may be shipped at room temperature to the laboratory at your convenience. Unfortunately, one limitation of tape samples is that they are not suitable for culturable analysis.

## **Bulk Samples (Microscopy and Culturable Fungi )**

Any small pieces of building material (e.g., drywall, carpet, baseboard, tack strip, insulation) or contents (e.g., furniture, drapes, clothing, paper) with visible or suspected fungal growth can be collected for analysis. The samples should be sealed into a clean Zip Lock® bag or other container. Water samples should be collected in tightly sealing, sterile, plastic containers. The sample is labeled in conjunction with the sample number and information on the COC form. If culturable analysis is requested, or if the sample is perishable, the package should be shipped via overnight courier. Wet samples should be transported in insulated coolers with cold packs to prevent proliferation of organisms during transit.

## **Dust (Culturable Fungi)**

Settled dust can be collected directly from hard surfaces or extracted from carpeting and other porous surfaces (e.g., furniture, clothing, books and papers) using a 0.45 $\mu$ m methyl cellulose ester (MCE) filter dust cassette and collected with a vacuum pump. The caps on the cassette inlet and outlet are removed and the outlet can be attached to the pump via 2 feet of plastic tubing. Although the samples may be collected for a variable length of time and over a variable area in accordance with the desired sampling goal and dust content of the surface being sampled, a standard pattern of collection helps to provide uniformity and comparability in sample results. For example, to collect carpet dust, go to a low traffic area and sample using a standard time, flow rate, and surface area (e.g., 2 min., 28.3 Lpm, 1 m<sup>2</sup>), while vacuuming the surface horizontally and then vertically to cover the entire area. After sampling is completed, replace the caps over the cassette inlet and outlet. The cassette is labeled in conjunction with the sample number and information on the COC form. Samples will not deteriorate in transit and may be shipped to the laboratory at your convenience.

## Surface Swab (Culturable Fungi)

Visible or suspected fungal growth on hard surfaces can be sampled using a sterile swab (e.g., BBL CultureSwab ) for culturable analysis. The sterile swab is removed from its packaging and can be moistened with the holding medium in the tube prior to use on dry surfaces. The swab is then placed onto the area of concern and rolled to allow a sufficient amount of material to accumulate on the swab tip. The swab is then inserted into the transport medium tube and tightly sealed. To provide a quantitative assessment of specific areas, a template of 1 to 100 cm<sup>2</sup> is used while swabbing the surface horizontally and then vertically to cover the entire area. The swab may also be used for non-culturable microscopic examination, but the tape sample is more appropriate for this type of analysis in most cases. Swabs are especially useful for wet surfaces where material will not adhere adequately to the tape or in hard to access areas. The swab is labeled in conjunction with the sample number and information on the COC form. Samples are perishable and should be shipped via overnight courier to the laboratory. Samples requiring enumeration of bacteria or yeasts should be shipped in an insulated cooler with cold pack to minimize growth in transit.

## Other Microbiological Sampling Methods

### Bacterial Sampling

In general, sample for bacterial contamination following the same guidelines as for the fungi. Some unique situations and differences in strategy include:

- Bacteria are not easily detected by microscopic examination, so culturable analyses should be conducted.
- Airborne bacteria can be collected on tryptic soy agar (TSA)(recommended for a wide spectrum of environmental bacteria).
- Airborne *E. coli*, coliforms, and other gram-negative bacteria can be identified using MacConkey agar (MAC).
- Airborne *Legionella* can only be detected using highly selective agar media (e.g., BCYE).
- If contamination by thermophilic actinomycetes is suspected, use TSA.
- Swabs of humidifiers, air conditioners, machine fluids and other wet sites are an excellent means of assessing overall bacterial contamination.
- Use of sterile, rayon-fiber tipped swabs with holding medium (e.g., BBL CultureSwab) is critical for optimal bacterial recovery. Naturally occurring antimicrobial agents found in cotton may be detrimental to recovery of bacterial contaminants.
- Swab and bulk materials for bacterial analysis should be sent to the laboratory in insulated coolers with a cold pack to minimize growth in transit.

# **APPENDIX C**

## **Chain-of-Custody Forms**

---

Date/Time:

# Chain-of-Custody Form

Natural Link MOLD LAB

4900 Mill Street  
Suite 3  
Reno, NV 89502

Account name \_\_\_\_\_

Sampling date \_\_\_\_\_

Project / P.O. \_\_\_\_\_

Submitter \_\_\_\_\_

Phone \_\_\_\_\_

**(866) 252-6653**  
**(866) 252-MOLD**

Phone (775) 356-6653

Fax (775) 356-6639

info@naturallinkmoldlab.com

Sample identification, description, and/or location	Sample volume	Analysis *					Alternative / additional analysis requested:	RUSH	
		FME	NFME	FC	BC	EC		24hr	48hr

(\*) FME, Fungal Microscopic Examination -- NFME, Non-Fungal Microscopic Exam -- FC, Fungal Culture -- BC, Bacterial Culture -- EC, E.coli (coliforms) ID


Submitter's Signature	Date ____/____/____ Time :      am   pm	Receiver's Signature	Date ____/____/____ Time :      am   pm
Submitter's Signature	Date ____/____/____ Time :      am   pm	Receiver's Signature	Date ____/____/____ Time :      am   pm

Lab use:		Control #:	

# APPENDIX D

## Sample Labels

---

 McGinley & Associates		McGinley & Associates, Inc. 8260 S. Valley View Blvd., Suite 604 Las Vegas, Nevada 89118 702.260.4961			
Analysis:	Lead:		Asbestos Bulk:		Mold:
Sampled By:					
Sample ID:					
Time/Date:					
Location:					
Description:					

# **APPENDIX E**

## **Site Health and Safety Plan**

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**Reno Office**  
815 Maestro Dr.  
Reno, NV 89511  
Ph: 775.829.2245  
Fax: 775.829.2213

**Las Vegas Office**  
8260 S. Valley View Blvd.  
Suite 604  
Las Vegas, NV 89118  
Ph: 702.260.4961  
Fax: 702.260.4968

[www.mcgin.com](http://www.mcgin.com)

# HEALTH AND SAFETY PLAN

## **Brownfields Project Environmental Sampling Tonopah Convention Center 301 Brougner Avenue Tonopah, Nevada**

*Prepared for:*

*Nevada Division of Environmental Protection  
901 South Stewart Street, Suite 4001  
Carson City, NV 89701-5249*

| Soil and Groundwater Remediation

| Regulatory Compliance

| Environmental Audits

| Hydrogeology

| Hazmat Response

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## FIGURES

Figure 1      Site Location Map

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Appendix B    MSDS Sheets for Asbestos and Lead  
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## 1. INTRODUCTION

McGinley & Associates Inc. (MGA) is pleased to submit this Health and Safety Plan (HASP) detailing personal safety precautions being performed on behalf of the Nevada Division of Environmental Protection (NDEP). This HASP addresses activities associated with collection of samples for asbestos, lead based paint, and mold. The sampling activities will be conducted at the Town of Tonopah Convention Center which is located at 301 Brougner Avenue, Tonopah, Nevada.

Planned site activities will include:

- Site reconnaissance;
- Collection of suspect building material for laboratory analysis.

### 1.1 Scope and Applicability of the Site Health and Safety Plan

The purpose of this HASP is to define the requirements and designate protocols to be followed at the site sampling activities. Applicability extends to all MGA employees, contractors and subcontractors. Each person will also be expected to provide his or her own protective equipment.

All on-site personnel shall be informed of the site emergency response procedures and any potential fire, explosion, health, or safety hazards of the operation. This HASP summarizes hazards and defines protective measures planned for the site. This plan must be reviewed and signed by all site personnel prior to commencing with field activities. An agreement of compliance is provided in Appendix A.

During development of this plan consideration was given to current safety standards as defined by EPA/OSHA/NIOSH, health effects and standards for known contaminants, and procedures designed to account for the potential for exposure to unknown substances. Specifically, the following reference sources have been consulted:

- OSHA 29 CFR parts 1910.120, 1910.134, 1926.350 and 1926.650;
- U.S. EPA, OERR ERT Standard Operating Safety Guides
- NIOSH/OSHA/USCG/EPA Occupational Health and Safety Guidelines
- (ACGIH) Threshold Limit Values

### 1.2 On-Site Personnel

All personnel entering the designated work areas at the Site are responsible for the following:

- Taking all reasonable precautions to prevent injury to themselves and to their fellow employees, and being alert to potentially harmful situations;
- Obeying all applicable laws and regulations relating to health and safety;
- Ensuring that activities do not impact the neighboring community;

- Performing only those tasks that they have been trained to complete and can do safely;
- Notifying their supervisor of any special medical conditions (i.e., allergies, contact lenses, diabetes) that may affect their ability to perform certain tasks;
- Notifying their supervisor of any prescription and/or non-prescription medication that they may be taking that might cause drowsiness, anxiety, or other unfavorable side effects;
- Learning and complying with Site security requirements;
- Complying with the Site's prohibition on drug and alcohol use, smoking, horseplay, and restricted eating/drinking areas;
- Practicing good housekeeping by keeping the work areas neat, clean and orderly;
- Immediately reporting all injuries, incidents and near-misses to the HSO;
- Properly using PPE specified by this HASP.
- Properly maintaining their designated PPE per manufacturers' recommendations.
- Complying with the HASP and all health and safety recommendations and precautions.

In the event that a person does not adhere to the provisions of the HASP, he/she will be requested to leave the work area. All non-conformance incidents will be recorded in the site log.

## 2. KEY PERSONNEL

The Site Health and Safety Officer (HSO) has total responsibility for ensuring that the provisions of this HASP are adequate and implemented in the field. Changing field conditions may require decisions to be made concerning adequate protection programs. Therefore, it is vital that personnel assigned as HSO be experienced and meet the additional training requirements specified by OSHA in 29 CFR 1910.120. The following personnel are critical to the planned activities at the Site. The organizational structure will be reviewed and updated periodically by the site supervisor.

Title/Responsibility	Name	Phone
<b>Town of Tonopah</b>		
Administrative Supervisor	Susan Dudley	(775) 482-6336
<b>McGinley and Associates, Inc.</b>		
Project Manager – Project management, regulatory liaison, coordinate field activities, site safety, data review, report preparation.	Brett Bottenberg	(702) 232-5247
Environmental Scientist – Oversee field activities, collect samples.	Gene Johnson	(775) 829-2245
<b>Contractors/Vendors</b>		
Natural Link Mold Lab – Analysis of mold samples	Sean Abbott	(775) 746-3838
Asbestos TEM Laboratories, Inc. - Analysis of paint and ACM samples	Sue Ehrlich	(775) 359-3377

## 2.1 Site Specific Health and Safety Personnel

The HSO is also responsible for conducting site inspections on a regular basis in order to ensure the effectiveness of this plan.

The HSO at the site is: Brett Bottenberg

Designated alternates include: Gene Johnson

## 2.2 Organizational Responsibility

Town of Tonopah:	Party initiating investigation of suspected asbestos, lead based paint, and mold impacts to the building.
MGA:	Primary agent for the Town of Tonopah providing field services and project oversight of asbestos, lead based paint, and mold sampling.
Subcontractors:	Various companies and organizations providing services or skilled trades.

## 3. TASK/OPERATION SAFETY AND HEALTH RISK ANALYSIS

### 3.1 Historical Overview of Site

The existing Tonopah Convention Center facility was built in the 1940s as the USO during World War II. The 11,354 square foot facility is approaching 70 years old and has undergone several additions. The facility has been used for many years to accommodate a variety of activities, including community events such as graduations, funerals, and weddings.

The Tonopah Town Board uses this facility to hold Town Board Meetings. The community has come to rely heavily on this facility for its community based events. Due to the age and condition of the facility, the Town wants to rehabilitate it to include it in the Town's plan to promote more outside events and subsequently promote economic development within the Town of Tonopah.

The purpose of this project is to assess the occurrence and extent of asbestos, lead, and mold contamination within the Convention Center building. In 2009, prior to renovation of the building's roof, samples of the roof building material were collected and analyzed for asbestos. Results indicated that some of the samples collected contained asbestos. The Town of Tonopah is also concerned that due to the age of the structure, paint found within the building may contain lead.

## 3.2 Chemical Hazards

The following sections provide descriptions of the principal health hazards of the potential contaminants affecting this investigation and include:

- Asbestos
- Lead

### 3.2.1 Asbestos

Asbestos fibers are usually mixed with various binder materials or resinous matrices. Collecting bulk samples of building materials may release extremely low concentrations of asbestos fibers. Asbestos occurs as bundles of fibers that, when disturbed, are easily separated into smaller and smaller sizes. Micron-size fibers tend to remain airborne and, because of their small size, can be inhaled down to the alveolar surface (smallest ends of air passageways) of the lungs.

Exposure to elevated levels of airborne asbestos fibers is known to cause a number of asbestos-related diseases, including asbestosis (fibrosis of the lung), mesothelioma (cancer of the lining of the lung), and other cancers of the lung, esophagus, stomach, and colon. Although the risk of developing asbestos-related diseases is greatest for individuals who are regularly exposed to relatively high airborne asbestos fiber concentrations (e.g., industrial asbestos workers), it is apparent that some degree of elevated risk exists for individuals chronically exposed to low airborne asbestos fiber concentrations, which may be present in a building that contains friable ACM. The actual degree of risk associated with prolonged exposure to asbestos levels in this range is still unknown at this time; however, it is prudent to take steps to limit asbestos exposure to the lowest extent possible.

OSHA has established standards for limiting the exposure of personnel working with asbestos. As described in the OSHA Standard (29 CFR 1910.1001), the current permissible exposure limit (PEL) for asbestos, as an 8-hour time weighted average (TWA), is 0.1 fiber per cubic centimeter of air (f/cc). The OSHA 8-hour TWA action limit is 0.05 f/cc. There is no OSHA standard regarding asbestos exposure for the general public.

### 3.2.2 Inorganic Lead

Inorganic lead exposure can occur via inhalation or ingestion of lead-containing dusts. Skin and eye contact are not considered routes of entry of lead dust into the body. The principal target organs of lead toxicity include the nervous system, kidneys, blood, gastrointestinal, and reproductive systems. Generalized symptoms of lead exposure include decreased physical fitness, fatigue, sleep disturbances, headaches, bone and muscle pain, constipation, abdominal pain, and decreased appetite. More severe exposure can result in anemia, severe gastrointestinal disturbance, a "lead-line" on the gums, neurological symptoms, convulsions, and death.

Neurological effects are among the most severe of inorganic lead's toxic effects and vary depending on the age of individual exposed. Effects observed in adults occur primarily in the peripheral nervous system, resulting in nerve destruction and degeneration. Wrist-drop and foot-drop are two characteristic manifestations of this toxicity.

The EPA also currently lists inorganic lead as a Group B2 probable human carcinogen via the oral route. This conclusion is based on feeding studies conducted in laboratory animals. The current PEL-TWA for inorganic lead is 0.05 mg/m<sup>3</sup>. Occupational exposure to lead is also

specifically regulated under WAC 296-62-07521, with an action level established at 0.03 mg/m<sup>3</sup> that triggers monitoring and other requirements. It is not anticipated that any sampling activities involving potential exposure to lead will trigger monitoring requirements for lead, because of the extremely low concentrations released to the air during paint sampling activities.

### **3.3 Biological Hazards**

Biological hazards that may be encountered during sampling activities and preventative measures include mold and fungal exposure. Mold growth is encouraged by warm and humid conditions. It is likely to grow and become a problem where there is water damage, high humidity, or dampness. It is estimated that about 50 to 100 common indoor mold types have the potential for creating health problems.

Humans are primarily exposed to these molds through inhalation. Exposure to molds can cause symptoms such as nasal stuffiness, eye irritation, or wheezing. Some people, such as those with serious allergies to molds, may have more severe reactions. Currently, there are no indoor air regulations pertaining to mold or fungal exposure.

### **3.4 General Hazards**

General hazards that may be encountered during sampling activities and preventative measures are described in the following sections and include:

- Slips, trips, and falls
- Elevated noise levels
- Electrical hazards
- Hazards associated with lifting and carrying
- Heat/cold stress

#### **3.4.1 Slips, Trips, and Falls**

Falls are a leading cause of occupational fatalities. These fatalities are considered preventable with the use of fall protection systems. The following is a list of common fall hazards:

- Elevated work at > 6 feet above lower level with unprotected sides or edges
- Wall openings > 4 feet above lower level
- Floor/Roof openings (hatches)
- Floor/Roof holes (deterioration), i.e. failing roof
- Ramps, walkways, bridges
- Excavations

Protection from fall hazards can be achieved in one of three ways: 1) fixed position systems, 2) personal fall protection, and 3) safety monitoring systems. A combination of these three protection systems is often used to ensure the safety of site workers. Fixed position systems consist of guardrails, safety nets, and floor covers. Personal fall protection will consist of a full-body harness with a 6-foot shock-absorbing lanyard. Good housekeeping, proper PPE, and daily safety meetings can minimize injuries from falls.



### **3.4.2 Elevated Noise Levels**

During on-site activities requiring the use of power equipment, hearing protection may be required to be worn for certain tasks or in designated areas where noise levels reach > 85 dBA. Training on proper use of hearing protection will be conducted prior to initiation of specified onsite work.

### **3.4.3 Hazards Associated with Sharp Tools**

Sampling activities may require the use of sharp tools when cutting or chipping samples from building materials. Cuts and punctures may occur if care is not heeded. Use extreme care when using sharp instruments. Retract blades into containers, or hold blades and sharp tools away from the body when walking.

### **3.4.4 Hazards Associated with Lifting and Carrying**

The human body is subject to severe damage in the form of back injury and/or hernia if caution is not observed in the handling process. General rules for minimizing injuries from manual lifting are:

- Get good footing.
- Place feet shoulder width apart.
- BEND AT KNEES to grasp object.
- Keep back straight.
- Get a good grip on object.
- Lift gradually by straightening the legs.
- GET HELP if object is too heavy for you to lift (usually 50-60 lbs lifting limit).

## **3.5 Task Hazard Analysis**

### **3.5.1 Collection of Asbestos Samples**

Inhalation of asbestos in dusts may occur if care is not heeded during collection of bulk suspect asbestos samples. Use wet methods to collect samples. Spray areas damaged by sampling with adhesive or encapsulant to hold down fibers.

### **3.5.2 Collection of Lead Based Paint Samples**

Inhalation of lead in dusts may occur if care is not heeded during collection of bulk suspect lead based paint samples. Use wet methods or adhesive tape as necessary to avoid generating any dusts. Repair areas damaged during sampling immediately.

### **3.5.3 Collection of Mold Samples**

If sampling activities disturb mold and mold spores become airborne, then the risk of respiratory exposure goes up. Actions that are likely to stir up mold include: breakup of moldy porous materials such as wallboard; invasive procedures used to examine or remediate mold growth in a wall cavity; actively stripping or peeling wallpaper to remove it; and using fans to dry items. Care should be taken to avoid the previously stated actions.

## **4. PERSONNEL TRAINING REQUIREMENTS**

Consistent with OSHA's 29 CFR 1910.120, regulation covering Hazardous Waste Operations and Emergency Response and, OSHA's 29 CFR 1926 Construction Industry Standards, workers are required to be trained in accordance with those standards. At a minimum, all

personnel are required to be trained to recognize the hazards on-site and the provisions of this HASP.

#### 4.1 Pre-assignment and Annual Refresher Training

Prior to arrival on site, each employer will be responsible for certifying that his/her employees meet the requirements of training, consistent with OSHA 29 CFR 1910.120 paragraph (e)(3) or (e)(9). The employer should be able to provide a document certifying that each general site worker has received 40 hours of instruction off the site, and 24 hours of training for any workers who are on site only occasionally for a specific task. If an individual employee has work experience and/or training that is equivalent to that provided in the initial training, an employer may waive the 40-hour training so long as that equivalent experience is documented or certified. All personnel must also receive 8 hours of refresher training annually.

#### 4.2 Training and Briefing Topics

The following items may be discussed by a qualified individual at the site pre-entry briefing(s) and at periodic tailgate safety meetings.

Physical Hazards

Chemical Hazards

Emergency Response Plan

Air Monitoring

Training Requirements

Animal Bites and Stings

Respiratory Protection

Medical Surveillance

Site Control

Personal Protective Equipment

Heavy Machinery

### 5. PERSONAL PROTECTIVE EQUIPMENT TO BE USED

This section describes the general requirements of the EPA designated Levels of Protection (A-D), and the specific levels of protection required for each task at the site.

#### 5.1 Levels of Protection

Personnel wear protective equipment when response activities involve known or suspected atmospheric contamination vapors, gases, or particulate that may be generated by site activities, or when direct contact with skin-affecting substances may occur. The specific levels of protection and necessary components for each have been divided into four categories according to the degrees of protection afforded:

Level A: Should be worn when the highest level of respiratory, skin, and eye protection is needed.

Level B: Should be worn when the highest level of respiratory protection is needed, but a lesser level of skin protection. Level B is the primary level of choice when encountering unknown environments.

Level C: Should be worn when the criteria for using air-purifying respirators are met,

and a lesser level of skin protection is needed.

**Level D:** Should be worn only as a work uniform and not in any area with respiratory or skin hazards. It provides minimal protection against chemical hazards.

Modifications of these levels are permitted, and routinely employed during site work activities to maximize efficiency. For example, Level C respiratory protection and Level D skin protection may be required for a given task. Likewise the type of chemical protective ensemble (i.e., material, format) will depend upon contaminants and degrees of contact. The Level of Protection selected is based upon the following:

- Type and measured concentration of the chemical substance in the ambient atmosphere and its toxicity.
- Potential for exposure to substances in air, liquids, or other direct contact with material due to work being done.
- Knowledge of chemicals on-site along with properties such as toxicity, route of exposure, contaminant matrix, and adequate warning properties.

In situations where the type of chemical, concentration, and possibilities of contact are not known, the appropriate Level of Protection must be selected based on professional experience and judgment until the hazards can be better identified. For all unknown situations on this site, Level D is the highest level anticipated.

## 5.2 Recommended Levels of Protection – Task Specific

The following specific personal protective ensembles are recommended for the site:

- **Respiratory Protection** - It is not anticipated that respiratory protection will be necessary during routine sampling activities, unless damaged materials containing asbestos, lead or mold are present. A half-mask respirator with HEPA cartridges will be worn by personnel whenever undue risk of exposure to lead or asbestos exists. Such situations could arise if sampling in areas with a large amount of suspect asbestos or lead dust or debris. Respirators, if used, shall be NIOSH/MSHA-approved. Cartridges shall be changed whenever breathing resistance increases noticeably. Cartridge changes shall be made only in areas outside the area in which respiratory protection is being used. All respiratory protection will follow OSHA Safety and Health Standards 29 CFR 1910.134.
- **Gloves** - Work gloves (Scorpio or equivalent) will be worn as necessary to avoid skin contact with sharp objects or rough edges on equipment.
- **Other Protective Equipment** - Safety glasses will be used while sampling for lead based paint.

## 5.3 Reassessment of Protection Program

The level of Protection provided by PPE selection shall be upgraded or downgraded based upon a change in site conditions or findings of investigations. When a significant change

occurs, the hazards should be reassessed and the HASP updated. Some indicators of the need for reassessment are:

- Commencement of a new work phase, such as the start of unexpected sampling or work that begins on a different portion of the site;
- Change in job tasks during a work phase;
- Contaminants other than those previously identified are encountered;
- Change in ambient levels of contaminants;
- Change in work scope which affects the degree of contact with contaminants.

## **5.4 SOP for Personal Protective Equipment**

Proper inspection of PPE features several sequences of inspection depending upon specific articles of PPE and its frequency of use. The different levels of inspection are as follows:

- Inspection and operational testing of equipment received from the factory or distributor;
- Inspection of equipment as it is issued to workers;
- Inspection after use or training and prior to maintenance;
- Periodic inspection of stored equipment; and
- Periodic inspection when a question arises concerning the appropriateness of the selected equipment, or when problems with similar equipment arise.

The primary inspection of PPE in use for activities at the site will occur prior to immediate use and will be conducted by the user. This ensures that the specific device or article has been checked-out by the user and that the user is familiar with its use.

## **6. MEDICAL SURVEILLANCE REQUIREMENTS**

Medical monitoring programs are designed to track the physical condition of employees on a regular basis as well as survey pre-employment or baseline conditions prior to potential exposures. The medical surveillance program is a part of each employers Health and Safety program. Exposure to toxic materials is not anticipated at the Site.

### **6.1 Exposure/Injury/Medical Support**

As a follow-up to an injury or possible exposure above established exposure limits, all employees are entitled to and encouraged to seek medical attention and physical testing. Depending upon the type of exposure, it is critical to perform follow-up testing within 24-28 hours. It will be up to the employer's medical consultant to advise the type of test required to accurately monitor for exposure effects.

## **7. EXPOSURE MONITORING/AIR MONITORING**

Exposure monitoring will not take place at the Site.

## **8. SITE CONTROL MEASURES**

No hazardous waste operations are anticipated to require sampling for this project, so site control requirements are not needed.

## **9. DECONTAMINATION PLAN**

Consistent with the levels of protection required, the decontamination process provides a step by step representation of the personnel decontamination steps for level D and C. These procedures should be modified to suit site conditions and protective ensembles in use. Decontamination involves the orderly controlled removal of contaminants. All site personnel should minimize contact with contaminants in order to minimize the need for extensive decontamination.

### **9.1 Personnel Decontamination**

All workers exposed to asbestos or lead will be required to enact an orderly removal of contaminated PPE. This can be accomplished through repeated change of disposable garments and or PPE wash at the end of the shift. Workers shall be instructed to the importance of decontamination to prevent cross contamination.

### **9.2 Sampling Equipment Decontamination**

All sampling tools (except cutter sleeves which will not be reused) will be thoroughly sprayed with amended water prior to collecting another sample. When decontaminating equipment that has been used to sample materials such as floor tile mastic or roofing materials, it may be necessary to use a nonflammable solution that dissolves tar rather than amended water. Wipes or other towels used during decontamination should be placed in a zipped plastic bag for later disposal. If decontamination is not possible immediately after sample collection, contaminated sampling equipment will be placed in zipped plastic bags until decontamination can be performed.

## **10. EMERGENCY RESPONSE/CONTINGENCY PLAN**

This section describes contingencies and emergency planning procedures to be implemented at the Site. This plan is compatible with local, state, and federal disaster and emergency management plans as appropriate.

### **10.1 Pre-Emergency Planning**

A field pre-construction / field activities meeting will be conducted at the project site prior to implementation of field services. The meeting will include personnel from MGA and the selected contractors. Each of the activities and procedures presented will be reviewed during this meeting.

In addition, tailgate site safety discussions will be held daily. All employees will be trained in and reminded of provisions of the emergency response plan, communication systems, and evacuation routes. The plan will be reviewed and revised if necessary, on a regular basis by the HSO. This will ensure that the plan is adequate and consistent with prevailing site conditions.

## 10.2 Emergency Recognition/Prevention

Section 3 provides a listing of chemical hazards onsite. Additional hazards as a direct result of site activities are listed in Section 3.2 as are prevention and control techniques/mechanisms. Personnel will be familiar with techniques of hazard recognition from pre-assignment training and site specific briefings. The HSO is responsible for ensuring that prevention devices or equipment is available to personnel.

## 10.3 Evacuation Routes/Procedures

Since all individuals sampling will be within shouting distance, no special alarm system is anticipated as necessary. Contact appropriate emergency authorities. No other situation calling for site evacuation is reasonably anticipated.

## 10.4 Emergency Contact/Notification System

The following list provides names and telephone numbers for emergency contact personnel. In the event of a medical emergency, personnel will take direction from the HSO and notify the appropriate emergency organization. In the event of a fire or spill, the site supervisor will notify the appropriate local, state, and federal agencies.

Organization	Telephone
Ambulance:	911
Police:	911
Fire:	911
Nye Regional Medical Center	775-482-6233
NDEP	775-687-4670
Regional EPA:	415-744-1500
EPA Emergency Response Team:	908-321-6660
National Response Center:	800-424-8802
Center for Disease Control:	404-488-4100
Chemtrec:	800-424-9555

## 10.5 Nearest Medical Assistance

The nearest medical facility is the Nye Regional Medical Center. The facility is located at 825 Main Street, Tonopah, Nevada. A map of the route to this facility which can provide emergency care for individuals who may experience an injury or exposure on site is included in Appendix C of this HASP. The route to the facility should be verified by the HSO prior to sampling activities, and should be familiar to all site personnel.

## 10.6 Emergency Medical Treatment Procedures

Any person who becomes ill or injured in the work area must be decontaminated to the maximum extent possible. If the injury or illness is minor, full decontamination should be completed and first aid administered prior to transport. If the patient's condition is serious, at least partial decontamination should be completed (i.e., complete disrobing of the victim and redressing in clean coveralls or wrapping in a blanket.) First aid should be administered while awaiting an ambulance or paramedics. All injuries and illnesses must immediately be reported to the project manager.

## 10.7 Fire or Explosion

In the event of a fire or explosion, the local fire department should be summoned immediately. Upon their arrival, the project manager or designated alternate will advise the fire commander of the location, nature, and identification of the hazardous materials on site. If it is safe to do so, site personnel may:

- Use fire-fighting equipment available on site to control or extinguish the fire; and
- Remove or isolate flammable or other hazardous materials which may sustain a fire.

## 10.8 Emergency Equipment/Facilities

All emergency equipment will be located in the command post and/or support zone and shall include:

- First aid kit;
- Fire extinguisher;
- Mobile telephone;
- Eye wash station.

## 11. HAZARD COMMUNICATION

In order to comply with 29 CFR 1910.1200, Hazard Communication, the following written Hazard Communication Program has been established. All employees will be briefed on this program and have a written copy for review.

### 11.1 Container Labeling

All containers received on site will be inspected to ensure the following:

- All containers will be clearly labeled as to the contents;
- The appropriate hazard warnings will be noted; and
- The name and address of the manufacturer will be listed.

All secondary containers will be labeled with either an extra copy of the original manufacturer's label or with generic labels which have a block for identify and blocks for the

hazard warning.

## **11.2 Material Safety Data Sheets (MSDSs)**

Copies of MSDSs for all hazardous chemicals known on site will be maintained in the work area. MSDSs will be available to all employees for review during each work shift.

## **11.3 Employee Training and Information**

Prior to starting work, each employee will attend a health and safety orientation and will receive information and training on the following:

- An overview of the requirements contained in the Hazard Communication Standard, 29 CFR 1910.1200;
- Chemicals present in their workplace operations;
- Location and availability of a written hazard program;
- Physical and health effects of the hazardous chemicals;
- Methods and observation techniques used to determine the presence or release of hazardous chemicals;
- How to lessen or prevent exposure to these hazardous chemicals through usage of control/work practices and personal protective equipment;
- Emergency procedures to follow if they are exposed to these chemicals;
- How to read labels and review MSDSs to obtain appropriate hazard information;
- Specialized hot work and tank processing techniques.



# **APPENDIX A**

## **Agreement of Compliance**

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# **HASP**

## **Statement of Compliance**

I have read and understand the HASP for the site investigation of asbestos, lead based paint, and mold at the project site in Tonopah, Nevada.

I agree to comply with the contents of the HASP and understand that not doing so may be reason for discharge from the site.

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

# **APPENDIX B**

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## **MSDS Sheets for Asbestos and Lead**

# Material Safety Data Sheet

## Asbestos

RTECS NUMBER : CI6478000  
CHEMICAL NAME : Asbestos, anthophyllite  
CAS REGISTRY NUMBER : 77536-67-5  
LAST UPDATED : 199712  
DATA ITEMS CITED : 26  
COMPOUND DESCRIPTOR : Tumorigen  
Mutagen  
Natural Product

### SYNONYMS/TRADE NAMES :

- \* Anthophyllite asbestos
- \* Asbestos
- \* Azbolen asbestos
- \* Ferroanthophyllite

### \*\*\* HEALTH HAZARD DATA \*\*\*

#### \*\* TUMORIGENIC DATA \*\*

TYPE OF TEST : TCLo - Lowest published toxic concentration  
ROUTE OF EXPOSURE : Inhalation  
SPECIES OBSERVED : Rodent - rat  
DOSE/DURATION : 11 mg/m3/1Y-I

#### TOXIC EFFECTS :

Tumorigenic - Carcinogenic by RTECS criteria  
Lungs, Thorax, or Respiration - tumors

#### REFERENCE :

BJCAAI British Journal of Cancer. (Macmillan Press Ltd., Houndmills,  
Basingstoke, Hants. RG21 2XS, UK) V.1- 1947- Volume(issue)/page/year:  
29,252,1974

TYPE OF TEST : TDLo - Lowest published toxic dose  
ROUTE OF EXPOSURE : Intraperitoneal  
SPECIES OBSERVED : Rodent - rat  
DOSE/DURATION : 250 mg/kg

#### TOXIC EFFECTS :

Tumorigenic - equivocal tumorigenic agent by RTECS criteria  
Tumorigenic - tumors at site of application

#### REFERENCE :

ZHYGAM Zeitschrift fuer die Gesamte Hygiene und Ihre Grenzgebiete. (VEB  
Verlag Volk und Gesundheit, Neue Gruenstr. 18, Berlin DDR-1020, Ger. Dem.  
Rep.) V.1- 1955- Volume(issue)/page/year: 32,89,1996

TYPE OF TEST : TDLo - Lowest published toxic dose  
ROUTE OF EXPOSURE : Intrapleural  
SPECIES OBSERVED : Rodent - rat  
DOSE/DURATION : 300 mg/kg/12W-I

#### TOXIC EFFECTS :

Tumorigenic - equivocal tumorigenic agent by RTECS criteria  
Tumorigenic - tumors at site of application

#### REFERENCE :

ZHYGAM Zeitschrift fuer die Gesamte Hygiene und Ihre Grenzgebiete. (VEB  
Verlag Volk und Gesundheit, Neue Gruenstr. 18, Berlin DDR-1020, Ger. Dem.  
Rep.) V.1- 1955- Volume(issue)/page/year: 32,89,1996

# Material Safety Data Sheet

## Asbestos

TYPE OF TEST : TDLo - Lowest published toxic dose  
ROUTE OF EXPOSURE : Intrapleural  
SPECIES OBSERVED : Rodent - rat  
DOSE/DURATION : 200 mg/kg

TOXIC EFFECTS :

Tumorigenic - neoplastic by RTECS criteria  
Lungs, Thorax, or Respiration - tumors

REFERENCE :

BJCAAI British Journal of Cancer. (Macmillan Press Ltd., Houndmills,  
Basingstoke, Hants. RG21 2XS, UK) V.1- 1947- Volume(issue)/page/year:  
28,173,1973

TYPE OF TEST : TDLo - Lowest published toxic dose  
ROUTE OF EXPOSURE : Intrapleural  
SPECIES OBSERVED : Rodent - hamster  
DOSE/DURATION : 83 mg/kg

TOXIC EFFECTS :

Tumorigenic - neoplastic by RTECS criteria  
Tumorigenic - tumors at site of application

REFERENCE :

31BYAP "Experimental Lung Cancer: Carcinogenesis and Bioassays,  
International Symposium, 1974," Karbe, E., and J.F. Park, eds.,  
Springer-Verlag New York, Inc., 1974 Volume(issue)/page/year: -,92,1974

TYPE OF TEST : TD - Toxic dose (other than lowest)  
ROUTE OF EXPOSURE : Intrapleural  
SPECIES OBSERVED : Rodent - rat  
DOSE/DURATION : 2400 mg/kg/34W-I

TOXIC EFFECTS :

Tumorigenic - equivocal tumorigenic agent by RTECS criteria  
Tumorigenic - tumors at site of application

REFERENCE :

IAPUDO IARC Publications. (WHO Publications Centre USA, 49 Sheridan Ave.,  
Albany, NY 12210) No.27- 1979- Volume(issue)/page/year: 30,343,1980

\*\* MUTATION DATA \*\*

TYPE OF TEST : Morphological transformation  
TEST SYSTEM : Rodent - hamster Embryo  
DOSE/DURATION : 3500 ug/m3

REFERENCE :

CRNGDP Carcinogenesis (London). (Oxford Univ. Press, Pinkhill House,  
Southfield Road, Eynsham, Oxford OX8 1JJ, UK) V.1- 1980-  
Volume(issue)/page/year: 9,891,1988

TYPE OF TEST : DNA inhibition  
TEST SYSTEM : Rodent - hamster Lung  
DOSE/DURATION : 250 mg/L

REFERENCE :

TIVIEQ Toxicology In Vitro. (Pergamon Press Inc., Maxwell House, Fairview  
Park, Elmsford, NY 10523) V.1- 1987- Volume(issue)/page/year: 1,71,1987

TYPE OF TEST : Cytogenetic analysis  
TEST SYSTEM : Rodent - hamster Ovary  
DOSE/DURATION : 10 mg/L

# Material Safety Data Sheet

## Asbestos

### REFERENCE :

CSHCAL Cold Spring Harbor Conferences on Cell Proliferation. (Cold Spring Harbor, NY) V.1-10, 1974-83. Volume(issue)/page/year: 4,941,1977

### \*\*\* REVIEWS \*\*\*

ACGIH TLV-Confirmed human carcinogen

DTLVS\* The Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs) booklet issues by American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, OH, 1996 Volume(issue)/page/year: TLV/BEI,1997

ACGIH TLV-TWA 2 fibers/cc

DTLVS\* The Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs) booklet issues by American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, OH, 1996 Volume(issue)/page/year: TLV/BEI,1997

IARC Cancer Review:Human Sufficient Evidence

IMEMDT IARC Monographs on the Evaluation of Carcinogenic Risk of Chemicals to Man. (WHO Publications Centre USA, 49 Sheridan Ave., Albany, NY 12210) V.1- 1972- Volume(issue)/page/year: 14,11,1977

IARC Cancer Review:Animal Sufficient Evidence

IMEMDT IARC Monographs on the Evaluation of Carcinogenic Risk of Chemicals to Man. (WHO Publications Centre USA, 49 Sheridan Ave., Albany, NY 12210) V.1- 1972- Volume(issue)/page/year: 14,11,1977

IARC Cancer Review:Animal Sufficient Evidence

IMEMDT IARC Monographs on the Evaluation of Carcinogenic Risk of Chemicals to Man. (WHO Publications Centre USA, 49 Sheridan Ave., Albany, NY 12210) V.1- 1972- Volume(issue)/page/year: 2,17,1973

IARC Cancer Review:Group 1

IMSUDL IARC Monographs, Supplement. (WHO Publications Centre USA, 49 Sheridan Ave., Albany, NY 12210) No.1- 1979- Volume(issue)/page/year: 7,106,1987

### \*\*\* U.S. STANDARDS AND REGULATIONS \*\*\*

MSHA STANDARD-air:TWA 5 fb/cc (fb > 5 um)

DTLWS\* "Documentation of the Threshold Limit Values for Substances in Workroom Air," Supplements. For publisher information, see 85INA8. Volume(issue)/page/year: 3,33,1973

OSHA-cancer hazard

CFRGBR Code of Federal Regulations. (U.S. Government Printing Office, Supt. of Documents, Washington, DC 20402) Volume(issue)/page/year: 29,1910.1001,1987

### \*\*\* OCCUPATIONAL EXPOSURE LIMITS \*\*\*

OEL-SWITZERLAND:TWA 1 fiber/ml;Carcinogen JAN 1993

# Material Safety Data Sheet

## Asbestos

OEL-UNITED KINGDOM:TWA 0.5 fiber/ml/4H JAN 1993

OEL-UNITED KINGDOM:TWA 1.5 fiber/ml/10H JAN 1993

OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA check ACGIH TLV

OEL IN NEW ZEALAND, SINGAPORE, VIETNAM check ACGIH TLV

\*\*\* NIOSH STANDARDS DEVELOPMENT AND SURVEILLANCE DATA \*\*\*

NIOSH RECOMMENDED EXPOSURE LEVEL (REL) :

NIOSH REL TO ASBESTOS-air:100M TWA 0.1 fb/cc in a 400L air sample

REFERENCE :

NIOSH\* National Institute for Occupational Safety and Health, U.S. Dept.  
of

Health, Education, and Welfare, Reports and Memoranda.

Volume(issue)/page/year: DHHS #92-100,1992

NIOSH OCCUPATIONAL EXPOSURE SURVEY DATA :

NOES - National Occupational Exposure Survey (1983)

NOES Hazard Code - X1654

No. of Facilities: 6009 (estimated)

No. of Industries: 64

No. of Occupations: 58

No. of Employees: 71986 (estimated)

No. of Female Employees: 5782 (estimated)

\*\*\* STATUS IN U.S. \*\*\*

NIOSH Analytical Method, 1994: Asbestos (bulk) by PLM, 9002; by PCM, 7400;  
by TEM, 7402

NTP 7th Annual Report on Carcinogens, 1992 : known to be carcinogenic

\*\*\* END OF RECORD \*\*\*

# MATERIAL SAFETY DATA SHEET

This Material Safety Data Sheet complies with the U.S. OSHA Hazard Communications Standard 29CFR 1910.1200 and the Hazardous Products Act of the Canada Labour Code.

PRODUCT: Lead (Fabrications / Forms)

## SECTION I

## SECTION II - HAZARDOUS INGREDIENTS

US-NIOSH INGREDIENT	CAS NO.	RTECS NO.	US OSHA 8 HR AL	US OSHA 8 HR PEL	ACGIH 8 HR TLV	WT. %
Lead	7439-92-1	0F7525000	0.03mg/m <sup>3</sup>	0.05mg/m <sup>3</sup>	0.15mg/m <sup>3</sup>	99.8+
AL = Action Level			PEL = Permissible Exposure Limit		TLV = Threshold Limit Value	

## SECTION III - PHYSICAL DATA

APPEARANCE & ODOR (AT NORMAL CONDITIONS)	:	Solid - silver metallic to gray metallic metal - no odor
SPECIFIC GRAVITY (H <sub>2</sub> O = 1)	:	11.34
MELTING POINT (DEGREES C)	:	328
BOILING POINT (DEGREES C)	:	1744
SOLUBILITY IN WATER	:	Insoluble
EVAPORATION RATE (BUTYL ACETATE = 1)	:	Not Applicable
VAPOR DENSITY (AIR = 1)	:	Not Applicable
VAPOR PRESSURE (mmHg)	:	Not Applicable
PH	:	Not Applicable

## SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT	:	Non-Flammable
FLAMMABLE LIMITS	:	Not Applicable
EXTINGUISHING MEDIA	:	No specific agents recommended
SPECIAL FIRE FIGHTING PROCEDURES	:	Use full protective clothing and NIOSH/MSHA self-contained breathing apparatus operated in a positive-pressure mode
UNUSUAL FIRE AND EXPLOSION HAZARDS	:	None

## SECTION V - REACTIVITY DATA

STABILITY	:	Stable
CONDITIONS TO AVOID	:	Not Applicable
INCOMPATIBILITY	:	Strong Oxidizers, Hydrogen Peroxide, Active Metals as Sodium, Potassium. Powdered lead fused ammonium nitrate may cause a violent reaction.
HAZARDOUS DECOMPOSITION PRODUCTS	:	NEVER mix molten metal with water - it will explode. At temperatures above the melting point lead oxide fumes may be evolved.
HAZARDOUS POLYMERIZATION	:	Will not occur

## SECTION VI - HEALTH HAZARD DATA

**NOTE:** EXPOSURE TO THE SOLID FORM OF THIS PRODUCT PRESENTS FEW HEALTH HAZARDS IN ITSELF. HOWEVER, NORMAL HANDLING OR PROCESSING OF THIS MATERIAL MAY RESULT IN GENERATION OF LEAD DUST AND/OR FUMES, WHICH MAY PRESENT A POTENTIAL HEALTH HAZARD.



## SECTION VI - HEALTH HAZARD DATA (CONTINUED)

ROUTES OF ENTRY	:	Inhalation of dust/fume & ingestion of dust are the two major routes of entry of inorganic lead into the human body.
COMMON METHODS OF CONTROL	:	Ingestion can be prevented by exercising normal, good personal hygiene prior to smoking or eating. Smoking and eating should be confined to noncontaminated areas. Users should not smoke while installing or handling this product and should wash hands, face, neck, and arms before eating, smoking, or applying cosmetics. Work clothes and equipment should remain in designated lead contaminated areas and should never be taken home or laundered with personal laundry. Contaminated clothing should be laundered separately before reuse. Most inhalation problems can be prevented by use of proper ventilation and respirator methods discussed in Section VII.
SYMPTOMS & EFFECTS OF OVEREXPOSURE :		Chronic (prolonged) overexposure to lead can result in systemic lead poisoning with symptoms of metallic taste, anemia, insomnia, weakness, constipation, abdominal pain, gastrointestinal disorders, joint and muscle pains, and muscular weakness, and may cause damage to the blood-forming, nervous, kidney, and reproductive systems. Damage may include reduced fertility in both men and women, damage to the fetus of exposed pregnant women, anemia, muscular weakness & kidney disfunction. Acute (Severe short-term) overexposure to lead may lead to nervous system disorders, characterized by drowsiness, seizures, coma and death. It should be recognized that exposures of this magnitude in an industrial or construction environment are extremely unlikely.
MEDICAL CONDITIONS POSSIBLE AGGRAVATED BY EXPOSURE	:	Diseases of the blood and blood forming organs, kidneys, nervous & possibly reproductive systems.
CARCINOGENICITY	:	Not listed as a carcinogen by NTP, OSHA, or ACGIH. IARC classifies "lead and its compounds" as a Group 2B carcinogen (possibly carcinogenic to humans)
ADDITIONAL INFORMATION	:	Lead and its compounds have been tentatively classified by the USEPA Carcinogen Assessment Group as a Group B2 Carcinogen (Probable human carcinogen - a combination of sufficient evidence in animals and inadequate data for humans). IARC lists lead and its compounds as a teratogen.
EMERGENCY & FIRST AID PROCEDURES	:	SKIN : Normal hygiene & first-aid procedures - wash with soap and water.
EYES	:	Flush well with running water to remove particulate. If irritation persists, get medical attention.
ACUTE INHALATION	:	Remove from exposure. Obtain immediate medical attention. If breathing has stopped, initiate artificial respiration.
INGESTION	:	Give water; induce vomiting only in a conscious non-convulsing individual; obtain immediate medical attention.

## SECTION VII - PROTECTION MEASURES

RESPIRATORY PROTECTION :	Respiratory protection is required where airborne exposures exceed U.S. OSHA/ACGIH permissible air concentrations. Respirator selection shall be made in accordance with the U.S. Occupational Exposure Standard for Lead, 29 CFR 1910.1025 & the Respiratory Protection Standard, 29 CFR 910.134.
VENTILATION :	Good general dilution ventilation or ventilation as described in "Industrial ventilation, A manual of Recommended Practice", by the ACGIH, is recommended to maintain exposure levels below the permissible exposure limits (PEL's) or threshold limit values (TLV's) specified by U.S. OSHA or other local state regulations.
or	

## SECTION VIII - PRECAUTIONS FOR SAFE HANDLING & USE

PRECAUTIONS TO BE TAKEN : IN HANDLING & STORING	Practice good housekeeping procedures to prevent dust accumulations. Keep material dry. Avoid storage near incompatible materials (See Section V) Keep product away from children & their environment, feed products, food and domestic animals.
products, OTHER PRECAUTIONS :	Special attention is drawn to the requirements of the U.S. OSHA Lead Standard (29 CFR 1910.1025) and Respiratory Protection Standard, 29 CFR 1910.134) should airborne exposures exceed the U.S. OSHA Action Level (AL) or PEL. Inadvertant contaminants to product such as moisture, ice, snow, grease, or oil can cause an explosion when charged to a molten metal bath or melting furnace. Preheat to eliminate this risk.

## SECTION IX - SPILL OR LEAK PROCEDURES

SPILL OR LEAK PROCEDURES:	1) Material in dust form - minimize exposure. Clean up using dustless methods (e.g. HEPA vacuum). Do not use compressed air. 2) Place in closed labeled containers for recycling or disposal. 3) Keep out of water ways. Note: Cleanup personnel should wear protective clothing and respiratory protection where dust/fume exposure exists.
WASTE DISPOSAL METHODS :	May have value on a recycled basis. If disposed of, do so in a Permitted disposal site in accordance with all federal, state, and local disposal or discharge regulations. The user of the product must determine whether the product and the form it is in falls under the U.S. Resource Conservation and Recovery Act (RCRA) as a hazardous waste.

## SECTION X - UNITED STATES SARA TITLE III INFORMATION

CHEMICAL NAME	EHS RQ (LBS) (1)	EHS TPQ (LBS) (2)	SECT. 313 (3)	313 CAT. (4)	311/312 CAT. (5)
Lead	N/A	N/A	Yes	Lead	H-1, H-2
(1) =	Reportable quantity of extremely hazardous substance, Section 302				
(2) =	Threshold planning quantity, extremely hazardous substance, Section 302				
(3) =	Toxic chemical list, Section 313				
(4) =	Chemical category as required by Section 313 (40 CFR 372.42). Subject to annual release reporting requirements.				
(5) =	Hazard category as required by Section 313/312 reporting:				
	Health H-1 = Immediate (Acute) Health Hazard		Physical P-3 = Fire Hazard		
	H-2 = Delayed (Chronic) Health Hazard		P-4 = sudden release of pressure hazard.		
			P-5 = Reactive Hazard		

## SECTION XI - UNITED STATES CERCLA SECTION 103 INFORMATION

THIS PRODUCT/MIXTURE CONTAINS THE FOLLOWING CHEMICALS SUBJECT TO THE RELEASE REPORTING REQUIREMENTS OF SECTION 302.

CHEMICAL NAME	RQ (LBS) (1)	
Lead	1.0	CERCLA STATUTORY RQ

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SECTION XI - UNITED STATES CERCLA SECTION 103 INFORMATION (CONTINUED)

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FOOTNOTES

(1) Reportable quantity (RQ) under CERCLA Section 302. Spills to the environment exceeding the reportable quantity in any 24 hour period must be reported to the U.S. National Response Center (800-424-8802). Reporting of releases of the hazardous substance(s) is NOT required if the diameter of the pieces of the solid metal(s) released is equal to or exceeds 100 micrometers (0.004 inches).

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SECTION XII - USDOT TRANSPORTATION INFORMATION

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DOT SHIPPING NAME : This product is not regulated by the USDOT as shipped.

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SECTION XIII - ADDITIONAL INFORMATION

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UNITED STATES - CLEAN WATER ACT: The use of lead pipes or sheet lead in any private or public potable water supply is prohibited by the Clean Water Act.

UNITED STATES - STATE HAZARDOUS  
SUBSTANCE LIST  
1986 Lead is on the state hazardous substance lists of MA and NJ, and on the California Safe Drinking Water and Toxic Enforcement Act of Chemical List.

CANADA - HPA WHMIS LIST : Lead is on this list.

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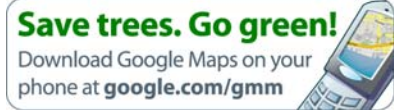
# **APPENDIX C**


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## **Route to Nearest Medial Assistance**




**Directions to Nye Regional Medical Center-Emr**  
825 S Main, Tonopah, NV 89049 - (775) 482-6233  
**0.7 mi – about 2 mins**




 301 Brougher Ave, Tonopah, NV 89049

1. Head **east** on **Brougher Ave** toward **Central St** go 472 ft  
total 472 ft

 2. Turn right onto **N Main St** go 0.1 mi  
total 0.2 mi

3. Continue onto **Erie St** go 0.4 mi  
total 0.6 mi

 4. Turn right  
Destination will be on the left go 381 ft  
total 0.7 mi

 **Nye Regional Medical Center-Emr**  
825 S Main, Tonopah, NV 89049 - (775) 482-6233

These directions are for planning purposes only. You may find that construction projects, traffic, weather, or other events may cause conditions to differ from the map results, and you should plan your route accordingly. You must obey all signs or notices regarding your route.

Map data ©2011 Google

Directions weren't right? Please find your route on [maps.google.com](http://maps.google.com) and click "Report a problem" at the bottom left.